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Key indicators

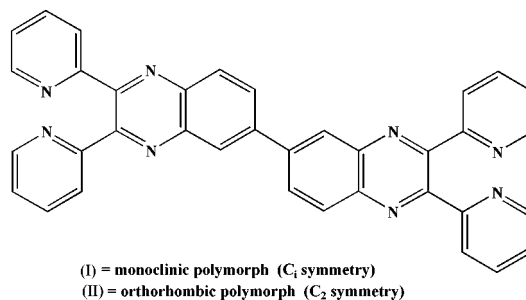
Single-crystal X-ray study
 $T = 223$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.036
 wR factor = 0.087
Data-to-parameter ratio = 7.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.An orthorhombic polymorph of 2,3,2',3'-
tetrakis(2-pyridyl)-6,6'-biquinoxalinyll

When recrystallized from a water–methanol solution, the title compound, $\text{C}_{36}\text{H}_{22}\text{N}_8$, gave orthorhombic crystals in the non-centrosymmetric space group $Fdd2$. The molecules are located on twofold rotation axes. The dihedral angle between the approximately planar quinoxaline moieties is $32.93(4)^\circ$. The two pyridine substituents lie out of the plane of the quinoxaline moiety by $56.6(2)$ and $33.5(2)^\circ$, and are inclined to one another by $50.2(2)^\circ$. Their N atoms are directed on opposite sides of the mean plane through the quinoxaline moiety.

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Comment

The title compound was first synthesized by Rillema *et al.* (1982). When recrystallized from ethanol, a monoclinic polymorph, (I), was obtained, possessing C_i symmetry (Neels & Stoeckli-Evans, 2003). When recrystallized from a water–methanol solution, the orthorhombic polymorph, (II), was obtained (Fig. 1). The bond lengths and angles are normal within experimental error, and are very similar to those observed for the monoclinic polymorph, (I).



The molecule of (II) possesses C_2 symmetry and, in contrast to (I), where the quinoxaline moieties are coplanar, in (II) they are inclined to one another by $32.93(4)^\circ$. A side view of the molecule is shown in Fig. 2. The largest deviation of any

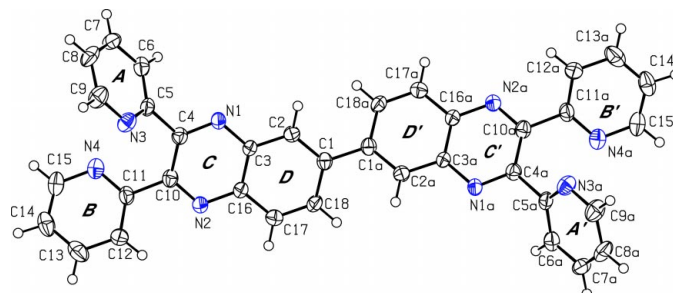


Figure 1

A perspective view of the molecular structure of (II), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

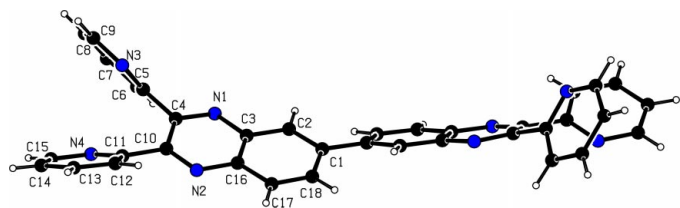


Figure 2
A side view of the molecular structure of (II).

atom from the mean plane describing the quinoxaline moiety is 0.062 (4) Å. Pyridine rings *A* and *B* are inclined to the mean plane through the quinoxaline system by 56.6 (2) and 33.5 (2)°, respectively. In (I), however, the corresponding angles are 40.1 (1) and 42.3 (1)°, respectively. The dihedral angle between the pyridine rings themselves is 50.2 (2)°, compared with 56.8 (2)° in (I). As in (I), the N atoms face one another but are directed on opposite sides of the quinoxaline moiety. The pyrazine ring is much less distorted than that in (I), the N1–C4–C10–N2 and N1–C3–C16–N2 torsion angles being 4.8 (5) and 2.9 (5)°, respectively, compared with 9.8 (4)° and 8.0 (4)° in (I).

In the crystal structure of (II), there is one intramolecular hydrogen bond (Table 1) but no intermolecular hydrogen bonds [whereas intermolecular hydrogen bonds are present in (I)] and no significantly short (< 3.2 Å) non-bonded interactions between symmetry-related molecules (Fig. 3).

Experimental

The title compound was synthesized according to the procedure described by Rillema *et al.* (1982). Small orange plate-like crystals, suitable for X-ray diffraction, were obtained by slow evaporation of a methanol–water mixture.

Crystal data

$C_{36}H_{22}N_8$	Mo $K\alpha$ radiation
$M_r = 566.62$	Cell parameters from 3165 reflections
Orthorhombic, <i>Fdd2</i>	$\theta = 2.3$ – 25.9°
$a = 21.0806$ (19) Å	$\mu = 0.09$ mm ⁻¹
$b = 20.338$ (2) Å	$T = 223$ (2) K
$c = 12.8637$ (13) Å	Plate, orange
$V = 5515.1$ (9) Å ³	0.50 × 0.40 × 0.10 mm
$Z = 8$	
$D_x = 1.365$ Mg m ⁻³	

Data collection

Stoe IPDS diffractometer	$R_{int} = 0.107$
φ scans	$\theta_{max} = 25.9^\circ$
10 285 measured reflections	$h = -25 \rightarrow 25$
1396 independent reflections	$k = -24 \rightarrow 24$
775 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 15$

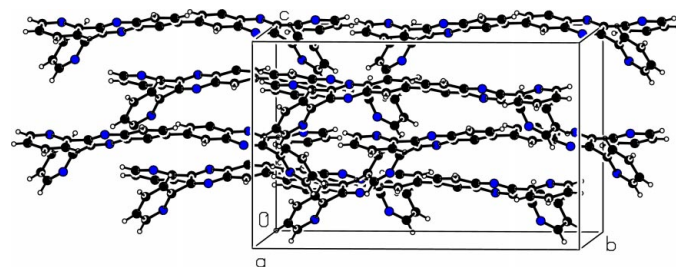


Figure 3
The molecular arrangement in the unit cell, viewed approximately down the *a* axis (H atoms have been omitted for clarity).

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2]$
$wR(F^2) = 0.087$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.70$	$(\Delta/\sigma)_{max} < 0.001$
1396 reflections	$\Delta\rho_{max} = 0.19$ e Å ⁻³
199 parameters	$\Delta\rho_{min} = -0.22$ e Å ⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C12–H12···N2	0.94	2.60	2.851 (5)	95

The crystals were thin plates and diffracted weakly. Only 56% of the data can be considered to be observed [$I > 2\sigma(I)$], which resulted in a relatively high R_{int} value and a low S value. The Friedel pairs were merged, as there was no significant anomalous dispersion. H atoms were introduced in calculated positions and treated as riding atoms, with C–H = 0.94 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *EXPOSE* (Stoe & Cie, 2000); cell refinement: *CELL* (Stoe & Cie, 2000); data reduction: *INTEGRATE* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON99* (Spek, 1990); software used to prepare material for publication: *SHELXL97*.

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