Monoclinic
$P2_1/c$
$a = 8.825$ (2) Å
$b = 16.518$ (2) Å
$c = 7.368$ (2) Å
$\beta = 111.39$ (2)°
$V = 1000.1$ (4) Å^3
$Z = 2$
$D_x = 1.701$ Mg m^{-3}
D_m not measured

Cell parameters from 34 reflections
$\theta = 5.24$–12.55°
$\mu = 4.082$ mm^{-1}
$T = 293$ (2) K
Rectangular block
0.48 × 0.20 × 0.18 mm
Yellow

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical ψ scans (Siemens, 1994)
$T_{\rm min} = 0.37$, $T_{\rm max} = 0.48$
3016 measured reflections
2304 independent reflections
1458 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.025$
$\theta_{\rm max} = 27.50°$
$h = -11 \rightarrow 11$
$k = -21 \rightarrow 1$
$l = -1 \rightarrow 9$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.098$
$S = 0.935$
2303 reflections
167 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.293$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.426$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Br—C1	1.895 (3)	O2—C4	1.353 (3)
N—C9	1.458 (4)	C1—C2	1.358 (4)
N—C10	1.471 (4)	C3—C7	1.467 (4)
N—C8	1.473 (3)	C9—C10$^{\rm i}$	1.497 (4)
O1—C7	1.195 (4)		
C9—N—C10	109.7 (2)	C10—N—C8	111.8 (2)
C9—N—C8	112.4 (3)	O1—C7—C3	125.4 (3)

Symmetry code: (i) $-1 - x, -y, -1 - z$.

Program used for data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994); for structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); for structure refinement: *SHELXL93* (Sheldrick, 1993); for geometrical calculations: *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1222). Services for accessing these data are described at the back of the journal.

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5-Amino-2,4-diphenyl-7-(pyrrolidin-1-yl)-1,6-naphthyridine-8-carbonitrile

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Abstract

In the title molecule, $C_{25}H_{21}N_5$, the naphthyridine ring system is almost planar. The phenyl-ring planes form dihedral angles of 28.00 (5) and 84.80 (5)° with the attached ring. Conformational disorder is observed in the pyrrolidine ring and both conformers adopt half-chair conformations. In the solid state, the molecules are linked to form centrosymmetrically-related hydrogen-bonded dimers.

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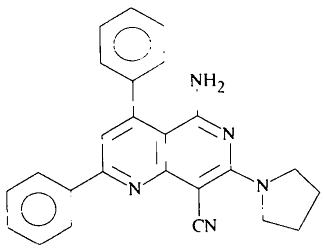
Comment

1,6-Naphthyridine derivatives are known to possess anti-inflammatory, anticonvulsant and insecticidal properties (Damon & Nadelson, 1981, 1982; Takeuchi & Hamada, 1976), but few structural data have been reported in the literature (Balogh *et al.*, 1986; Gomez de Anderez *et al.*, 1992). The crystal structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

The bond lengths observed in the naphthyridine ring system, the average value of the C—C bond lengths in the phenyl rings [1.382 (3) Å] and the N—C distances in the structure are all comparable with previously reported values (Gomez de Anderez *et al.*, 1992; Allen *et al.*, 1987). Due to steric interactions, angles C4—C10—C5 and C8—C7—N14 are widened from 120°. The naphthyridine ring system is nearly planar, with the fused rings forming a dihedral angle of 3.30 (4)°. The phenyl-ring planes form dihedral angles of 28.00 (5) and 84.80 (5)° with the attached pyridine ring. The amino group is twisted from the attached ring by 20 (2)°. Both the major and minor conformers of the pyrrolidine ring adopt a half-chair conformation with asymmetry parameter ΔC_2(N14) equal to 0.036 (1) for the major conformer and 0.006 (2) for the minor conformer (Nardelli, 1983). The plane defined by atoms N14, C15 and C18 of the pyrrolidine ring makes a dihedral of 11.32 (9)° with the least-squares plane through the attached pyridine ring (C5, N6, C7–C10).

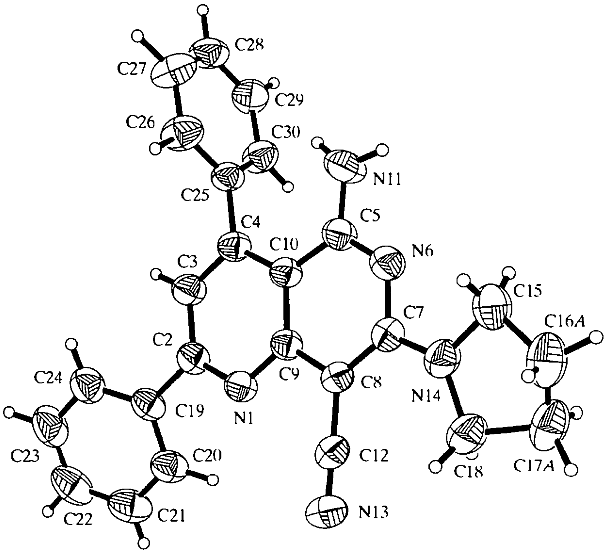


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. For clarity, only the major (60%) conformer of the pyrrolidine ring is shown. H atoms are drawn as spheres of arbitrary radii.

In the crystal, centrosymmetrically-related molecules form dimeric pairs through intermolecular C—H···N hydrogen bonds [C26—H26 0.99 (2), H26···N1^i 2.54 (2), C26···N1^i 3.510 (2) Å and C26—H26···N1^i 166 (2)°; symmetry code: (i) $-x$, $-y$, $-z$].

Experimental

The title compound was synthesized from benzal acetophenone (0.5 g, 2.4 mmol), malononitrile (0.3 g, 4.8 mmol) and a few drops of pyrrolidine in ethanol on refluxing for 18 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography over alumina. Yield: 0.45 g (48%); m.p. 507–509 K (Murugan, 1997). Single crystals were grown by slow evaporation of a methanol solution of the compound.

Crystal data

$C_{25}H_{21}N_5$
M_r = 391.47
Triclinic
$P\bar{1}$
a = 9.0996 (7) Å
b = 10.9985 (9) Å
c = 11.3214 (8) Å
α = 98.956 (7)°
β = 109.026 (6)°
γ = 103.602 (6)°
V = 1007.3 (2) Å^3
Z = 2
D_x = 1.291 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 5.412–12.478°
μ = 0.079 mm^{-1}
T = 293 (2) K
Parallelepiped
0.54 × 0.36 × 0.30 mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5406 measured reflections
4560 independent reflections
2916 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023

$\theta_{\rm max}$ = 27.5°
$h = -1 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 0.938
4560 reflections
358 parameters
H atoms: see below

$\Delta\rho_{\rm max}$ = 0.251 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.158 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.006 (2)

$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2]$
where $P (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.330 (2)	C5—C10	1.447 (2)
N1—C9	1.361 (2)	N6—C7	1.360 (2)
C2—C3	1.399 (2)	C7—C8	1.405 (2)
C3—C4	1.376 (2)	C8—C9	1.427 (2)
C4—C10	1.420 (2)	C9—C10	1.417 (2)
C5—N6	1.323 (2)		
N14—C7—C8	125.02 (13)	C4—C10—C5	126.81 (12)

Atoms C16 and C17 of the pyrrolidine ring were found to be disordered, suggesting conformational disorder. The occupancies of the disordered positions C16*A*, C16*B*, C17*A* and C17*B* were initially refined and later fixed at 60 and 40% for the major (*A*) and minor (*B*) conformations, respectively. The corresponding C—C distances in the major and minor conformers were restrained to be equal, with an effective standard deviation of 0.02 Å. The anisotropic displacement parameters of the disordered positions were also restrained. 17 H atoms were located from a difference Fourier map and refined isotropically; the remaining four (belonging to the disordered atoms C16 and C17) were geometrically fixed and allowed to ride on their attached atoms.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1995).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1213). Services for accessing these data are described at the back of the journal.

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2-(1-Naphthoyl)-5,8-dihydronaphthalen-1-ol†

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(*Received 3 December 1997; accepted 7 January 1998*)

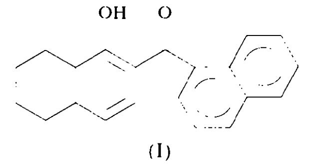
Abstract

In the title molecule, $C_{21}H_{16}O_2$, the naphthalene ring system is planar and the dihydronaphthalene ring is distorted from planarity. The dihydrobenzene ring adopts a flattened-boat conformation. The dihedral angle between the naphthalene and dihydronaphthalene ring systems is 64.13 (5)°. The hydroxyl and carbonyl O atoms are involved in O—H···O intramolecular hydrogen bonding.

Comment

Dihydronaphthalene derivatives generally serve as useful intermediates for the synthesis of several cyclic polymethylene phenols, which are useful antifibrillatory agents, disinfectants and water softeners (Hauck *et al.*, 1977). Furthermore, hydroxyketone derivatives of naphthalene are very useful in synthesizing subunits of daunomycinone and adiramycin, which are important anticancer drugs (Crouse *et al.*, 1981).

Bond lengths and angles in the naphthalene ring system of the title compound, (I), agree with the reported values (Allen *et al.*, 1987). In the dihydronaphthalene moiety, the C_{sp^2}—C_{sp^3} distances C19—C20 [1.493 (3) Å] and C16—C21 [1.507 (3) Å] are longer than the C18—C19 [1.481 (3) Å] and C16—C17 [1.470 (4) Å] distances due to steric interactions. The length of the C17—C18 [1.306 (3) Å] bond shows it to have double-bond character. Similar features are observed in 2-acetyl-5,8-dihydronaphthalen-1-ol (Chinnakali *et al.*, 1998). The naphthalene ring system is planar, with the maximum deviation of 0.027 (2) Å oc-



(I)

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† IUPAC name: 1-hydroxy-5,8-dihydro-2-naphthyl 1-naphthyl ketone.

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