Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 4-(3-amino-2,4-dicyano-5,6,7,8-tetrahydronaphthalen-1-yl)benzoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.135
Data-to-parameter ratio = 8.3

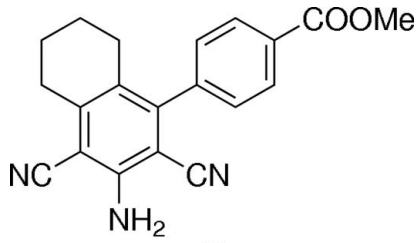
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 August 2005
Accepted 6 September 2005
Online 14 September 2005

In the molecule of the title compound, $C_{20}H_{17}N_3O_2$, the two benzene rings are nearly perpendicular, whereas the acetate group is slightly twisted with respect to the attached benzene ring. Intermolecular N—H···N and N—H···O hydrogen bonds generate a zigzag chain which extends parallel to the *b* axis.

Comment

2,6-Dicyanoanilines comprising one electron donor and two electron acceptors have been reported with high fluorescence quantum yields (Cui *et al.*, 2005). The structure determination of the title compound, (I), was undertaken as part of our studies of 2,6-dicyanoaniline derivatives (Cui *et al.*, 2005).



(I)

The molecular structure of (I) is shown in Fig. 1. The two benzene rings are nearly perpendicular, making a dihedral angle of 86.94 (9)°. The COOMe group is slightly twisted by 7.4 (3)° with respect to the attached benzene ring.

N—H···N hydrogen bonding results in the formation of dimers, which are then connected to each other through N—H···O hydrogen bonds, building zigzag chains parallel to the *b* axis (Fig. 2).

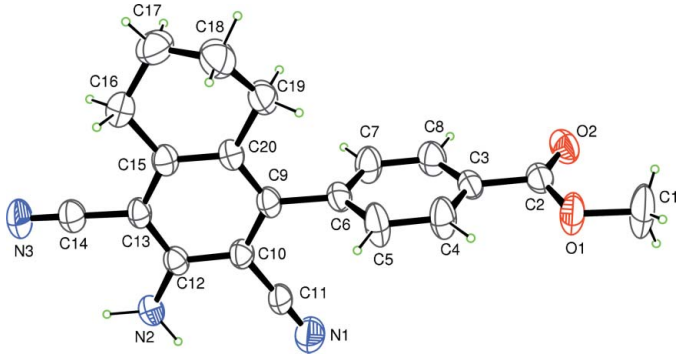


Figure 1
A view of the molecule of compound (I). Displacement ellipsoids are drawn at the 40% probability level.

Experimental

A mixture of 4-formylbenzoic acid methyl ester (10 mmol), cyclohexanone (20 mmol), malononitrile (30 mmol) and ammonium acetate (10 mmol) was placed in the cavity of a microwave synthesizer. After irradiation at 300 W for 2 min, the reaction mixture was extracted with ethyl acetate (50 ml). The organic phase was separated, dried with anhydrous Na_2SO_4 and evaporated *in vacuo*. The residue was purified by flash column chromatography on silica gel (eluting with hexane–ethyl acetate, 3:1) to give the desired product (yield 50%). Block-shaped crystals of (I) were obtained from an EtOH solution after allowing it to stand for 4 d (m.p. 499–500 K). MS (EI): m/z = 331 $[M]^+$; HRMS: m/z $[M]^+$ calculated for $C_{20}H_{17}N_3O_2$: 331.13; found: 331.13.

Crystal data

$C_{20}H_{17}N_3O_2$	D_x = 1.259 Mg m^{-3}
M_r = 331.37	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7944 reflections
a = 7.9614 (4) Å	θ = 2.6–27.4°
b = 22.054 (1) Å	μ = 0.08 mm^{-1}
c = 10.1707 (6) Å	T = 293 (1) K
β = 101.747 (2)°	Block, colourless
V = 1748.38 (16) Å^3	0.28 × 0.21 × 0.16 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	3666 independent reflections
ω scans	1873 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	R_{int} = 0.039
T_{min} = 0.953, T_{max} = 0.987	θ_{max} = 27.4°
13708 measured reflections	$h = -9 \rightarrow 10$
	$k = -28 \rightarrow 28$
	$l = -13 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2 + 0.3435P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.053	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.135	$\Delta\rho_{max}$ = 0.59 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
1886 reflections	
227 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N3^{iii}	0.86	2.36	3.167 (4)	157
N2—H2B···O2^{ii}	0.86	2.26	3.054 (4)	154

Symmetry codes: (ii) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$ (iii) $-x, -y, -z+1$.

The discrepancy between the number of independent reflections and the number of reflections used in the refinement occurs because the scope of the 2θ refinement was restricted. The H atoms were treated as riding on their parent atoms, with C—H = 0.93 (phenyl) or 0.97 Å (CH_2 and CH_3) and N—H = 0.86 Å, and with U_{iso}(H) = $1.2U_{eq}$(C,N) (aromatic, CH_2, NH_2) or $1.5U_{eq}$(C) (CH_3).

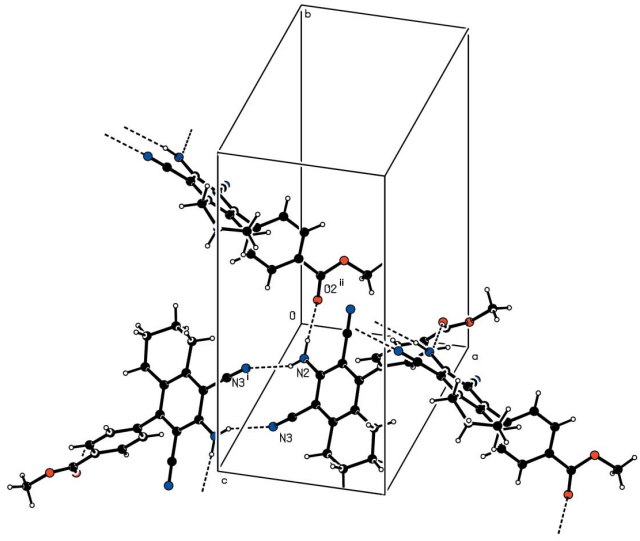


Figure 2
The molecular packing of (I), showing the hydrogen-bonding network. Hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $-x, -y, \frac{1}{2}-z$; (ii) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$.]

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank the Postdoctoral Science Foundation of China for financial support (grant No. 2005037257).

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