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# *N*(6)-Acetyl-5,7-diphenyl-5,6,7,8-tetrahydrobenzo[*b*]-1,6-naphthyridine

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The single-crystal X-ray diffraction study of the title molecule [alternative name: *N*(2)-acetyl-1,3-diphenyl-1,2,3,4-tetrahydrobenzo[*b*][1,6]naphthyridine], $C_{26}H_{22}N_2O$, confirms that it is a 1,6-naphthyridine derivative whose structural characterization was not possible from $^1$H, $^{13}$C NMR and mass spectral data. The tetrahydro-1,6-napthyridine ring has a boat conformation. The equatorial and axial orientations of the two phenyl groups and the coplanarity of the acetyl group with the tetrahydronaphthyridine ring are also confirmed.

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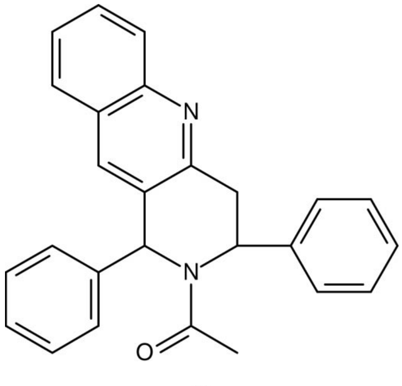
**Key indicators**

Single-crystal X-ray study
$T$ = 293 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.055
$wR$ factor = 0.167
Data-to-parameter ratio = 16.4

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

## Comment

Naphthyridine derivatives have extensive pharmacological properties (Di Braccio *et al.*, 1997; Hong *et al.*, 1997; Chen *et al.*, 1997; Mohan & Mishra, 1997; Damon & Nadelson, 1981; Singh *et al.*, 1995). Structural data for only a few 1,6-naphthyridines are available in the literature (Balogh *et al.*, 1986; Anderez *et al.*, 1992; Govindasamy *et al.*, 2000). The present investigation of *N(*2)-acetyl-1,3-diphenyl-1,2,3,4-tetrahydrobenzo[*b*][1,6]naphthyridine, (I), was undertaken to establish the molecular structure and to determine the precise conformational changes caused by the substituents on the 1,6-naphthyridine ring system in the molecule of the crystalline acetylated product.



(I)

As shown by the X-ray analysis, the tetrahydro-1,6-naphthyridine ring has a boat conformation. The average numerical torsion angles, 43.4 (3) and 44.4 (3)°, involving the four C atoms of the plane of the boat show deviation from the ideal value of 54° (Nasipuri, 1994) due to the presence of planar coordinated N atoms. The C14—C8—C7—C24 and C14*A*—C8*A*—C7*A*—C24*A* torsion angles of −170.8 (2) and 168.8 (2)° show that the phenyl groups attached to C7 and C7*A* are equatorially disposed in the above ring system. The torsion

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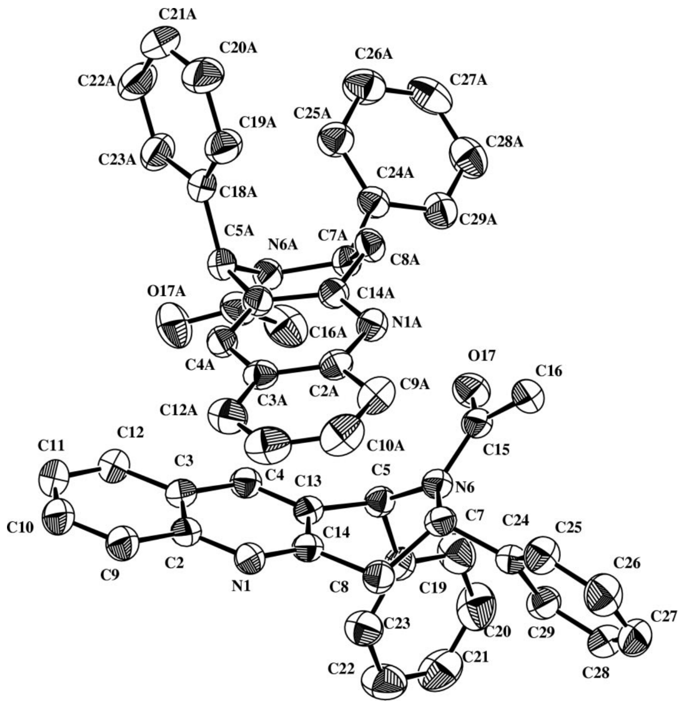

**Figure 1**
View of the title molecule with 50% probability ellipsoids. H atoms have been omitted for clarity.

angles 87.8 (3) and −85.9 (3)°, defined by the atoms C14—C13—C5—C18 and C14*A*—C13*A*—C5*A*—C18*A*, respectively, show that the phenyl groups attached to C5 and C5*A* are axially oriented in the naphthyridine ring. The sum of the bond angles around N6 and N6*A*, 358.7 (2) and 358.8 (2)°, show the near coplanar orientation of the acetyl group with the C5—N6—C7 part of the molecule. The atoms C5, C5*A*, C7 and C7*A* are chiral, their relative configurations being either *SR* or *RS* as revealed by the use of *PLATON*97 (Spek, 1990). The intermolecular and intramolecular C—H···O hydrogen bonds involving the acetyl O and the C atoms of the naphthyridine ring are listed in Table 2.

## Experimental

The title compound was obtained by the acetylation of the corresponding amine with acetic anhydride and triethylamine in benzene under reflux conditions. Diffraction quality crystals were obtained by recrystallizing the crude product from a benzene–petroleum ether mixture. The parent amine itself was obtained by the action of sodium azide with 2,4,6,8-tetraphenyl-3,7-diazabicyclo[3.3.1]nonan-9-one (Sivakumar, 2000), as a non-crystalline product.

### *Crystal data*

$C_{26}H_{22}N_2O$
$M_r = 378.46$
Triclinic, $P\bar{1}$
$a = 10.394$ (2) Å
$b = 10.493$ (3) Å
$c = 20.265$ (4) Å
$\alpha = 76.03$ (2)°
$\beta = 79.83$ (2)°
$\gamma = 72.21$ (2)°
$V = 2029.6$ (8) Å$^3$

$Z = 4$
$D_x = 1.239$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 40 reflections
$\theta = 10$–30°
$\mu = 0.08$ mm$^{-1}$
$T = 293$ (2) K
Rectangular block, colorless
0.05 × 0.05 × 0.04 mm

### *Data collection*

Siemens *P*3 diffractometer
$\omega$–$2\theta$ scans
9786 measured reflections
9279 independent reflections
4446 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\rm max} = 27.7°$

$h = 0 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -25 \rightarrow 26$
3 standard reflections
every 300 reflections
intensity decay: none

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.167$
$S = 0.99$
9279 reflections
566 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.38$ e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0238 (18)

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

| | | | |
|---|---|---|---|
| N1—C14 | 1.309 (3) | N1*A*—C14*A* | 1.319 (3) |
| N1—C2 | 1.368 (3) | N1*A*—C2*A* | 1.368 (3) |
| C5—N6 | 1.483 (3) | C5*A*—N6*A* | 1.487 (3) |
| N6—C15 | 1.365 (3) | N6*A*—C15*A* | 1.362 (3) |
| N6—C7 | 1.481 (3) | N6*A*—C7*A* | 1.479 (3) |
| C14—N1—C2 | 116.5 (2) | C14*A*—N1*A*—C2*A* | 118.2 (2) |
| N1—C2—C3 | 123.4 (2) | N1*A*—C2*A*—C3*A* | 122.3 (2) |
| N6—C5—C13 | 109.67 (19) | N6*A*—C5*A*—C13*A* | 109.29 (19) |
| N6—C5—C18 | 113.21 (19) | N6*A*—C5*A*—C18*A* | 112.41 (19) |
| C15—N6—C7 | 121.4 (2) | C15*A*—N6*A*—C7*A* | 120.9 (2) |
| C15—N6—C5 | 116.6 (2) | C15*A*—N6*A*—C5*A* | 118.2 (2) |
| C7—N6—C5 | 120.71 (18) | C7*A*—N6*A*—C5*A* | 119.67 (18) |
| N6—C7—C24 | 114.60 (19) | N6*A*—C7*A*—C24*A* | 113.91 (19) |
| N6—C7—C8 | 110.8 (2) | N6*A*—C7*A*—C8*A* | 111.00 (19) |
| N1—C14—C13 | 124.6 (2) | N1*A*—C14*A*—C13*A* | 123.6 (2) |
| N1—C14—C8 | 119.3 (2) | N1*A*—C14*A*—C8*A* | 119.9 (2) |
| O17—C15—N6 | 121.1 (2) | O17*A*—C15*A*—N6*A* | 122.1 (3) |
| N6—C15—C16 | 118.8 (2) | N6*A*—C15*A*—C16*A* | 118.7 (2) |
| C13—C5—N6—C7 | 41.7 (3) | C13*A*—C5*A*—N6*A*—C7*A* | −46.4 (3) |
| C5—N6—C7—C8 | 0.1 (3) | C5*A*—N6*A*—C7*A*—C8*A* | 5.4 (3) |
| N6—C7—C8—C14 | −44.3 (3) | N6*A*—C7*A*—C8*A*—C14*A* | 41.8 (3) |
| N6—C5—C13—C14 | −41.4 (3) | N6*A*—C5*A*—C13*A*—C14*A* | 41.4 (3) |
| C7—N6—C15—O17 | −176.4 (2) | C7*A*—N6*A*—C15*A*—O17*A* | 173.7 (2) |
| C5—N6—C15—O17 | −9.1 (3) | C5*A*—N6*A*—C15*A*—O17*A* | 6.5 (4) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5···O17 | 0.98 (2) | 2.21 (2) | 2.685 (3) | 109 |
| C5A—H5*A*···O17*A* | 0.95 (2) | 2.27 (2) | 2.732 (3) | 110 |
| C7*A*—H7*A*···O17 | 1.01 (2) | 2.45 (2) | 3.322 (3) | 145 |
| C9*A*—H9*A*···O17*A*$^{\rm i}$ | 1.04 (2) | 2.37 (2) | 3.325 (3) | 153 |
| C11*A*—H11*A*···O17$^{\rm ii}$ | 0.93 (2) | 2.47 (2) | 3.276 (5) | 145 |

Symmetry codes: (i) $1 + x, y, z$; (ii) $x, 1 + y, z$.

The asymmetric unit of the cell consists of two independent molecules with a total of 58 non-H atoms. All the H atoms were included in calculated positions. While the C—H distances were refined by a riding model, the displacement parameters of the H atoms were tied to common values.

Data collection: *P*3/*P*4 (Siemens, 1989); cell refinement: *P*3/*P*4; data reduction: *P*3/*P*4 and *XDISK* (Siemens, 1989); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983).

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