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Crystal Structure
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N-[(2-Phenyl-2*H*-1,2,3-triazol-4-yl)methylene]-2-(2-(2-phenyl-2*H*-1,2,3-triazol-4-yl)-3-{2-[(2-phenyl-2*H*-1,2,3-triazol-4-yl)methyleneamino]ethyl}imidazolidin-1-yl)ethanamine

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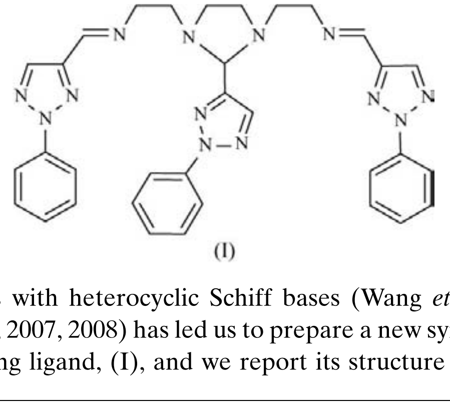
The reaction of 2-phenyl-2*H*-1,2,3-triazole-4-carbaldehyde with triethylenetetramine leads to the formation of a new binucleating ligand, *viz.* the title compound, $C_{33}H_{33}N_{13}$, demonstrating that this structure has the potential for more flexible rational design and tailoring. The title molecule is rendered quite rigid by the formation of a five-membered imidazolidine ring and there are four independent instances of π–π interactions. Both sides of each of the three aromatic arms take part in these interactions, forming a neat three-dimensional array structure.

Comment

The field of binucleating ligands and their metal complexes has received considerable attention in recent years because of their importance in bioinorganic chemistry, molecular magnetism and catalysis (Nanda *et al.*, 2006; Fondo *et al.*, 2005). In the past decade, nitrogenous heterocyclic Schiff bases have gained more and more recognition because they may act as polydentate ligands and adopt a variety of coordination modes (Drabent *et al.*, 2004). Our interest in metal complexes with heterocyclic Schiff bases (Wang *et al.*, 2001; Feng *et al.*, 2007, 2008) has led us to prepare a new symmetrical binucleating ligand, (I), and we report its structure here.



(I)

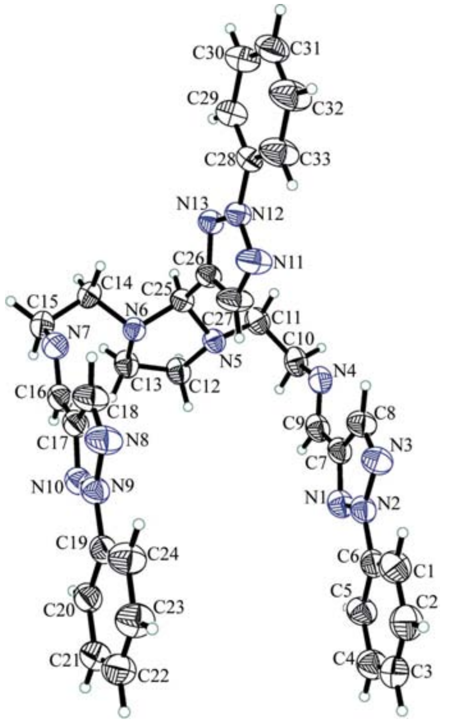


Figure 1
A view of the molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

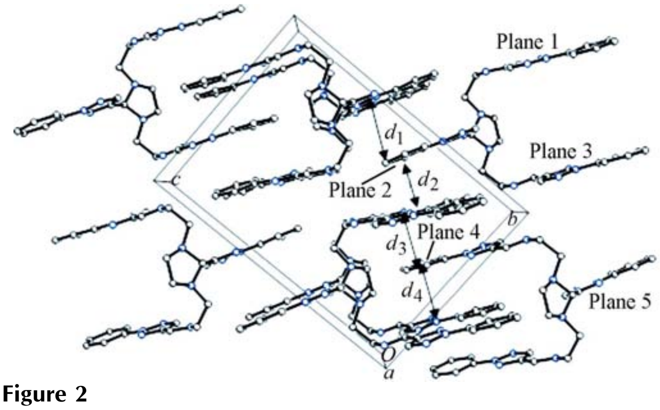


Figure 2
A perspective view, showing the three-dimensional supramolecular array in (I) and the intermolecular π–π stacking interactions. (See *Comment* for values of *d*.)

A view of (I) is shown in Fig. 1, and selected bond lengths and angles are listed in Table 1. The molecular skeleton contains an imidazolidine ring that adopts an envelope conformation. The N4—C10—C11—N5 and N6—C14—C15—N7 fragments both adopt *gauche* conformations [−65.0 (2) and 65.9 (2)°, respectively]. The C1–C8/N1–N3 and C17–C24/N8–N10 ring systems are nearly coplanar, the dihedral angle between them being 5.90°. Overall, the molecular structure resembles a 'Y'. The C9═N4 and C16═N7 distances (Table 1) correspond to formal C═N double bonds (Yang *et al.*, 1995). The C—C and C—N distances within the saturated imidazolidine ring (N5/C12/C13/N6/C25) are in the range 1.452 (2)–1.492 (3) Å. A similar behaviour is found for $[Ag(\mu_3\text{-hmt})(p\text{-nba})]\cdot 2.5H_2O$ (1.457–1.496 Å; hmt is hexamethylenetetramine and *p*-nba is 4-nitrobenzoate; Mukhopadhyay *et al.*, 2001).

No classical hydrogen bonding is observed in the crystal structure of (I). However, there exist strong intermolecular aromatic π–π interactions between adjacent molecules, resulting in a highly ordered packing, as shown in Fig. 2. Both sides of each of the three aromatic arms take part in these interactions, forming a very neat (and no doubt favourable) three-dimensional array structure. There are four independent instances of π–π interactions in this novel structure. The face-to-face distances between adjacent (exactly) parallel aromatic surfaces (d_1 and d_4) are 3.85 (1) and 3.41 (8) Å. The interactions d_2 and d_3 are between nonparallel planes. The maximum and minimum distances between interacting atoms from planes 2 and 3 are 3.942 (2) and 3.398 (3) Å. The average value is 3.68 Å. The corresponding values from atoms of planes 3 and 4 are 3.842 (3), 3.335 (2) and 3.54 Å, respectively, indicating significant π–π stacking interaction.

Experimental

The title compound was synthesized by refluxing 2-phenyl-2*H*-1,2,3-triazole-4-carbaldehyde (3 mmol) and triethylenetetramine (1 mmol) in ethanol (40 ml) in the presence of glacial acetic acid (2 ml). The mixture was refluxed for 4 h under magnetic stirring. After cooling to room temperature, a white powdery product was obtained (yield 85%, m.p. 381.0–381.4 K). The colourless filtrate was allowed to evaporate slowly, affording X-ray quality crystals of the title compound. Analysis calculated for $C_{33}H_{33}N_{13}$: C 64.80, H 5.44, N 29.77%; found: C 64.92, H 5.47, N 29.93%. IR (KBr pellet, cm^{-1}): 3443 (*b*), 2922 (*s*), 2882 (*s*), 2826 (*s*), 1654 (*vs*), 1595 (*vs*), 1497 (*s*), 1346 (*s*), 752 (*s*).

Crystal data

$C_{33}H_{33}N_{13}$
$M_r = 611.72$
Triclinic, $P\bar{1}$
$a = 7.7473$ (16) Å
$b = 12.350$ (3) Å
$c = 17.211$ (4) Å
$\alpha = 87.487$ (5)°
$\beta = 82.227$ (5)°
$\gamma = 80.247$ (5)°
$V = 1607.7$ (6) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
0.42 × 0.35 × 0.14 mm

Data collection

Rigaku R-AXIS SPIDER diffractometer
Absorption correction: empirical (using intensity measurements) (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.967$, $T_{\max} = 0.989$
15461 measured reflections
7203 independent reflections
3365 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.186$
$S = 1.02$
7203 reflections
416 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N4—C9	1.251 (3)	N6—C13	1.469 (2)
N5—C25	1.452 (2)	N7—C16	1.255 (3)
N5—C12	1.458 (2)	C12—C13	1.492 (3)
N6—C25	1.457 (2)	C25—C26	1.488 (3)
C9—N4—C10	118.7 (2)	C16—N7—C15	119.08 (19)
C11—N5—C12	114.60 (15)	N6—C13—C12	104.87 (16)
C14—N6—C13	114.57 (16)	N5—C25—N6	102.03 (14)
C25—N6—C13	105.04 (15)		
N4—C10—C11—N5	−65.0 (2)	C13—N6—C25—N5	−39.93 (19)
C25—N5—C12—C13	−24.7 (2)	N6—C25—C26—N13	−112.1 (2)
N6—C14—C15—N7	65.9 (2)	N5—C25—C26—C27	−46.4 (3)

H atoms were positioned geometrically, with C—H distances of 0.95–0.98 Å, and treated as riding, with $U_{\rm iso}$(H) values of $1.2U_{\rm eq}$(C).

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF3079). Services for accessing these data are described at the back of the journal.

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