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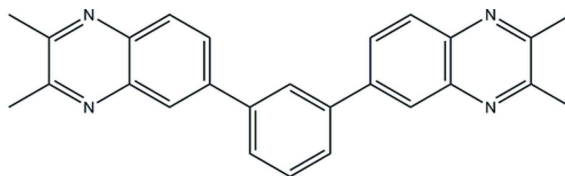
# Crystal structure of 1,3-bis(2,3-dimethylquinoxalin-6-yl)benzene

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The title compound, C<sub>26</sub>H<sub>22</sub>N<sub>4</sub> (I), was synthesized by C–H iridium-catalyzed borylation followed by Suzuki coupling. The molecular structure of (I) consists of a central benzene ring with 3-dimethylquinoxalin-6-yl groups at the 1 and 3 positions. These 2,3-dimethylquinoxalin-6-yl groups twist significantly out of the plane of the benzene ring. There are intermolecular  $\pi$ – $\pi$  interactions which result in a two-dimensional extended structure. The layers extend parallel to the *ab* plane and stack along the *c* axis.

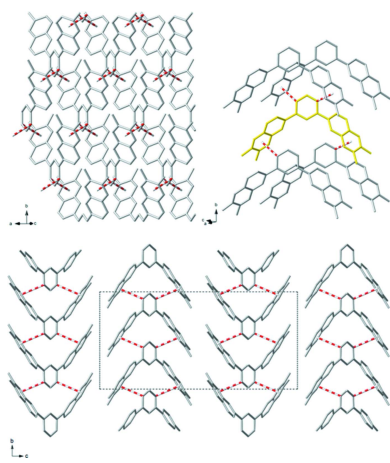
## 1. Chemical context

The title complex, (I), is one of the 1<sup>st</sup> generation of quinoxaline-terminated polyphenylene dendrimers that were prepared to study the effect of multivalency on the electrochemistry of quinoxalines (Carino *et al.*, 2015). The synthesis is based on C–H iridium-catalyzed borylation (Cho *et al.*, 2002) followed by Suzuki coupling, which was previously used in our group in the preparation of polyphenylene dendrimers (Finke & Moore, 2008).



## 2. Structural commentary

The molecular structure of (I) (Fig. 1) consists of a central phenyl ring with 2,3-dimethylquinoxalin-6-yl groups at the 1

**Figure 1**

Plot showing 35% probability displacement ellipsoids for non-H atoms and circles of arbitrary size for H atoms for (I). The unlabeled atoms are related by the symmetry operator  $(-x + 1, y, -z + \frac{1}{2})$ .

and 3 positions. The C1 and C4 carbon atoms of the central phenyl ring each occupy special positions ( $\frac{1}{2}, y, \frac{1}{2}$ ) and thus one-half of the molecule is generated by the symmetry operation  $(-x + 1, y, -z + \frac{1}{2})$ . The 2,3-dimethylquinoxalin-6-yl group is twisted significantly out of the plane of the central phenyl ring as evidenced by the C1–C2–C5–C6 torsion angle of  $-39.8(2)^\circ$ . The two six-membered rings of the 2,3-dimethylquinoxalin-6-yl group deviate from planarity as well; the dihedral angle between a best fit plane through the C5–C6–C7–C10–C11–C12 ring and a best fit plane through the C7–N1–C8–C9–N2–C10 ring is  $3.8(15)^\circ$ . The methyl groups also lie slightly out of the plane of the C7–N1–C8–C9–N2–C10 ring [N1–C8–C9–C14,  $\tau = -176.41(16)^\circ$ ; N2–C9–C8–C13,  $\tau = -176.95(15)^\circ$ ]. Similarly, the two methyl groups are not quite coplanar with a C13–C8–C9–C14 torsion angle of  $3.5(2)^\circ$ .

### 3. Supramolecular features

The molecules of (I) form extended layers *via* intermolecular  $\pi$ – $\pi$  interactions linking each molecule to its four nearest neighbors, Fig. 2*a,b*. The two-dimensional layers lie parallel to the *ab*-plane and stack along the *c* axis, Fig. 2*c*. The interactions occur between the central benzene ring and one of the heterocycles on a neighboring molecule. The orientation of these interacting groups is between ‘parallel offset’ and ‘perpendicular t-shaped’ as the C3–H3A bond points towards the C7<sup>ii</sup>–N1<sup>ii</sup>–C8<sup>ii</sup>–C9<sup>ii</sup>–N2<sup>ii</sup>–C10<sup>ii</sup> ring centroid [symmetry code: (ii)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ ]. The dihedral angle between a best fit plane through the C1–C2–C3–C4–C3<sup>i</sup>–C2<sup>i</sup> [symmetry code: (i)  $-x + 1, y, -z + \frac{1}{2}$ ] ring and a best-fit plane through the C7<sup>ii</sup>–N1<sup>ii</sup>–C8<sup>ii</sup>–C9<sup>ii</sup>–N2<sup>ii</sup>–C10<sup>ii</sup> ring is  $41.70(11)^\circ$ . The distance between the centroid of C7<sup>ii</sup>–N1<sup>ii</sup>–C8<sup>ii</sup>–C9<sup>ii</sup>–N2<sup>ii</sup>–C10<sup>ii</sup> ring

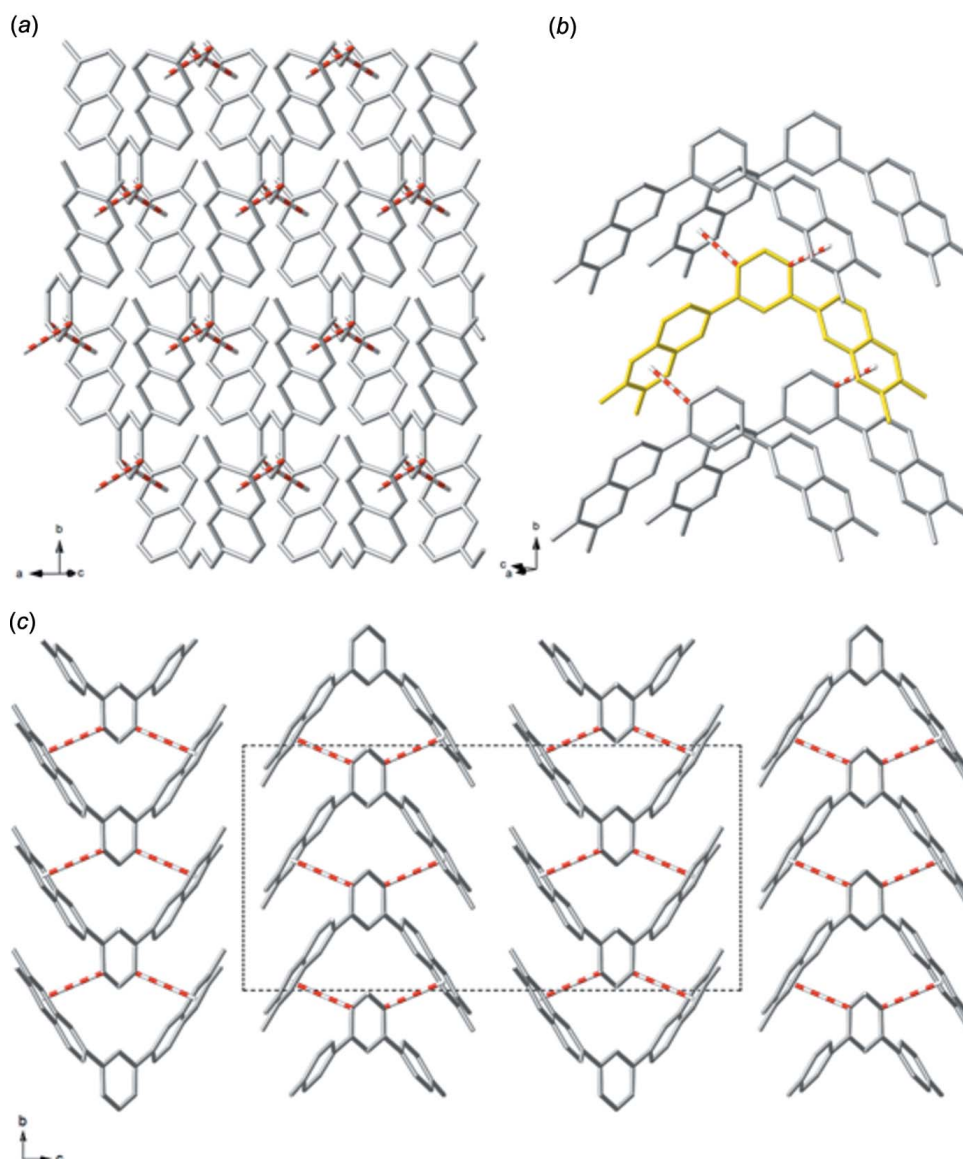


Figure 2

A plot of (a) a two-dimensional layer of (I), (b) a molecule of (I) highlighted in yellow showing it interacting with its four nearest neighbors, and (c) a view along the *a* axis showing the separation between the layers and an overlay of the unit cell. All H atoms have been omitted for clarity. The intermolecular interactions are indicated by red dashed lines.

and C3 is 3.311 (3) Å. The centroid(C7–N1–C8–C9–N2–C10)··centroid(C7–N1–C8–C9–N2–C10) distance between the layers of 4.721 (3) Å is too long to be considered another  $\pi$ – $\pi$  interaction. It appears the methyl groups on the quinoxaline prevent the layers from coming closer together.

#### 4. Database survey

A search of the Cambridge Crystal Database (Groom & Allen, 2014) returns zero results for 2,3-dimethylquinoxalin-6-yl groups attached to a phenyl ring. There are five reported crystal structures of 2,3-dimethylquinoxaline; the unsolvated species (Wozniak *et al.*, 1993), the dimethylglyoxime co-crystal (Hökelek *et al.*, 2001; Radhakrishnan *et al.*, 2007), the 2,6-dihydroxytoluene co-crystal, and the 2,6-dihydroxytoluene/4-dimethylaminopyridine co-crystal (Mir *et al.*, 2015). A related compound, 2,3-dimethyl-6-nitroquinoxaline, has been reported (Ghalib *et al.*, 2010) in which there is a nitro group bonded to the six-membered carbon ring of the quinoxaline. The dimeric version has also been characterized crystallographically, 2,2',3,3'-tetramethyl-6,6'-biquinoxaline, in which a single bond between the two six-membered carbon rings links a pair of 2,3-dimethylquinoxaline molecules (Salvatore *et al.*, 2006).

The five 2,3-dimethylquinoxaline structures have a range of the dihedral angle between a best-fit plane through the six-membered carbon ring and a best-fit plane through the six-membered nitrogen heterocycle of 0.02 (5)–1.59 (7)°. The two crystallographically independent molecules of the nitro-substituted compound have dihedral angles of 0.18 (3) and 1.07 (2)°, while this angle is 4.93 (2)° for the dimeric complex. The methyl groups for all of these molecules lie slightly out of the plane of the heterocycle with a range of N–C–C–Me torsion angles of 176.64 (7)–179.90 (5)°. The methyl groups in the database compounds range from nearly coplanar [Me–C–C–Me,  $\tau$  = 0.09 (11)°] to significantly more twisted out of plane [Me–C–C–Me,  $\tau$  = 3.33 (5)°]. Similar to (I), the dimeric molecule deviates significantly from being a planar molecule with a C2–C1–C1<sup>iii</sup>–C2<sup>iii</sup> [symmetry code: (iii)  $-x, y, \frac{1}{2} - z$ ] torsion angle of  $-43.40$  (10)° between the two 2,3-dimethylquinoxaline moieties.

#### 5. Synthesis and crystallization

Compound (I) was synthesized through the intermediate 2,3-dimethyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-quinoxaline (2) (see Fig. 3). In an Ar-filled dry box, a flame-dried vial with stirbar was charged with 2,3-dimethylquinoxaline (349.1 mg, 2.21 mmol), bis(pinacolato)diboron

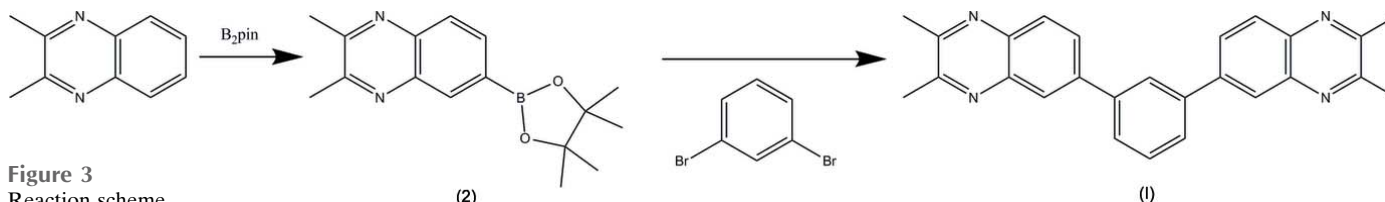
**Table 1**  
Experimental details.

Crystal data	
Chemical formula	C <sub>26</sub> H <sub>22</sub> N <sub>4</sub>
<i>M<sub>r</sub></i>	390.47
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.828 (3), 11.837 (5), 24.079 (11)
$\beta$ (°)	91.902 (5)
<i>V</i> (Å <sup>3</sup> )	1945.0 (15)
<i>Z</i>	4
Radiation type	Mo K $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.08
Crystal size (mm)	0.30 × 0.17 × 0.17
Data collection	
Diffractometer	Siemens Platform/APEXII CCD
Absorption correction	Integration (SADABS; Bruker, 2014)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.645, 1.000
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	7176, 1955, 1356
<i>R<sub>int</sub></i>	0.066
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.623
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.047, 0.138, 1.04
No. of reflections	1955
No. of parameters	140
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.23, $-0.22$

Computer programs: APEX2, SAINT (Bruker, 2014) and XPREP and XCIF (Bruker, 2014), SHELXTL (Sheldrick, 2008), CrystalMaker (CrystalMaker, 1994) and publCIF (Westrip, 2010).

B<sub>2</sub>pin] (423.0 mg, 1.67 mmol), [Ir(COD)(OMe)]<sub>2</sub> (44 mg, 0.07 mmol), dtbpy (37 mg, 0.14 mmol) and cyclohexane (10 ml). The mixture was stirred inside the glovebox at 363 K for 4.5 h. Then, B<sub>2</sub>pin (427.0 mg, 1.68 mmol), [Ir(COD)(OMe)]<sub>2</sub> (47 mg, 0.07 mmol), dtbpy (39 mg, 0.14 mmol) was added and the mixture further mixed at 363 K for 15 h. The reaction was filtered through silica, and the silica washed with chloroform. The combined filtrate was evaporated and the product was purified by silica chromatography using 5% EA in hexane. (417.4 mg, 1.47 mmol) 88% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (p.p.m.) 8.42 (s, 1H), 7.96 (d, *J* = 9.6 Hz, 1H), 7.88 (d, *J* = 9.6 Hz, 1H), 2.65 (s, 3H), 2.64 (s, 3H), 1.31 (s, 12H).

A vial with a stirbar was charged with (2) (200 mg, 0.70 mmol), 1,3-dibromobenzene (70 mg, 0.30 mmol), Pd(OAc)<sub>2</sub> (3.3 mg, 0.015 mmol), S-phos (12 mg, 0.03 mmol), THF (2 mL) and 5M NaOH (0.5 mL). The vial was sealed and heated to 343 K for 3 h. The solution was cooled and partitioned between Et<sub>2</sub>O (10 mL) and water (10 mL). The aqueous layer was extracted with Et<sub>2</sub>O (2 × 10 mL), the



**Figure 3**  
Reaction scheme.

combined organic layers were washed with water and brine, and dried over anhydrous  $\text{MgSO}_4$ . Column chromatography on silica gel eluting with 8:2 hexane:EA to provide (I) (312.2 mg). Suitable single crystals were grown from slow diffusion of hexanes into a dichloromethane solution of (I).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (p.p.m.) 8.29 (s, 2H), 8.09 (d,  $J = 9.6$  Hz, 2H), 8.04 (d,  $J = 9.6$  Hz, 2H), 8.03 (s, 1H), 7.80 (d,  $J = 7$  Hz, 2H), 7.63 (t,  $J = 7$  Hz, 1H), 2.77 (s, 12H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (p.p.m.) 154.1, 153.6, 141.4, 141.2, 140.8, 140.6, 129.8, 128.8, 128.4, 127.0, 126.7, 126.2, 23.4, 23.3. MS–ESI ( $m/z$ ): calculated for  $\text{C}_{26}\text{H}_{23}\text{N}_4$  [ $M + \text{H}$ ] $^+$ : 391.2 found: 391.2.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. A structural model consisting of half of the target molecule per asymmetric unit was developed. Methyl H atom positions,  $R\text{--CH}_3$ , were optimized by rotation about  $R\text{--C}$  bonds with idealized  $\text{C--H}$ ,  $R\text{--H}$  and  $\text{H--H}$  distances. The remaining H atoms were included as riding idealized contributors. For methyl H atoms  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ ;  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for remaining H atoms. The reflection 0 0 2 was omitted from the final refinements because it was partially blocked by the beamstop.

## Acknowledgements

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## supporting information

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## Crystal structure of 1,3-bis(2,3-dimethylquinoxalin-6-yl)benzene

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## Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINTE* (Bruker, 2014); data reduction: *SAINTE* (Bruker, 2014), *XPREF* (Bruker, 2014) and *SADABS* (Bruker, 2014); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *CrystalMaker* (*CrystalMaker*, 1994); software used to prepare material for publication: *XCIF* (Bruker, 2014) and *pubCIF* (Westrip, 2010).

## 1,3-Bis(2,3-dimethylquinoxalin-6-yl)benzene

## Crystal data

$C_{26}H_{22}N_4$

$M_r = 390.47$

Monoclinic, *C2/c*

Hall symbol: -C 2yc

$a = 6.828$  (3) Å

$b = 11.837$  (5) Å

$c = 24.079$  (11) Å

$\beta = 91.902$  (5)°

$V = 1945.0$  (15) Å<sup>3</sup>

$Z = 4$

$F(000) = 824$

$D_x = 1.333$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1141 reflections

$\theta = 3.4$ – $26.1$ °

$\mu = 0.08$  mm<sup>-1</sup>

$T = 173$  K

Prism, orange

$0.30 \times 0.17 \times 0.17$  mm

## Data collection

Siemens Platform/APEXII CCD  
diffractometer

Radiation source: normal-focus sealed tube

Graphite monochromator

profile data from  $\varphi$  and  $\omega$  scans

Absorption correction: integration  
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.645$ ,  $T_{\max} = 1.000$

7176 measured reflections

1955 independent reflections

1356 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.066$

$\theta_{\max} = 26.3$ °,  $\theta_{\min} = 3.4$ °

$h = -8 \rightarrow 8$

$k = -14 \rightarrow 14$

$l = -29 \rightarrow 29$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.138$

$S = 1.04$

1955 reflections

140 parameters

0 restraints

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 0.4908P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.23$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>



Extinction correction: SHELXTL (Bruker, 2014),  $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0082 (13)

### Special details

**Experimental.** One distinct cell was identified using *APEX2* (Bruker, 2014). Six frame series were integrated and filtered for statistical outliers using *SAINTE* (Bruker, 2014) and then the combined data was corrected for absorption by integration, sorted, merged and scaled using *SADABS* (Bruker, 2014). No decay correction was applied.

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Structure was phased by direct methods. Systematic conditions suggested the ambiguous space group. The space group choice was confirmed by successful convergence of the full-matrix least-squares refinement on  $F^2$ . The final difference Fourier map had no significant features. A final analysis of variance between observed and calculated structure factors showed little dependence on amplitude or resolution.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.8418 (2)	0.49563 (11)	0.40609 (6)	0.0291 (4)
N2	1.2363 (2)	0.57112 (12)	0.40420 (6)	0.0287 (4)
C1	0.5000	0.75167 (19)	0.2500	0.0262 (6)
H1A	0.5000	0.6714	0.2500	0.031*
C2	0.6402 (2)	0.80925 (14)	0.28278 (7)	0.0265 (4)
C3	0.6397 (3)	0.92738 (14)	0.28160 (7)	0.0294 (4)
H3A	0.7363	0.9680	0.3027	0.035*
C4	0.5000	0.9858 (2)	0.2500	0.0297 (6)
H4A	0.5000	1.0661	0.2500	0.036*
C5	0.7892 (2)	0.74799 (14)	0.31783 (7)	0.0273 (4)
C6	0.7440 (3)	0.65150 (13)	0.34720 (7)	0.0278 (4)
H6A	0.6127	0.6250	0.3469	0.033*
C7	0.8910 (3)	0.59216 (13)	0.37760 (7)	0.0266 (4)
C8	0.9866 (3)	0.43970 (14)	0.43105 (7)	0.0285 (4)
C9	1.1886 (3)	0.47661 (14)	0.42908 (7)	0.0286 (4)
C10	1.0870 (2)	0.63148 (14)	0.37843 (7)	0.0272 (4)
C11	1.1300 (3)	0.73199 (14)	0.35013 (7)	0.0290 (4)
H11A	1.2601	0.7605	0.3511	0.035*
C12	0.9854 (3)	0.78862 (14)	0.32128 (7)	0.0289 (4)
H12A	1.0165	0.8571	0.3030	0.035*
C13	0.9366 (3)	0.33468 (15)	0.46213 (8)	0.0343 (5)
H13A	0.7939	0.3282	0.4641	0.051*
H13B	0.9950	0.3384	0.4998	0.051*
H13C	0.9881	0.2687	0.4428	0.051*
C14	1.3495 (3)	0.40683 (15)	0.45522 (8)	0.0355 (5)
H14A	1.4766	0.4374	0.4449	0.053*
H14B	1.3373	0.3286	0.4422	0.053*
H14C	1.3399	0.4088	0.4957	0.053*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0268 (8)	0.0301 (8)	0.0305 (8)	-0.0014 (6)	0.0008 (7)	0.0005 (6)
N2	0.0240 (8)	0.0340 (8)	0.0280 (8)	0.0010 (6)	0.0013 (7)	-0.0021 (6)
C1	0.0236 (13)	0.0262 (12)	0.0290 (13)	0.000	0.0039 (11)	0.000
C2	0.0260 (10)	0.0298 (9)	0.0239 (9)	-0.0005 (7)	0.0050 (8)	0.0003 (7)
C3	0.0315 (11)	0.0305 (9)	0.0262 (9)	-0.0027 (7)	0.0015 (8)	-0.0030 (7)
C4	0.0360 (15)	0.0255 (12)	0.0278 (13)	0.000	0.0034 (12)	0.000
C5	0.0278 (10)	0.0297 (9)	0.0243 (9)	0.0006 (7)	-0.0004 (8)	-0.0031 (7)
C6	0.0219 (9)	0.0319 (9)	0.0295 (10)	-0.0019 (7)	0.0001 (8)	-0.0010 (7)
C7	0.0269 (10)	0.0292 (9)	0.0238 (9)	0.0000 (7)	0.0034 (8)	-0.0007 (7)
C8	0.0299 (10)	0.0289 (9)	0.0263 (9)	0.0012 (8)	-0.0016 (8)	-0.0017 (7)
C9	0.0304 (10)	0.0321 (9)	0.0233 (9)	0.0029 (7)	0.0001 (8)	-0.0047 (7)
C10	0.0241 (10)	0.0316 (9)	0.0257 (9)	0.0000 (7)	-0.0003 (8)	-0.0037 (7)
C11	0.0243 (10)	0.0339 (10)	0.0289 (10)	-0.0050 (7)	0.0024 (8)	-0.0037 (7)
C12	0.0291 (10)	0.0317 (9)	0.0258 (9)	-0.0032 (7)	0.0022 (8)	-0.0001 (7)
C13	0.0338 (11)	0.0333 (10)	0.0355 (11)	0.0010 (8)	-0.0029 (9)	0.0049 (8)
C14	0.0299 (11)	0.0392 (10)	0.0371 (11)	0.0045 (8)	-0.0015 (9)	0.0015 (8)

*Geometric parameters (Å, °)*

N1—C8	1.318 (2)	C6—H6A	0.9500
N1—C7	1.380 (2)	C7—C10	1.416 (2)
N2—C9	1.315 (2)	C8—C9	1.449 (3)
N2—C10	1.376 (2)	C8—C13	1.496 (2)
C1—C2	1.3980 (19)	C9—C14	1.496 (2)
C1—C2 <sup>i</sup>	1.3980 (19)	C10—C11	1.407 (2)
C1—H1A	0.9500	C11—C12	1.364 (2)
C2—C3	1.399 (2)	C11—H11A	0.9500
C2—C5	1.488 (2)	C12—H12A	0.9500
C3—C4	1.385 (2)	C13—H13A	0.9800
C3—H3A	0.9500	C13—H13B	0.9800
C4—C3 <sup>i</sup>	1.385 (2)	C13—H13C	0.9800
C4—H4A	0.9500	C14—H14A	0.9800
C5—C6	1.384 (2)	C14—H14B	0.9800
C5—C12	1.423 (2)	C14—H14C	0.9800
C6—C7	1.410 (2)		
C8—N1—C7	116.81 (15)	C9—C8—C13	120.00 (15)
C9—N2—C10	117.13 (15)	N2—C9—C8	121.45 (15)
C2—C1—C2 <sup>i</sup>	121.6 (2)	N2—C9—C14	118.15 (16)
C2—C1—H1A	119.2	C8—C9—C14	120.40 (16)
C2 <sup>i</sup> —C1—H1A	119.2	N2—C10—C11	119.60 (16)
C1—C2—C3	118.34 (16)	N2—C10—C7	121.43 (16)
C1—C2—C5	121.66 (16)	C11—C10—C7	118.91 (15)
C3—C2—C5	120.00 (15)	C12—C11—C10	120.21 (16)
C4—C3—C2	120.79 (16)	C12—C11—H11A	119.9

C4—C3—H3A	119.6	C10—C11—H11A	119.9
C2—C3—H3A	119.6	C11—C12—C5	121.83 (16)
C3 <sup>i</sup> —C4—C3	120.1 (2)	C11—C12—H12A	119.1
C3 <sup>i</sup> —C4—H4A	120.0	C5—C12—H12A	119.1
C3—C4—H4A	120.0	C8—C13—H13A	109.5
C6—C5—C12	118.32 (15)	C8—C13—H13B	109.5
C6—C5—C2	122.17 (16)	H13A—C13—H13B	109.5
C12—C5—C2	119.51 (16)	C8—C13—H13C	109.5
C5—C6—C7	120.66 (16)	H13A—C13—H13C	109.5
C5—C6—H6A	119.7	H13B—C13—H13C	109.5
C7—C6—H6A	119.7	C9—C14—H14A	109.5
N1—C7—C6	119.33 (16)	C9—C14—H14B	109.5
N1—C7—C10	120.71 (15)	H14A—C14—H14B	109.5
C6—C7—C10	119.96 (16)	C9—C14—H14C	109.5
N1—C8—C9	122.28 (16)	H14A—C14—H14C	109.5
N1—C8—C13	117.71 (16)	H14B—C14—H14C	109.5
C2 <sup>i</sup> —C1—C2—C3	-0.93 (12)	C10—N2—C9—C8	-2.2 (2)
C2 <sup>i</sup> —C1—C2—C5	179.81 (18)	C10—N2—C9—C14	177.32 (15)
C1—C2—C3—C4	1.9 (2)	N1—C8—C9—N2	3.1 (3)
C5—C2—C3—C4	-178.83 (14)	C13—C8—C9—N2	-176.95 (15)
C2—C3—C4—C3 <sup>i</sup>	-0.97 (12)	N1—C8—C9—C14	-176.41 (16)
C1—C2—C5—C6	-39.8 (2)	C13—C8—C9—C14	3.5 (2)
C3—C2—C5—C6	140.92 (18)	C9—N2—C10—C11	-178.64 (15)
C1—C2—C5—C12	139.70 (15)	C9—N2—C10—C7	-1.4 (2)
C3—C2—C5—C12	-39.6 (3)	N1—C7—C10—N2	4.6 (3)
C12—C5—C6—C7	-3.0 (3)	C6—C7—C10—N2	-175.09 (15)
C2—C5—C6—C7	176.57 (15)	N1—C7—C10—C11	-178.22 (14)
C8—N1—C7—C6	176.03 (15)	C6—C7—C10—C11	2.1 (3)
C8—N1—C7—C10	-3.6 (2)	N2—C10—C11—C12	175.60 (15)
C5—C6—C7—N1	-179.42 (15)	C7—C10—C11—C12	-1.7 (3)
C5—C6—C7—C10	0.2 (3)	C10—C11—C12—C5	-1.1 (3)
C7—N1—C8—C9	0.0 (2)	C6—C5—C12—C11	3.5 (3)
C7—N1—C8—C13	-179.94 (14)	C2—C5—C12—C11	-176.07 (15)

Symmetry code: (i)  $-x+1, y, -z+1/2$ .