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ChinaCorrespondence e-mail:
jxyang@icm.sdu.edu.cn**Key indicators**

Single-crystal X-ray study

 $T = 293$ KMean $\sigma(C-C) = 0.004$ Å R factor = 0.051 wR factor = 0.156

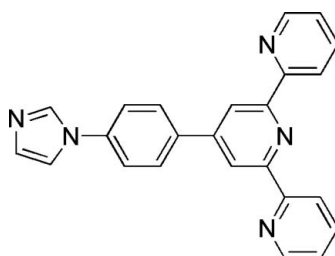
Data-to-parameter ratio = 13.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**4'-[4-(Imidazol-1-yl)phenyl]-2,2':6',2''-
terpyridine (IPTP)**

The title compound (IPTP), $C_{24}H_{17}N_5$, has been synthesized and characterized by 1H NMR, ^{13}C NMR and single-crystal X-ray diffraction. The three pyridyl rings are almost coplanar. The benzene ring forms dihedral angles of 49.59 (17) and 19.18 (13)° with the central pyridyl ring and the imidazole ring, respectively.

Comment

Polypyridine ligands have played an important role in many areas. In particular, the chelating ligand terpyridine (terpy) and its functionalized derivatives have been studied extensively as outstanding complexing agents for a wide range of transition metal ions (Heller & Schubert, 2003). This property has been widely used in analytical chemistry (Schubert & Eschbaumer, 2002), in photochemistry for the design of luminescent devices (Harriman & Ziesel, 1998) and in biochemistry (Trawick *et al.*, 1998). Although the number of publications or investigations of terpyridine complexes has increased enormously, comparably few preparations of functionalized terpyridine derivatives have been reported as yet. The title compound, IPTP or (I), is a novel compound containing terpyridine and imidazole coordination sites. We report here the synthesis, characterization and crystal structure of (I).



(I)

The molecular structure of the title compound was determined by single-crystal X-ray diffraction and is shown in Fig. 1. A packing diagram is shown in Fig. 2. Bond lengths and angles in the compound are given in Table 1. The three pyridyl rings are almost co-planar, with interplanar angles of 7.15 (4) (between ring N5/C13–C17 and the central pyridyl ring), 8.38 (15) (between rings N5/C13–C17 and N4/C20–C24) and 11.67 (14)° (between ring N4/C20–C24 ring and the central pyridyl ring). The benzene ring forms dihedral angles of 49.59 (17) and 19.18 (13)°, respectively, with the central pyridyl ring and the imidazole ring.

Experimental

For the preparation of 3-(4-imidazole-1-ylphenyl)-1-(pyridin-2-yl)prop-2-en-1-one, a flask charged with a mixture of 4-imidazole-1-

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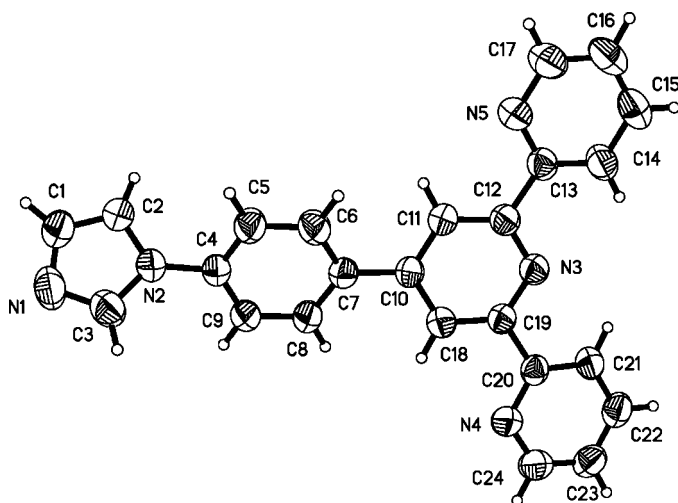


Figure 1
The molecular structure of IPTP, showing 50% probability displacement ellipsoids.

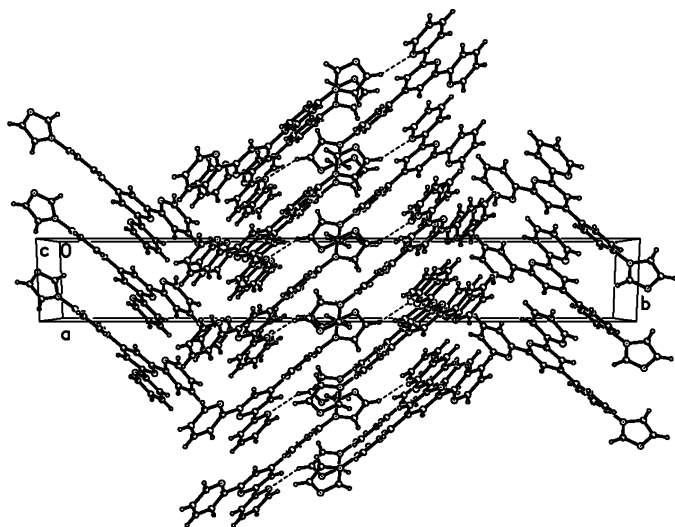


Figure 2
A packing diagram of IPTP. Hydrogen bonds are shown as dashed lines.

benzaldehyde (8.6 g, 50 mmol), 2-acetylpyridine (6.1 g, 50 mmol) and 2% aqueous sodium hydroxide (150 ml) was stirred vigorously at room temperature for 30 min, and was then heated to about 333 K for 6 h. The reaction was monitored by thin-layer chromatography. After the reaction was complete, the reaction mixture was cooled to room temperature. A light-yellow solid was precipitated which was then filtered, washed thoroughly with water and air-dried to give 13.6 g of 3-(4-imidazole-1-ylphenyl)-1-(pyridin-2-yl)prop-2-en-1-one (yield 98.0%).

For the preparation of IPTP, 2-acetylpyridine (1.8 g, 15 mmol) and 3-(4-imidazole-1-ylphenyl)-1-(pyridin-2-yl)prop-2-en-1-one (4.1 g, 15 mmol) and NaOH (2.4 g, 60 mmol) powder were crushed together with a pestle and mortar for 2 h. The yellow powder was added to a stirred solution of ammonium acetate (10 g, excess) in ethanol (100 ml). The reaction mixture was heated at reflux for 10 h. Upon cooling to room temperature, the precipitate was filtered, washed with water three times and dried to afford the product. Recrystallization from ethanol afforded pale-yellow plate-shaped crystals. ^1H NMR (CDCl_3): δ 7.27 (*t*, 1H), 7.37 (*m*, 3H), 7.55 (*d*, 2H), 7.90 (*m*, 2H),

7.98 (*s*, 1H), 8.04 (*d*, 2H), 8.67 (*s*, 1H), 8.70 (*s*, 1H), 8.74 (*m*, 2H), 8.76 (*s*, 2H). ^{13}C NMR (CDCl_3): δ 117.7, 118.2, 120.9, 121.2, 123.5, 128.5, 129.9, 134.9, 136.3, 136.5, 148.5, 148.7, 155.3, 155.5, 155.7.

Crystal data

$\text{C}_{24}\text{H}_{17}\text{N}_5$
 $M_r = 375.43$
Monoclinic, $P2_1/n$
 $a = 5.4807$ (5) Å
 $b = 38.653$ (5) Å
 $c = 8.953$ (1) Å
 $\beta = 103.470$ (9)°
 $V = 1844.5$ (4) Å³
 $Z = 4$

$D_x = 1.352$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 41 reflections
 $\theta = 5.2\text{--}12.5^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
Plate, pale yellow
 $0.45 \times 0.34 \times 0.10$ mm

Data collection

Bruker *P4* diffractometer
 ω scans
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.918$, $T_{\max} = 0.992$
4935 measured reflections
3625 independent reflections
1574 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -6 \rightarrow 1$
 $k = -1 \rightarrow 47$
 $l = -11 \rightarrow 11$
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.156$
 $S = 0.93$
3625 reflections
263 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³
Extinction correction: *SHELXTL*
Extinction coefficient: 0.025 (2)

Table 1

Selected geometric parameters (Å, °).

C1–N1	1.343 (4)	C12–N3	1.345 (3)
C2–N2	1.370 (4)	C13–N5	1.338 (3)
C3–N1	1.314 (4)	C17–N5	1.333 (3)
C3–N2	1.342 (3)	C19–N3	1.339 (3)
C4–N2	1.425 (3)	C20–N4	1.336 (3)
C7–C10	1.484 (4)	C24–N4	1.331 (3)
C3–N1–C1	104.3 (3)	C19–N3–C12	117.5 (2)
C3–N2–C2	104.9 (3)	C24–N4–C20	116.5 (3)
C3–N2–C4	128.6 (3)	C17–N5–C13	118.0 (3)
C2–N2–C4	126.4 (2)		

All H atoms were positioned geometrically and allowed to ride on their attached atoms, with $\text{C–H} = 0.93$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

- Bruker (1996). *XSCANS User's Manual*. Version 2.2. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Harriman, A. & Ziessel, R. (1998). *Coord. Chem. Rev.* **171**, 331–339.

Heller, M. & Schubert, U. S. (2003). *Eur. J. Org. Chem.*, 947–961.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Schubert, U. S. & Eschbaumer, C. (2002). *Angew. Chem. Int. Ed.* **41**, 2892–2926.

Trawick, B. N., Daniher, A. T. & Bashkin, J. K. (1998). *Chem. Rev.* **98**, 939–960.