

4'-Vinyl-2,2':6',2''-terpyridine

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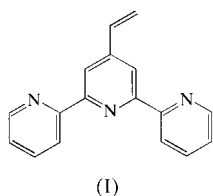
Received 12 May 2000

Accepted 8 June 2000

The title compound, $C_{17}H_{13}N_3$, is a versatile precursor for polymeric terpyridine derivatives and their metal complexes. The molecule has *transoid* and near-coplanar pyridine rings. However, the vinyl group is forced out of the plane of the terpyridyl moiety by a close H...H contact.

Comment

4'-Vinyl-2,2':6',2''-terpyridine (Potts & Konwar, 1991) is a versatile synthetic intermediate, which undergoes homo- or copolymerization reactions under free-radical or electrochemical conditions. The metal-containing polymers obtained in this way have potentially interesting photophysical or electrocatalytic properties (Potts *et al.*, 1987; Guadalupe *et al.*, 1988; Potts & Usifer, 1988; Hurrell *et al.*, 1989). We have obtained this compound, (I), as a synthetic intermediate during our studies of complexes of substituted meridional trisimine ligands (Solanki *et al.*, 1998, 1999), and now report its crystal structure (Fig. 1).



As with other 2,2':6',2''-terpyridine derivatives (Constable *et al.*, 1990, 1995; Constable, Cargill Thompson *et al.*, 1992; Constable, Kahn *et al.*, 1992; Bessel *et al.*, 1992; Fallahpour *et al.*, 1999), the three pyridine rings are *transoid* to each other and are nearly coplanar, the dihedral angles between the least-squares planes of the three rings being N1–C6/C7–C12 = 7.1 (2)° and N1–C6/C13–C18 = 2.2 (2)°. The torsion angles about the C4–C19 bond are C5–C4–C19–C20 = –155.28 (17)° and C3–C4–C19–C20 = 24.4 (3)°, so that the vinyl substituent is not coplanar with the central pyridine ring. This can be attributed to the effect of steric repulsions between the H atoms H3 and H20B, which lie only 2.35 Å apart.

In the lattice, the molecules are arranged into π – π -stacked pairs related by inversion (symmetry code: $-x, 1 - y, -z$).

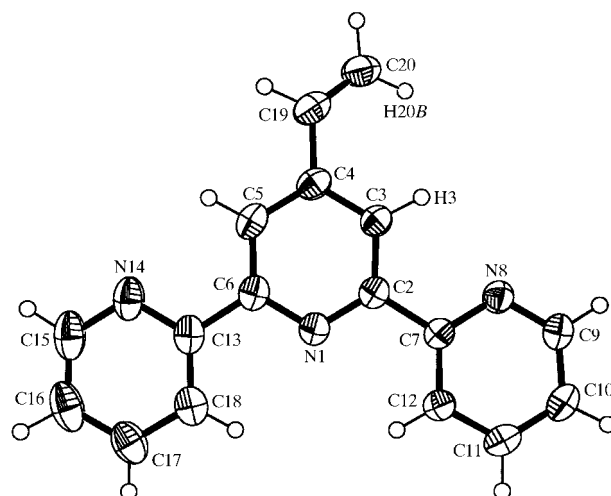


Figure 1

Molecular structure showing 50% probability displacement ellipsoids and the atom-numbering scheme employed. H atoms have arbitrary radii and those referred to in the text are labelled.

This leads to a π – π interaction between pyridine rings N1–C6 and C7–C12 of one molecule, and N1'–C6' and C7'–C12' of a neighbouring molecule. The average interplanar spacing for this interaction is 3.42 Å, the dihedral angle between the least-squares planes of the two rings is 7.1 (2)° and the offset of their centroids is 2.15 Å (Hunter & Sanders, 1990). These pairs of molecules are in turn arranged translationally into one-dimensional chains parallel to the *a* axis, leading to a second π – π interaction between C7–C12 and C7''–C12'' of molecules related by another inversion centre ($1 - x, 1 - y, -z$). These rings are therefore strictly coplanar by symmetry, their interplanar spacing being 3.42 Å while their centroids are offset by 1.43 (2) Å. Adjacent π – π chains are related by the operation of the *c*-glide ($-\frac{1}{2} - x, -\frac{1}{2} + y, z$) and are rotated by 61.9 (2)° with respect to each other.

Experimental

The compound was isolated in moderate yield from the reaction of 4'-(2-hydroxyethyl)-2,2':6',2''-terpyridine (Potts *et al.*, 1987) with SeO_2 in refluxing dioxane. The NMR spectra of the resultant white solid matched those previously reported for this compound (Potts *et al.*, 1987). Crystals were grown from ether:hexane (1/1).

Crystal data

$C_{17}H_{13}N_3$
 $M_r = 259.30$
Orthorhombic, *Pbca*
 $a = 7.9476$ (2) Å
 $b = 11.5546$ (3) Å
 $c = 29.4257$ (8) Å
 $V = 2702.20$ (12) Å³
 $Z = 8$
 $D_x = 1.275$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 14681 reflections
 $\theta = 2.77$ – 27.49°
 $\mu = 0.078$ mm⁻¹
 $T = 150$ (2) K
Plate, colourless
 $0.67 \times 0.50 \times 0.25$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
Area-detector scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{min} = 0.950$, $T_{max} = 0.981$
14681 measured reflections

3093 independent reflections
2246 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.074$
 $\theta_{max} = 27.49^\circ$
 $h = -10 \rightarrow 10$
 $k = -14 \rightarrow 15$
 $l = -38 \rightarrow 38$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.147$
 $S = 1.044$
 3093 reflections
 233 parameters
 All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0781P)^2 + 0.3186P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$

All H atoms were located in a Fourier difference map, and were allowed to refine freely. The C—H bond lengths are in the range 0.951 (16)–1.03 (2) Å.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1996); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (McArdle, 1995); software used to prepare material for publication: local program.

The authors acknowledge the Royal Society of London for a University Research Fellowship to MAH, and the EPSRC for a postdoctoral fellowship to XL and for the purchase of a diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1415). Services for accessing these data are described at the back of the journal.

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