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Ethyl 2-amino-4-(2-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 = 0.057
wR factor = 0.187
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.

The title compound, $C_{23}H_{21}NO_4$, was synthesized by the reaction of 1-naphthol with ethyl cyanoacetate and 2-methoxybenzaldehyde in ethanol under microwave irradiation. In the molecular structure, there is an intramolecular N—H···O hydrogen bond.

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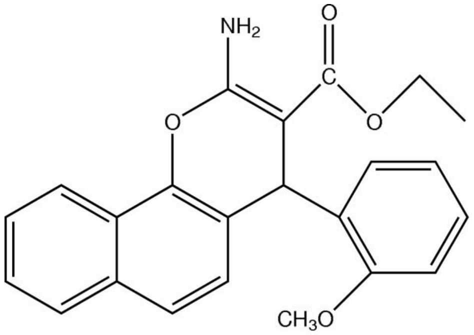
Comment

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 intramolecular N—H···O hydrogen

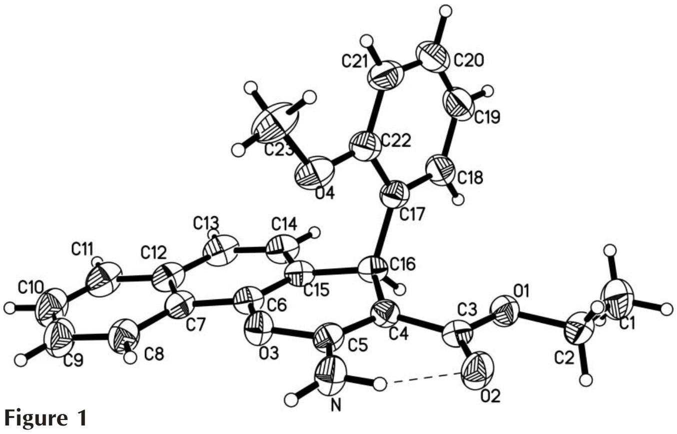


Figure 1
A view of the molecular structure of (I). The dashed line indicates the intramolecular N—H···O hydrogen bond.

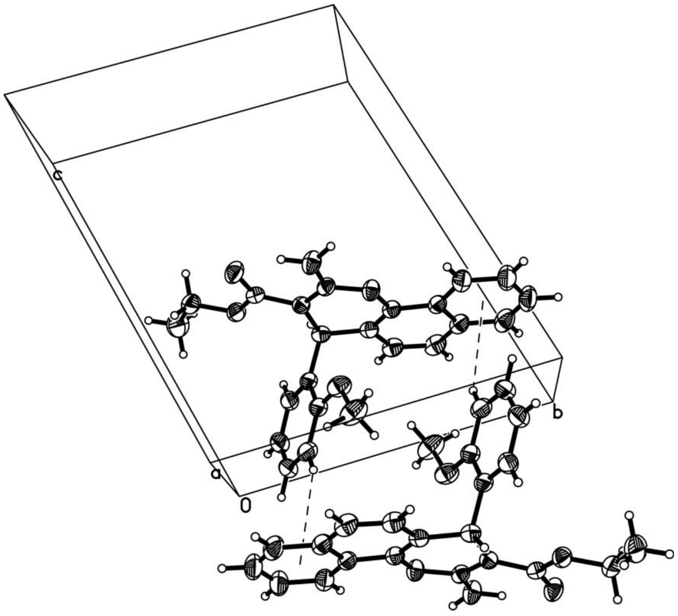


Figure 2
The C—H$\cdots\pi$ interactions in (I), shown as dashed lines.

bond (Table 2). There is also an intermolecular contact which indicate a weak C—H$\cdots\pi$ interaction (Fig. 2). 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 cyanoacetate (5 mmol) and 2-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. 427–429 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.^{1}H NMR (CDCl$_3$): δ 8.19 (*d*, 1H), 7.72 (*d*, 1H), 7.41–7.52 (*m*, 3H), 7.29 (*d*, 1H), 7.21–7.25 (*m*, 1H), 7.07–7.10 (*m*, 1H), 6.79–6.82 (*m*, 2H), 6.44 (*s*, 2H), 5.55 (*s*, 1H), 4.02 (*m*, 2H), 3.83 (*s*, 3H), 1.08 (*t*, 3H).

Crystal data

$C_{23}H_{21}NO_4$
$M_r = 375.41$
Triclinic, $P\bar{1}$
$a = 6.9960$ (14) Å
$b = 10.542$ (2) Å
$c = 13.522$ (3) Å
$\alpha = 101.45$ (3)°
$\beta = 96.33$ (3)°
$\gamma = 96.82$ (3)°
$V = 961.3$ (3) Å^3
$Z = 2$
$D_x = 1.297$ 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
4100 measured reflections
3775 independent reflections
2310 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\rm max} = 26.0°$
$h = 0 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.187$
$S = 1.05$
3775 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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.050 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.344 (3)	O4—C23	1.435 (3)
O1—C2	1.449 (3)	N—C5	1.343 (3)
O2—C3	1.230 (3)	C1—C2	1.494 (4)
O3—C5	1.363 (3)	C3—C4	1.443 (3)
O3—C6	1.391 (3)	C16—C17	1.521 (3)
O4—C22	1.372 (3)		
C3—O1—C2	116.1 (2)	C15—C6—O3	122.4 (2)
C5—O3—C6	118.39 (18)	O3—C6—C7	113.8 (2)
C22—O4—C23	118.9 (2)	C8—C7—C6	123.5 (2)
O1—C2—C1	107.4 (2)	C13—C12—C11	123.2 (3)
O2—C3—O1	120.9 (2)	C14—C15—C16	120.7 (2)
O2—C3—C4	126.2 (2)	C4—C16—C17	115.16 (18)
O1—C3—C4	112.9 (2)	C15—C16—C17	110.03 (18)
C5—C4—C3	118.3 (2)	C18—C17—C16	120.4 (2)
C3—C4—C16	120.8 (2)	C22—C17—C16	122.1 (2)
C4—C5—N	126.9 (2)	O4—C22—C21	124.0 (3)
C4—C5—O3	123.66 (19)	O4—C22—C17	115.6 (2)
N—C5—O3	109.4 (2)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N—H2$\cdots$O2	0.91 (3)	1.93 (3)	2.671 (4)	137 (2)
C21—H21*A*$\cdots$*Cg*3$^{\rm i}$	0.93	2.96	3.7227	141

Symmetry codes: (i) $-x+1, -y+1, -z$. *Cg*3 is the centroid of atoms C7–C12 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 $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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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