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# 2,4-Bis(pyridine-2-carbonyl)-1-(2-pyridyl)-3,5-di-*p*-tolylcyclohex-1-ol

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.006 Å
*R* factor = 0.059
*wR* factor = 0.198
Data-to-parameter ratio = 13.5

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



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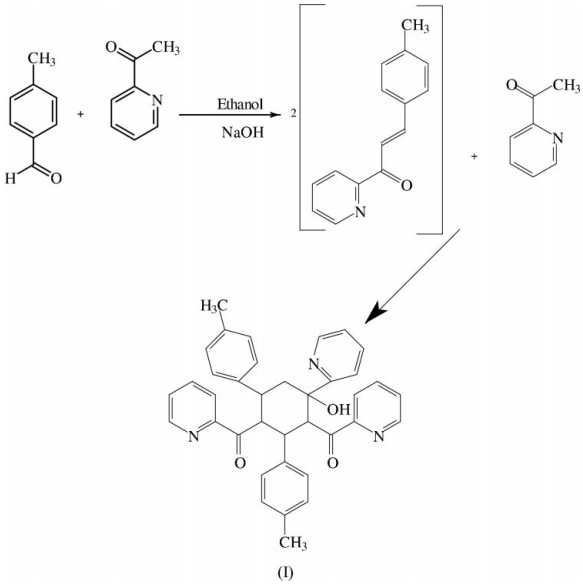
In the title compound, $C_{37}H_{33}N_3O_3$, the central cyclohexane ring adopts a chair conformation. The molecular structure is stabilized by C—H···N, C—H···O, O—H···N and π–π interactions. Interestingly, π–π and C—H···π weak interactions play a major role in the crystal packing.

## Comment

The crystal structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation and hydrogen-bonding characteristics.




(I)

The cyclohexane ring adopts a chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q$ = 0.567 (4) Å, $\theta$ = 1.8 (4)° and $\varphi$ = 353 (8)°. The pyridine-2-carbonyl group attached to C33 and the hydroxy group are in axial orientations, whereas the other substituents are attached to the cyclohexane ring in equatorial positions (Fig. 1). The C12–C17 benzene ring makes dihedral angles of 35.3 (2) and 75.1 (2)°, respectively, with the N1/C18–C22 and N2/C6–C10 pyridine rings. The C24–C29 benzene ring makes dihedral angles of 83.8 (2) and 51.6 (2)°, respectively, with the N1/C18–C22 and N3/C1–C5 pyridine rings. The dihedral angle between the N2/C6–C10 and N3/C1–C5 pyridine rings is 21.5 (2)°. In the cyclohexane ring, the C30—C31 [1.564 (6) Å] and C32—C33 [1.568 (5) Å] bond distances are longer than normal, due to the electron-acceptor capability of the acetylpyridine groups and strain induced by bulky substituents on the cyclohexane ring (Thomson *et al.*, 1995; Vasilyev *et al.*, 1990)

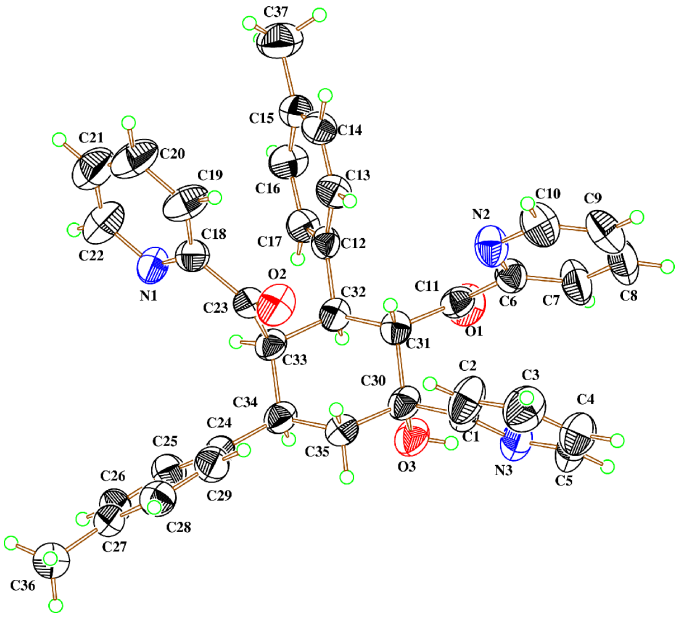



**Figure 1**
A *ZORTEP* (Zsolnai, 1998) plot of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

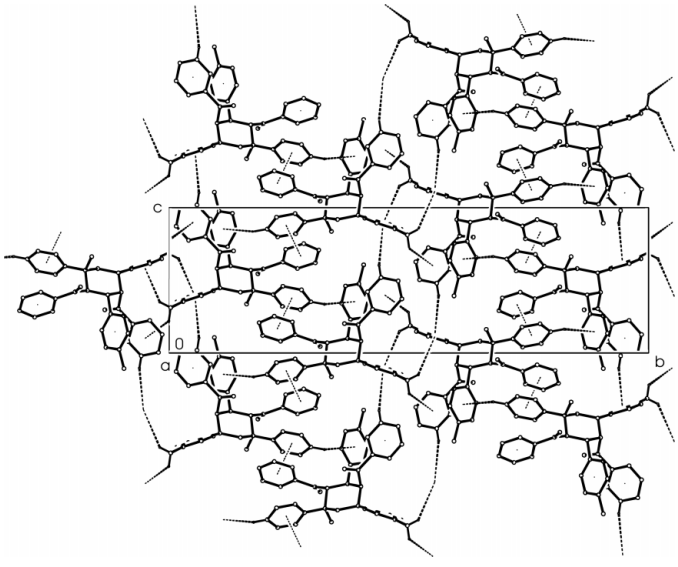



**Figure 2**
Packing of the molecules viewed down the *a* axis. The dashed lines indicate the $\pi$–$\pi$ and C—H$\cdots\pi$ interactions.

The molecular structure is stabilized by intramolecular C—H$\cdots$N, C—H$\cdots$O and O—H$\cdots$N hydrogen bonds (Table 2) and a $\pi$–$\pi$ interaction between the N2/C6–C10 and N3/C1–C5 pyridine rings [centroid–centroid distance = 3.657 (4) Å]. The $\pi$–$\pi$ and weak C—H$\cdots\pi$ interactions (Desiraju, 1989) play a major role in the crystal packing (Fig. 2), in addition to van der Waals forces. The N3/C1–C5 and N2/C6–C10 pyridine rings of the glide-related molecule at $(\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2})$ are stacked with a centroid–centroid separation of 3.898 (4) Å. A C—H$\cdots\pi$ interaction, C4—H4$\cdots$*Cg*1$^{i}$ (*Cg*1 is the C12–C17 ring centroid), is also observed between these two molecules (Table 2). The weak interactions result in the formation of a zigzag chain along the *c* axis (Fig. 2). In the chain, molecules translated by a unit along the *c* axis are linked *via* C20—H20$\cdots$*Cg*2$^{ii}$ (*Cg*2 is the C24–C29 ring centroid) interactions. Adjacent chains are interlinked through C36—H36*C*$\cdots$*Cg*2$^{iii}$ interactions. All symmetry codes are as given in Table 2.

## Experimental

*p*-Tolualdehyde (0.72 g, 5.99 mmol) in ethanol (30 ml) was added dropwise to a stirred solution of NaOH (0.6 g) in water (6 ml). 2-Acetylpyridine (1.089 g, 8.99 mmol) was added dropwise and the solution was stirred at room temperature for 80 h. The precipitate formed during this period was filtered off and washed with water and then with diethyl ether. The crude product was recrystallized from chloroform to give colourless crystals.

*Crystal data*

$C_{37}H_{33}N_3O_3$
$M_r$ = 567.66
Monoclinic, $P2_1/n$
$a$ = 8.534 (7) Å
$b$ = 34.067 (10) Å
$c$ = 10.458 (5) Å
$\beta$ = 98.91 (5)°
$V$ = 3004 (3) Å$^3$
$Z$ = 4
$D_x$ = 1.255 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta$ = 2.1–25.0°
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 (2) K
Needle, colourless
0.41 × 0.15 × 0.12 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$ scans
Absorption correction: none
5638 measured reflections
5267 independent reflections
2164 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017
$\theta_{max}$ = 25.0°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 40$
$l = -12 \rightarrow 12$
3 standard reflections
every 100 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.198
$S$ = 0.94
5267 reflections
391 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1021P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| C1—N3 | 1.338 (5) | C18—N1 | 1.338 (5) |
| C5—N3 | 1.328 (6) | C22—N1 | 1.331 (5) |
| C6—N2 | 1.334 (5) | C23—O2 | 1.211 (5) |
| C6—C11 | 1.499 (6) | C30—O3 | 1.423 (4) |
| C11—O1 | 1.221 (5) | | |
| N3—C1—C30 | 115.3 (4) | C13—C12—C32 | 123.3 (4) |
| C2—C1—C30 | 123.9 (4) | N1—C18—C19 | 123.4 (4) |
| N3—C5—C4 | 124.8 (5) | N1—C18—C23 | 117.7 (4) |
| N2—C6—C7 | 122.6 (4) | N1—C22—C21 | 124.1 (5) |
| N2—C6—C11 | 116.3 (4) | O2—C23—C33 | 122.7 (4) |
| N2—C10—C9 | 124.5 (5) | C18—C23—C33 | 118.4 (4) |
| O1—C11—C31 | 122.5 (4) | C29—C24—C25 | 116.4 (4) |
| C6—C11—C31 | 117.7 (4) | C29—C24—C34 | 123.8 (4) |
| C17—C12—C13 | 116.6 (4) | | |
| C35—C30—C31—C32 | −56.4 (4) | C32—C33—C34—C35 | 52.7 (4) |
| C30—C31—C32—C33 | 54.7 (4) | C33—C34—C35—C30 | −56.4 (4) |
| C31—C32—C33—C34 | −52.4 (4) | C31—C30—C35—C34 | 56.7 (4) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3$A$···N3 | 0.82 | 2.10 | 2.586 (5) | 118 |
| C31—H31···N2 | 0.98 | 2.43 | 2.887 (5) | 108 |
| C33—H33···N1 | 0.98 | 2.38 | 2.890 (6) | 112 |
| C35—H35$B$···O2 | 0.97 | 2.46 | 3.070 (6) | 121 |
| C4—H4···$Cg1^{i}$ | 0.93 | 3.04 | 3.805 (6) | 141 |
| C20—H20···$Cg2^{ii}$ | 0.93 | 3.04 | 3.824 (6) | 143 |
| C36—H36$C$···$Cg2^{iii}$ | 0.96 | 2.94 | 3.685 (6) | 135 |

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $x, y, 1+z$; (iii) $2-x, -y, 1-z$. $Cg1$ is the C12–C17 ring centroid and $Cg2$ is the C24–C29 ring centroid

The high proportion of unobserved data may be a result of the high mosaicity of the crystal. All the H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.2 or 1.5 times $U_{eq}$(parent atom).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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