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

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Key indicators

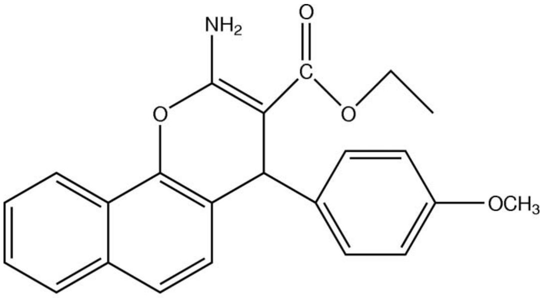
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.184
Data-to-parameter ratio = 14.5

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

The title compound, $C_{23}H_{21}NO_4$, was synthesized by the reaction of 1-naphthol with ethyl cyanocaetate and 4-methoxybenzaldehyde in ethanol under microwave irradiation. In the structure of $C_{23}H_{21}NO_4$, there are intramolecular and intermolecular N–H···O hydrogen bonds, also C–H···π interactions.

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, these are versatile synthons (Hatakeyama *et al.*, 1988). We report here the crystal structure of the title compound,(I).



(I)

The molecular structure of (I) is shown in Fig. 1, where the dashed line indicates the N—H···O intramolecular hydrogen

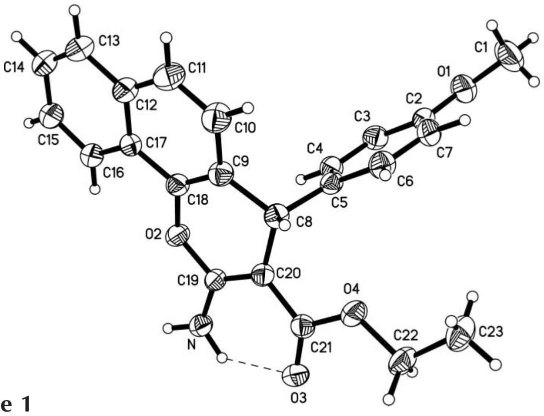


Figure 1
A view of the molecular structure of (I). The dashed line indicates the intramolecular N—H···O hydrogen bond.

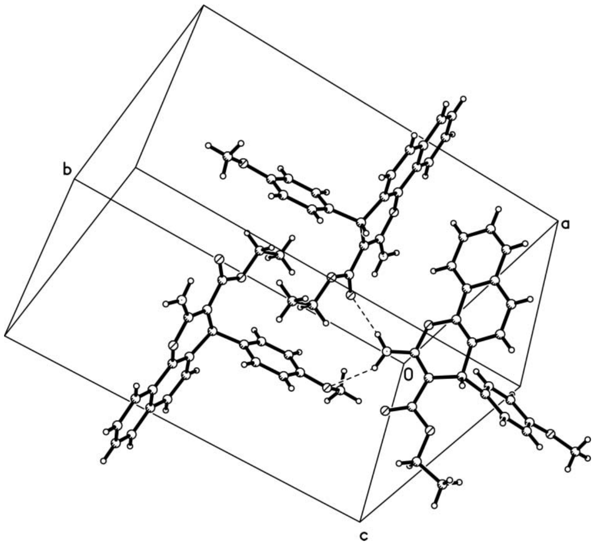


Figure 2
The crystal structure of (I). Dashed lines indicate intermolecular N—H···O hydrogen bonds.

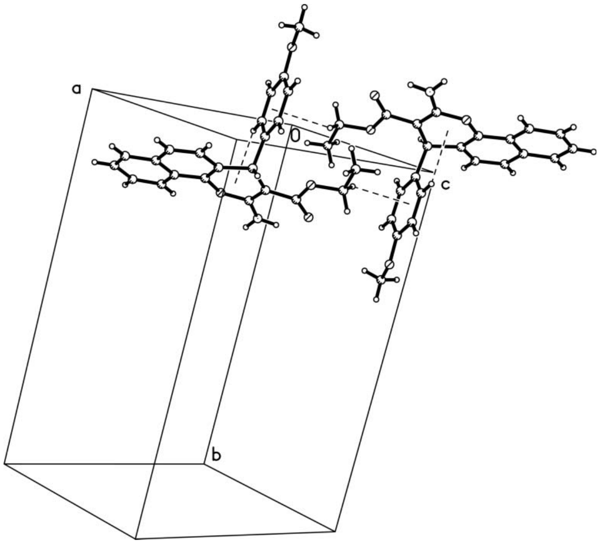


Figure 3
The C—H···π interactions in (I), shown as dashed lines.

bond (Table 2). In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds (Table 2 and Fig. 2). There are also intra- and intermolecular contacts which indicate weak C—H···π interactions (Fig. 3). Full details of the hydrogen-bond geometries are given in Table 2. The combination of rather weak interactions generates a three-dimensional network.

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (5 mmol) with ethyl cyanocaetate (5 mmol) and 4-methoxybenzaldehyde (5 mmol) in ethanol (2 ml), using piperidine as catalyst under microwave irradiation. Pure compound (I) was obtained by recrystallization from ethanol (m.p. 428–430 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR (CDCl$_3$): δ 8.21 (*d*, 1H), 7.75 (*d*, 1H), 7.46–7.56 (*m*, 3H), 7.14–7.18 (*m*, 3H), 6.74 (*d*, 2H), 6.42 (*s*, 2H), 5.01 (*s*, 1H), 4.10 (*q*, 2H), 3.73 (*s*, 3H), 1.20 (*t*, 3H).

Crystal data

$C_{23}H_{21}NO_4$
$M_r = 375.41$
Monoclinic, $P2_1/c$
$a = 12.060$ (2) Å
$b = 18.511$ (4) Å
$c = 8.9360$ (18) Å
$\beta = 102.99$ (3)°
$V = 1943.8$ (7) Å^3
$Z = 4$
$D_x = 1.283$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10–13°$
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4053 measured reflections
3800 independent reflections
2000 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 26.0°$
$h = -14 \rightarrow 14$
$k = -22 \rightarrow 0$
$l = 0 \rightarrow 11$
3 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.184$
$S = 0.99$
3800 reflections
262 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.09P)^2 + 0.2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0084 (19)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.380 (3)	O4—C22	1.449 (3)
O1—C1	1.415 (4)	N—C19	1.333 (3)
O2—C19	1.370 (3)	C5—C8	1.522 (4)
O2—C18	1.393 (3)	C20—C21	1.450 (4)
O3—C21	1.222 (3)	C22—C23	1.498 (4)
O4—C21	1.351 (3)		
C2—O1—C1	117.8 (3)	C9—C18—O2	122.5 (2)
C19—O2—C18	118.3 (2)	O2—C18—C17	114.0 (2)
C21—O4—C22	117.0 (2)	N—C19—C20	127.8 (2)
C7—C2—O1	125.2 (3)	N—C19—O2	109.6 (2)
O1—C2—C3	115.6 (3)	C20—C19—O2	122.6 (2)
C6—C5—C8	121.9 (2)	C19—C20—C21	118.4 (2)
C4—C5—C8	120.5 (2)	C21—C20—C8	119.6 (2)
C20—C8—C5	114.7 (2)	O3—C21—O4	121.9 (3)
C5—C8—C9	108.9 (2)	O3—C21—C20	126.7 (3)
C10—C9—C8	120.2 (3)	O4—C21—C20	111.4 (2)
C11—C12—C13	122.8 (3)	O4—C22—C23	106.7 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H1···O3$^{\rm i}$	0.87 (3)	2.14 (3)	3.002 (3)	172 (3)
N—H2···O3	0.91 (4)	2.10 (3)	2.711 (3)	123 (3)
N—H2···O1$^{\rm ii}$	0.91 (4)	2.41 (4)	3.207 (4)	147 (3)
C4—H4*A*···*Cg*1	0.93	2.68	3.028 (1)	142
C22—H22*A*···*Cg*2$^{\rm iii}$	0.97	2.90	3.636 (4)	134

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y, -z+2$. *Cg*1 is the centroid of the O2/C18/C9/C8/C20/C19 ring and *Cg*2 is the centroid of the C2–C7 ring.

The N-bound H atoms were located in a difference Fourier map and refined freely. The C-bound H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(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* (Siemens, 1996).

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