

2-Amino-4-(4-methoxyphenyl)-4H-benzo[h]-chromene-3-carbonitrile

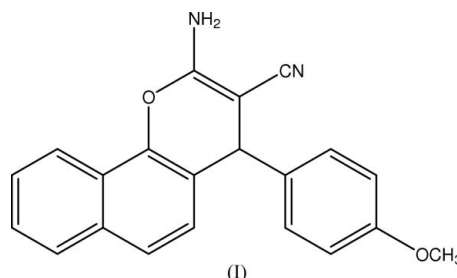
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.051
 wR factor = 0.202
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2$, was synthesized by the reaction of 1-naphthol with malononitrile and 4-methoxybenzaldehyde in ethanol under microwave irradiation. A weak intramolecular $\text{C}-\text{H} \cdots \pi$ interaction is found.Received 6 September 2005
Accepted 19 September 2005
Online 28 September 2005

Comment

Benzopyrans and their derivatives are important in natural and synthetic organic chemistry owing to their biological and pharmacological properties (Morioka & Takahashi, 1977), such as antisterility (Brooks, 1998) and anticancer activities (Hyana & Saimoto, 1987). In addition, polyfunctionalized benzopyrans constitute the structural units 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).We report here the crystal structure of the title compound, (I). The molecular structure of (I), shown in Fig. 1, exhibits a weak intramolecular $\text{C}-\text{H} \cdots \pi$ interaction (Table 2).

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (5 mmol) with malononitrile (5 mmol) and 4-methoxybenzaldehyde (5 mmol) in ethanol (4 ml) using piperidine as catalyst under microwave irradiation. Pure compound (I) was obtained by recrystallization from ethanol (m.p. 464–466 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis: ^1H NMR (CDCl_3 , δ , p.p.m.): 8.23 (*d*, 1H), 7.88 (*d*, 1H), 7.55–7.65 (*m*, 3H), 7.16 (*d*, 2H), 7.08–7.10 (*m*, 3H), 6.87 (*d*, 2H), 4.84 (*s*, 1H), 3.71 (*s*, 3H).

Crystal data

 $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 328.36$
Triclinic, $P\bar{1}$
 $a = 6.4140$ (13) Å
 $b = 10.643$ (2) Å
 $c = 13.308$ (3) Å
 $\alpha = 108.59$ (3)°
 $\beta = 96.11$ (3)°
 $\gamma = 96.95$ (3)°
 $V = 844.5$ (3) Å³ $Z = 2$
 $D_x = 1.291$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 25
reflections
 $\theta = 10$ – 12°
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
Block, colourless
 $0.4 \times 0.4 \times 0.2$ mm

Data collection

Enraf–Nonius CAD4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: none
3594 measured reflections
3287 independent reflections
2023 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 26.0^\circ$
 $h = 0 \rightarrow 7$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 15$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.202$
 $S = 1.02$
3287 reflections
235 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.130P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{Å}^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.051 (10)

Table 1

Selected geometric parameters (Å, °).

O1–C2	1.375 (3)	N1–C9	1.153 (3)
O1–C1	1.410 (4)	N2–C11	1.344 (3)
O2–C11	1.361 (2)	C5–C8	1.532 (3)
O2–C12	1.393 (3)	C9–C10	1.414 (3)
C2–O1–C1	118.5 (2)	C9–C10–C8	117.47 (18)
C11–O2–C12	119.09 (16)	N2–C11–C10	127.1 (2)
O1–C2–C3	116.3 (2)	N2–C11–O2	110.26 (18)
O1–C2–C7	124.4 (2)	C10–C11–O2	122.6 (2)
C6–C5–C8	121.15 (18)	C21–C12–O2	122.50 (18)
C4–C5–C8	121.51 (19)	O2–C12–C13	114.15 (18)
C21–C8–C5	111.38 (16)	C14–C13–C12	123.1 (2)
C10–C8–C5	111.96 (17)	C19–C18–C17	122.2 (2)
N1–C9–C10	177.3 (2)	C20–C21–C8	120.52 (19)
C11–C10–C9	119.4 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the O2/C11/C10/C8/C21/C12 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C6–H6A \cdots Cg1	0.93	2.77	3.1243	104

The N-bound H atoms were located in a difference Fourier map and refined freely [$N2-H2 = 0.96$ (3) Å and $N2-H1 = 0.90$ (3) Å].

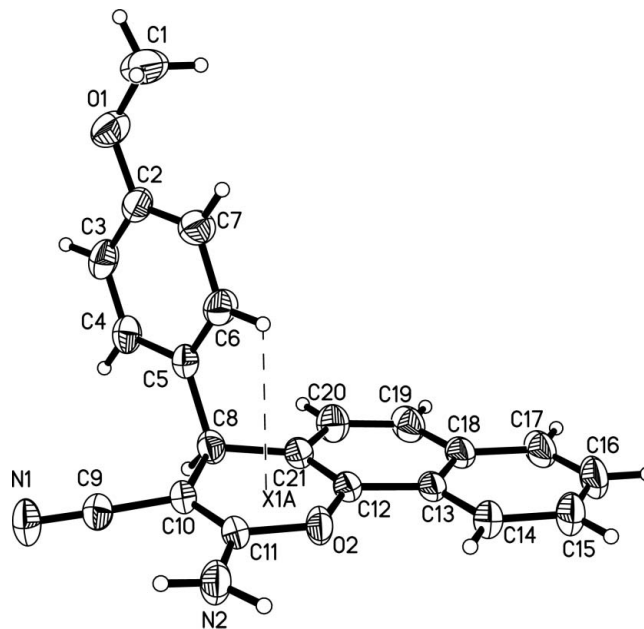


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The weak C–H \cdots π interaction is shown as a dashed line.

The C-bound H atoms were placed in calculated positions (C–H = 0.93–0.97 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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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