

## Methyl 3-amino-1-(3-nitrophenyl)-1H-naphtho[2,1-b]pyran-2-carboxylate

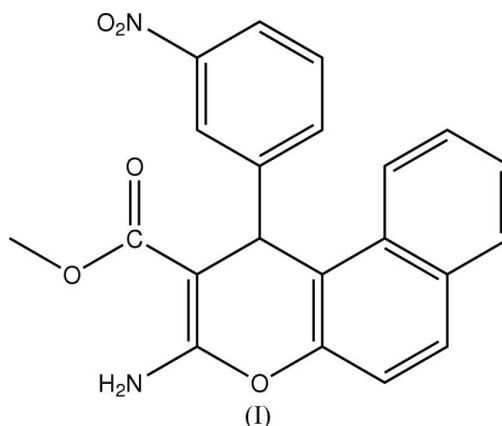
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## Key indicators

Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.062  
 $wR$  factor = 0.186  
Data-to-parameter ratio = 13.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The title compound,  $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_5$ , was synthesized by the  
reaction of 2-naphthol with methyl cyanoacetate and 3-  
nitrobenzaldehyde in ethanol under microwave irradiation.  
The pyran ring adopts a boat conformation.Received 15 June 2006  
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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,  
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 in-built pyran ring, they may  
serve as versatile synthons (Hatakeyama *et al.*, 1988). We  
report here the crystal structure of the title compound, (I).In the molecule of (I) (Fig. 1), all bond lengths and angles  
are normal. The pyran ring adopts a boat conformation. The  
dihedral angle between the C3–C14 and C16–C21 planes is  
 $86.93(7)^\circ$ . An intramolecular N–H...O hydrogen bond  
(Table 2) influences the orientation of the carboxylate group.  
The crystal packing is stabilized by van der Waals forces.

## Experimental

Compound (I) was prepared by the reaction of 2-naphthol (5 mmol)  
with methyl cyanoacetate (5 mmol) and 3-nitrobenzaldehyde  
(5 mmol) in ethanol (2 ml) using piperidine (0.5 mmol) as catalyst  
under microwave irradiation for 8 min. Pure compound (I) was  
obtained by recrystallization from ethanol (m.p. 446–448 K). Crystals  
of (I) suitable for X-ray diffraction were obtained by slow evapora-  
tion of an ethanol solution.

Crystal data

C<sub>21</sub>H<sub>16</sub>N<sub>2</sub>O<sub>5</sub>  
*M<sub>r</sub>* = 376.36  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 12.094 (2) Å  
*b* = 8.5620 (17) Å  
*c* = 18.186 (4) Å  
 β = 108.92 (3)°  
*V* = 1781.4 (7) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.403 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 μ = 0.10 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, colourless  
 0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 ω/2θ scans  
 Absorption correction: none  
 3659 measured reflections  
 3490 independent reflections

2032 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.021  
 θ<sub>max</sub> = 26.0°  
 3 standard reflections  
 every 200 reflections  
 intensity decay: none

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.062  
*wR*(*F*<sup>2</sup>) = 0.186  
*S* = 1.03  
 3490 reflections  
 254 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.09P)^2 + 0.27P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.23 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.19 e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.017 (3)

Table 1

Selected torsion angles (°).

O2–C2–C3–C4	–2.8 (5)	O5–N2–C20–C19	6.3 (4)
O1–C2–C3–C15	0.1 (4)	O4–N2–C20–C21	5.7 (4)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1–H1A...O2	0.86	2.09	2.687 (5)	126

The amino H atoms were initially located in a difference Fourier map, and were then placed in idealized positions and constrained to ride on their parent atoms, with N–H = 0.86 Å and *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(N). The C-bound H atoms were placed in calculated positions (C–H = 0.93–0.98 Å) and refined as riding, with *U*<sub>iso</sub>(H) = 1.2–1.5*U*<sub>eq</sub>(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*

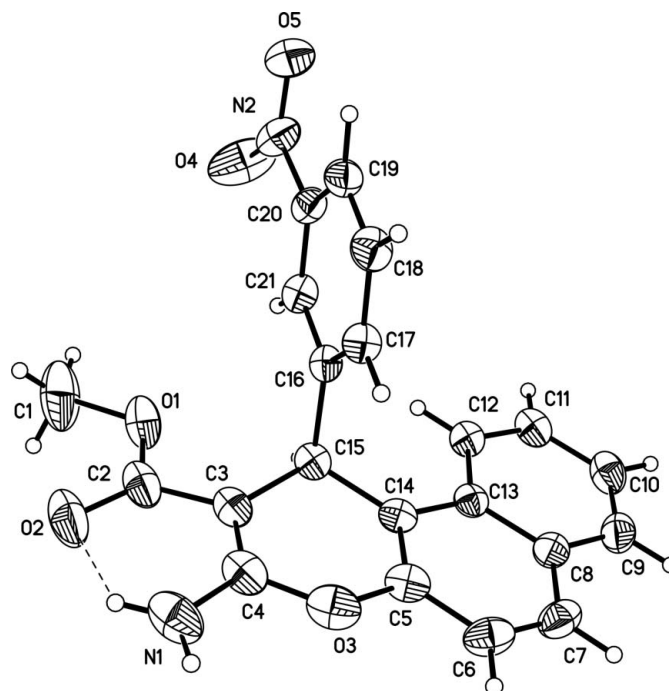


Figure 1

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.

(Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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