

## 2,2',3,3'-Tetramethyl-6,6'-biquinoxaline

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## Key indicators

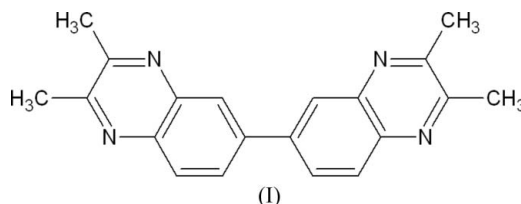
Single-crystal X-ray study  
T = 100 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
R factor = 0.039  
wR factor = 0.111  
Data-to-parameter ratio = 9.7

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

The molecule of the title compound,  $\text{C}_{20}\text{H}_{18}\text{N}_4$ , has twofold rotational symmetry. Each aromatic ring of the fused quinoxaline ring system is skewed from the other by  $4.93 (7)^\circ$ . The two quinoxaline units make a dihedral angle of  $43.52 (2)^\circ$ .

## Comment

Quinoxalines and their derivatives have received considerable attention over the past several years due to their electronic properties (Page *et al.*, 1998; Simpson & Gordon, 1995), hydrogen-bonding ability (Pascal & Ho, 1993; Wozniak *et al.*, 1993) and their capacity to coordinate to metals (Wu *et al.*, 2002; Willett *et al.*, 2001). During our investigations, we have prepared a number of substituted quinoxalines which readily coordinate to copper iodide, forming novel structures. Our current work involves the synthesis of new nitrogen heterocycles (Gibson *et al.*, 2006) which may lead to novel three-dimensional structures upon coordination to cuprous salts. We report here the crystal structure of 2,2',3,3'-tetramethyl-6,6'-biquinoxaline, (I) (Fig. 1).



The structure of (I) exhibits bond distances and angles that are unexceptional, all falling within ranges established in the literature for similar nitrogen heterocycles (Brown *et al.*, 2004). Only one half of the molecule is crystallographically independent. The two halves are related by a twofold rotation axis. There are two other interesting features in this structure. The first is that the quinoxaline units themselves are not perfectly planar. Each aromatic ring of the fused quinoxaline ring system is skewed from the other by  $4.93 (7)^\circ$  (based on least-squares planes calculation of atoms C1–C4/C7/C8 and C4,–C7/N1/N2). The other interesting feature is that the quinoxaline ring systems are not coplanar. This is possibly the result of van der Waals repulsion between adjacent H atoms ( $\text{H}2 \cdots \text{H}2^i$  and  $\text{H}8 \cdots \text{H}8^i$ ); thus, the C2–C2<sup>i</sup> bond distance is  $3.071 (2) \text{ \AA}$  and the C8–C8<sup>i</sup> distance is  $3.001 (2) \text{ \AA}$  [symmetry code: (i)  $2 - x, y, -z + \frac{1}{2}$ ]. The dihedral angle between the planes of the two quinoxaline units is  $43.52 (2)^\circ$ .

## Experimental

A 20 ml test tube was charged with 3,3'-diaminobenzidine (214 mg, 1 mmol) and 2,3-butanedione (172 mg, 2 mmol). This was heated in a

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boiling water bath for 1 h, until the reaction mixture was homogeneous. The residue was then dissolved in boiling ethanol (100% EtOH, 15 ml). Upon cooling to 273 K, colourless crystals of (I) were obtained (280 mg, 89% yield; m.p. 488–489 K).

Crystal data

$C_{20}H_{18}N_4$	$Z = 4$
$M_r = 314.38$	$D_x = 1.354 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Cu $K\alpha$ radiation
$a = 12.0225 (6) \text{ \AA}$	$\mu = 0.65 \text{ mm}^{-1}$
$b = 6.7301 (3) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 19.1118 (10) \text{ \AA}$	Plate, colourless
$\beta = 93.966 (3)^\circ$	$0.15 \times 0.15 \times 0.04 \text{ mm}$
$V = 1542.68 (13) \text{ \AA}^3$	

Data collection

Bruker SMART APEX-II CCD diffractometer	8311 measured reflections
$\omega$ and $\varphi$ scans	1410 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	1148 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.91, T_{\max} = 0.98$	$R_{\text{int}} = 0.050$
	$\theta_{\max} = 69.0^\circ$

Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + 0.9428P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.19 \text{ e \AA}^{-3}$
1410 reflections	$\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$
146 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0010 (2)

The aromatic and methyl H atoms were placed in idealized positions ( $C-H = 0.96-1.00 \text{ \AA}$ ) and allowed to ride on their parent atoms, with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ .

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: XSHHELL (Bruker, 2004); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

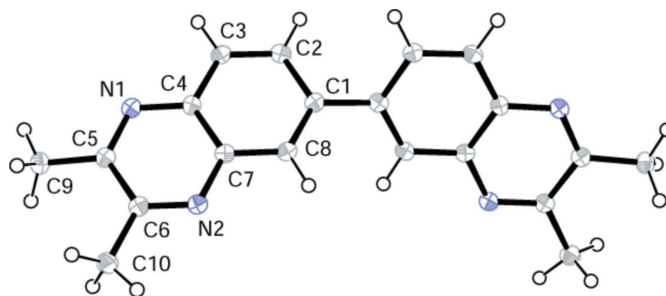


Figure 1 The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by  $(2 - x, y, -z + \frac{1}{2})$ .

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