

2008

## 2,2,3,30-Tetraphenyl-7,70-biquinoxaline

Eric E. Dueno

Ricky J. Paul

Ralph N. Salvatore

Robert D. Pike

*William & Mary*, [rdpike@wm.edu](mailto:rdpike@wm.edu)

Follow this and additional works at: <https://scholarworks.wm.edu/aspubs>

 Part of the [Chemistry Commons](#)

---

### Recommended Citation

Dueno, Eric E.; Paul, Ricky J.; Salvatore, Ralph N.; and Pike, Robert D.,  
2,2,3,30-Tetraphenyl-7,70-biquinoxaline (2008). *Acta Crystallographica Section E*, E64.  
<https://doi.org/10.1107/S1600536807033521>

This Article is brought to you for free and open access by the Arts and Sciences at W&M ScholarWorks. It has been accepted for inclusion in Arts & Sciences Articles by an authorized administrator of W&M ScholarWorks. For more information, please contact [scholarworks@wm.edu](mailto:scholarworks@wm.edu).

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2,2,3,3'-Tetraphenyl-7,7'-biquinoxaline

Eric E. Dueno,<sup>a\*</sup> Ricky Joseph Paul Gibson,<sup>a</sup>  
Ralph Nicholas Salvatore,<sup>a</sup> Robert D. Pike<sup>b</sup> and Cesar H.  
Zambrano<sup>c</sup>

<sup>a</sup>Department of Chemistry, Eastern Kentucky University, 521 Lancaster Avenue, Richmond, KY 40475, USA, <sup>b</sup>Department of Chemistry, The College of William and Mary, PO Box 8795, Williamsburg, VA 23187-8795, USA, and <sup>c</sup>Departamento de Química, Universidad San Francisco de Quito, Pampite & Robles - Cumbaya, Quito, Ecuador

Correspondence e-mail: eric.dueno@eku.edu

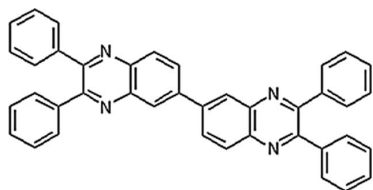
Received 29 June 2007; accepted 9 July 2007

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.091; data-to-parameter ratio = 9.7.

In the crystal structure of the title compound,  $\text{C}_{40}\text{H}_{26}\text{N}_4$ , molecules reside on crystallographic centers of inversion and are linked *via*  $\text{C}-\text{H}\cdots\text{N}$  interactions about inversion centers into one-dimensional chains: longer  $\text{C}-\text{H}\cdots\pi(\text{arene})$  interactions complete the intermolecular interactions.

## Related literature

For the synthesis of quinoxalines, see: Kowalski *et al.* (2006); Kou *et al.* (2006); Baek & Tan (2006). For applications of quinoxalines see: Mollegaard *et al.* (2000); Aldakov *et al.* (2005); Kaiwar *et al.* (1997); Anzenbacher *et al.* (2000). For related literature, see: Brown *et al.* (2004); Bruno *et al.* (2002); Gibson *et al.* (2006); Page *et al.* (1998); Pascal & Ho (1993); Salvatore *et al.* (2006); Simpson & Gordon (1995); Willett *et al.* (2001); Wozniak *et al.* (1993); Wu *et al.* (2002).



## Experimental

## Crystal data

$\text{C}_{40}\text{H}_{26}\text{N}_4$   $\gamma = 91.7510$  (10) $^\circ$   
 $M_r = 562.65$   $V = 704.19$  (3) Å<sup>3</sup>  
 Triclinic,  $P\bar{1}$   $Z = 1$   
 $a = 5.70240$  (10) Å  $\text{Cu K}\alpha$  radiation  
 $b = 9.9534$  (2) Å  $\mu = 0.61$  mm<sup>-1</sup>  
 $c = 12.9785$  (3) Å  $T = 100$  (2) K  
 $\alpha = 105.3520$  (10) $^\circ$   $0.19 \times 0.12 \times 0.06$  mm  
 $\beta = 96.6170$  (10) $^\circ$

## Data collection

Bruker SMART APEXII CCD 12179 measured reflections  
 diffractometer 2429 independent reflections  
 Absorption correction: numerical 2156 reflections with  $I > 2\sigma(I)$   
 (SADABS; Sheldrick, 2004)  $R_{\text{int}} = 0.033$   
 $T_{\text{min}} = 0.893$ ,  $T_{\text{max}} = 0.964$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$  251 parameters  
 $wR(F^2) = 0.091$  All H-atom parameters refined  
 $S = 1.06$   $\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup>  
 2429 reflections  $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å,  $^\circ$ ).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{C}18^{\text{i}}$	0.977 (15)	2.722 (15)	3.5521 (16)	143.1 (12)
$\text{C}7-\text{H}7\cdots\text{N}2^{\text{ii}}$	0.979 (15)	2.593 (16)	3.3635 (15)	135.6 (11)
$\text{C}10-\text{H}10\cdots\text{C}3^{\text{iii}}$	0.974 (13)	2.935 (12)	3.2994 (15)	103.4 (8)
$\text{C}10-\text{H}10\cdots\text{C}6^{\text{iii}}$	0.974 (13)	2.974 (13)	3.1801 (15)	93.1 (8)
$\text{C}11-\text{H}11\cdots\text{N}1^{\text{iii}}$	0.963 (15)	2.893 (14)	3.3233 (14)	108.3 (10)
$\text{C}12-\text{H}12\cdots\text{C}18^{\text{iv}}$	0.975 (15)	2.959 (15)	3.6146 (16)	125.6 (10)
$\text{C}13-\text{H}13\cdots\text{C}18^{\text{iv}}$	0.999 (15)	2.978 (15)	3.6141 (16)	122.5 (10)
$\text{C}16-\text{H}16\cdots\text{N}2^{\text{ii}}$	0.978 (14)	2.815 (14)	3.7256 (14)	155.2 (11)
$\text{C}17-\text{H}17\cdots\text{C}1^{\text{v}}$	0.969 (13)	2.993 (14)	3.7019 (16)	131.0 (9)
$\text{C}17-\text{H}17\cdots\text{C}8^{\text{v}}$	0.969 (13)	2.860 (14)	3.6338 (15)	137.5 (9)
$\text{C}18-\text{H}18\cdots\text{N}1^{\text{vi}}$	0.975 (14)	2.808 (14)	3.6217 (14)	141.5 (10)

Symmetry codes: (i)  $x, y+1, z$ ; (ii)  $-x, -y+1, -z+1$ ; (iii)  $x+1, y, z$ ; (iv)  $-x+2, -y+1, -z+2$ ; (v)  $-x+1, -y+1, -z+1$ ; (vi)  $x, y-1, z$ .

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: XSELL (Bruker, 2004); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 (Sheldrick, 1997).

EED acknowledges the National Science Foundation for primary support of this research (EPSCOR grant No. 450901). RDP is indebted to the NSF (CHE-0443345) and the College of William and Mary for the purchase of the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2021).

## References

- Aldakov, D., Palacios, M. A. & Anzenbacher, P. Jr (2005). *Chem. Mater.* **17**, 5238–5241.  
 Anzenbacher, P. Jr, Try, A. C., Miyaji, H., Jursikova, K., Lynch, V. M., Marquez, M. & Sessler, J. L. (2000). *J. Am. Chem. Soc.* **122**, 10268–10272.  
 Baek, J.-B. & Tan, L.-S. (2006). *Macromolecules*, **39**, 2794–2803.  
 Brown, D. J., Taylor, E. C. & Wipf, P. (2004). *The Chemistry of Heterocyclic Compounds*, Vol. 61, *Quinoxalines*, Suppl. II, pp. 100–105. New York: Wiley.  
 Bruker (2004). APEX2 (Version 2.0), SAINT-Plus (Version 7.12A) and XSELL (Version 6.3.1). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Gibson, R. J. P., Kass, J. P., Zambrano, C. H., Fronczek, F. R. & Dueno, E. E. (2006). *Acta Cryst.* **E62**, o2947–o2948.  
 Kaiwar, S. P., Vodacek, A., Blough, N. V. & Pilato, R. S. (1997). *J. Am. Chem. Soc.* **119**, 3311–3316.  
 Kou, B.-B., Zhang, F., Yang, T.-M. & Liu, G. (2006). *J. Comb. Chem.* **8**, 841–847.

- Kowalski, J. A., Leonard, S. F. & Lee, G. E. Jr (2006). *J. Comb. Chem.* **8**, 774–779.
- Mollegaard, N. E., Bailly, C., Waring, M. J. & Nielsen, P. E. (2000). *Biochemistry*, **39**, 9502–9507.
- Page, S. E., Gordon, K. C. & Burrell, A. K. (1998). *Inorg. Chem.* **37**, 4452–4459.
- Pascal, R. A. Jr & Ho, D. M. (1993). *J. Am. Chem. Soc.* **115**, 8507–8508.
- Salvatore, R. N., Kass, J. P., Gibson, R. J. P., Zambrano, C. H., Pike, R. D. & Dueno, E. E. (2006). *Acta Cryst.* **E62**, o4547–o4548.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
- Simpson, T. J. & Gordon, K. C. (1995). *Inorg. Chem.* **34**, 6323–6329.
- Willett, R. D., Jeitler, R. J. & Twamley, B. (2001). *Inorg. Chem.* **40**, 6502–6505.
- Wozniak, K., Krygowski, T. M., Grech, E., Kolodziejski, W. & Klinowski, J. (1993). *J. Phys. Chem.* **97**, 1862–1867.
- Wu, C. D., Lu, C.-Z., Zhuang, H.-H. & Huang, J.-S. (2002). *Inorg. Chem.* **41**, 5636–5637.

## supporting information

*Acta Cryst.* (2008). E64, o110–o111 [https://doi.org/10.1107/S1600536807033521]

**2,2,3,3'-Tetraphenyl-7,7'-biquinoxaline**

**Eric E. Dueno, Ricky Joseph Paul Gibson, Ralph Nicholas Salvatore, Robert D. Pike and Cesar H. Zambrano**

**S1. Comment**

Quinoxalines and their derivatives have received considerable attention in the past several years due to their electronic properties (Page *et al.*, 1998; Simpson & Gordon, 1995), H-bonding ability (Pascal *et al.*, 1993; Wozniak *et al.*, 1993), and their capacity to coordinate to metals forming interesting three-dimensional structures (Wu *et al.*, 2002; Willett *et al.*, 2001). During our investigations, we have prepared a number of substituted quinoxalines and phenazines, some of which coordinate to metal salts forming novel structures (Dueno, *et al.*, unpublished). Our current work involves the synthesis of new nitrogen heterocycles (Gibson, *et al.*, 2006; Salvatore, *et al.*, 2006) which may lead to novel three dimensional structures upon coordination to metal salts. Here, we report the crystal structure of 2,2',3,3'-Tetraphenyl-7,7'-biquinoxaline (I), (Figure 1).

The structure of (I) has bond distances and angles that are unexceptional, as all fall within ranges found in the literature for similar nitrogen heterocycles (Brown *et al.*, 2004). The one molecule present in the asymmetric unit cell lies on an inversion center, so that half the molecule is related to its counterpart by symmetry (symmetry code,  $-x, -y, -z$ ). As expected, the steric bulk of the phenyl substituents prevents them from being coplanar with the quinoxaline rings: the dihedral angle N2—C5—C15—C20 (phenyl ring 1) is 59.80 (11)°, and the dihedral angle N1—C4—C9—C10 (phenyl ring 2) is 25.98 (11)°. An interesting feature worth mentioning is that the two rings that make up the quinoxaline unit are not perfectly planar, for the angle between the N containing ring and the carbon-only ring is 3.50 (11)° (based on a least squares mean planes of N1—C4—C5—N2—C6—C3 and C1—C2—C3—C6—C7—C8). It is conceivable that this deviation from a planar structure is due to Van der Waals repulsion interactions between the aromatic substituents. Another interesting aspect of this molecule is that the packing diagram shows short contact interactions between phenyl substituents on one molecule and the quinoxaline ring of an adjacent molecule. Intermolecular distances range from 3.181 (2) Å (C10—C6) to 3.376 (2) Å (C11—C4), which suggests some degree of  $\sigma(\text{CH})\cdots\pi$  interaction (Figure 2).

**S2. Experimental**

A 50 ml round-bottomed flask was charged with biphenyl-3,3',4,4'-tetramine (214 mg, 1 mmol), benzil (420 mg, 2 mmol), iodine (51 mg, 0.2 mmol), and acetonitrile (15 ml). The reaction was monitored by thin-layer chromatography until complete consumption of the starting materials (15 min). The resulting amber solution was concentrated to dryness under reduced pressure. The dark-brown crude product was then subjected to flash column chromatography using silica gel (eluent: 9:1 hexane–EtOAc) in order to remove residual iodine. The pale-yellow solution was evaporated to dryness under reduced pressure to give (I) (yield 0.413 mg, 74%), as a white powder (m.p. 573 K). This powder was then crystallized from a minimal amount of toluene, and afforded (I) as pale-yellow cubes.

## S3. Refinement

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

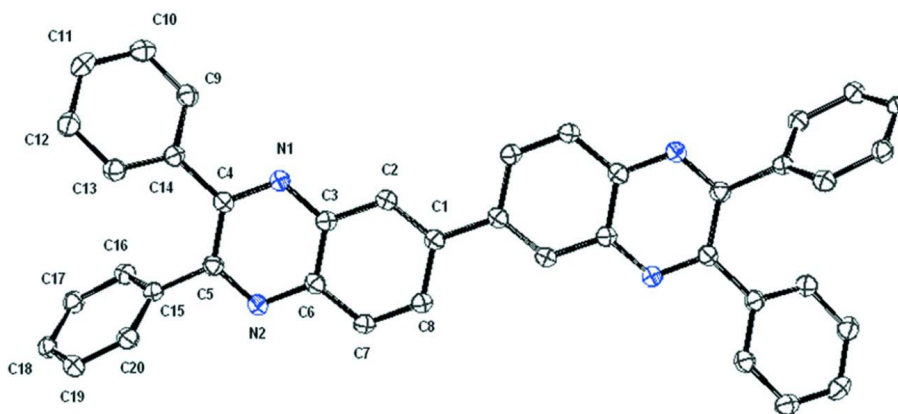


Figure 1

ORTEP (Farrugia, 1997) drawing of (I). Displacement ellipsoids have been drawn at the 50% probability level. Unlabeled atoms are related to labeled atoms by symmetry (code  $-x, -y, -z$ ). H atoms have been omitted for clarity.

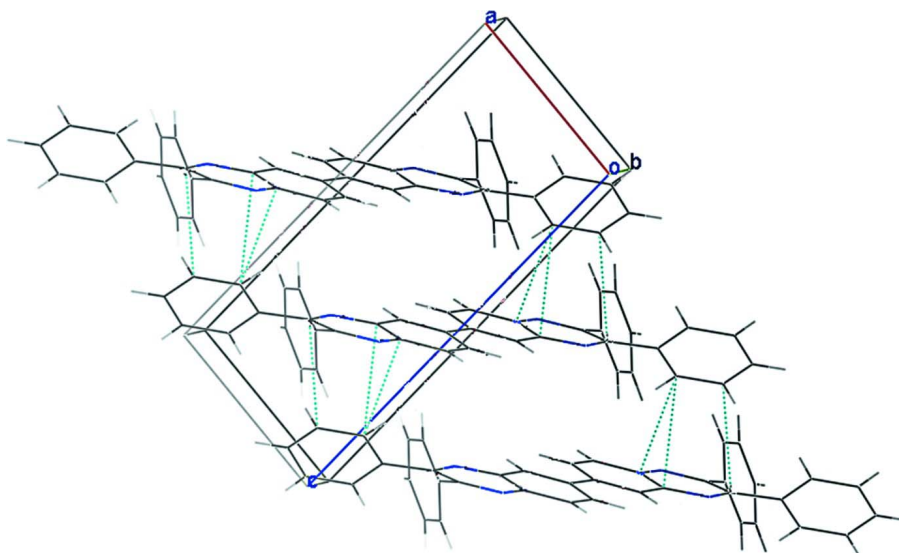


Figure 2

Mercury (Bruno *et al.*, 2002) packing diagram of (I) along the  $b$  axis showing short contact interactions.

## 2,2,3,3'-Tetraphenyl-7,7'-biquinoxaline

*Crystal data*

$C_{40}H_{26}N_4$

$M_r = 562.65$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 5.7024 (1) \text{ \AA}$

$b = 9.9534 (2) \text{ \AA}$

$c = 12.9785 (3) \text{ \AA}$

$\alpha = 105.352 (1)^\circ$

$\beta = 96.617 (1)^\circ$

$\gamma = 91.751 (1)^\circ$

$V = 704.19 (3) \text{ \AA}^3$

$Z = 1$

$F(000) = 294$

$D_x = 1.327 \text{ Mg m}^{-3}$

Cu  $K\alpha$  radiation,  $\lambda = 1.54178 \text{ \AA}$

Cell parameters from 585 reflections

$\theta = 3.6\text{--}67.0^\circ$   
 $\mu = 0.61\text{ mm}^{-1}$   
 $T = 100\text{ K}$

Block, colourless  
 $0.19 \times 0.12 \times 0.06\text{ mm}$

*Data collection*

Bruker SMART APEXII CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\omega$  and  $\psi$  scans  
 Absorption correction: numerical  
 (SADABS; Sheldrick, 2004)  
 $T_{\min} = 0.893$ ,  $T_{\max} = 0.964$

12179 measured reflections  
 2429 independent reflections  
 2156 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$   
 $\theta_{\max} = 67.0^\circ$ ,  $\theta_{\min} = 3.6^\circ$   
 $h = -6 \rightarrow 6$   
 $k = -11 \rightarrow 11$   
 $l = -15 \rightarrow 15$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.091$   
 $S = 1.06$   
 2429 reflections  
 251 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 All H-atom parameters refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.1323P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.23\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.18\text{ e \AA}^{-3}$

*Special details*

**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.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*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	0.51854 (16)	0.85128 (10)	0.69940 (8)	0.0221 (2)
N2	0.23046 (17)	0.60236 (10)	0.64109 (8)	0.0224 (2)
C1	0.03603 (19)	0.94087 (11)	0.52202 (9)	0.0209 (3)
C2	0.2496 (2)	0.94755 (12)	0.58600 (9)	0.0226 (3)
C3	0.31629 (19)	0.83612 (11)	0.62942 (9)	0.0211 (3)
C4	0.57593 (19)	0.74558 (12)	0.73939 (9)	0.0212 (3)
C5	0.43238 (19)	0.61520 (12)	0.70434 (9)	0.0211 (3)
C6	0.1661 (2)	0.71321 (12)	0.60385 (9)	0.0217 (3)
C7	-0.0511 (2)	0.70597 (12)	0.53726 (9)	0.0231 (3)
C8	-0.1142 (2)	0.81609 (12)	0.49890 (9)	0.0225 (3)
C9	0.79004 (19)	0.77207 (11)	0.82117 (9)	0.0219 (3)
C10	0.9637 (2)	0.87320 (12)	0.81830 (10)	0.0239 (3)
C11	1.1675 (2)	0.90121 (13)	0.89186 (10)	0.0269 (3)

C12	1.2013 (2)	0.82934 (13)	0.97005 (10)	0.0288 (3)
C13	1.0272 (2)	0.73173 (13)	0.97578 (10)	0.0281 (3)
C14	0.8232 (2)	0.70364 (12)	0.90232 (10)	0.0252 (3)
C15	0.49998 (19)	0.48375 (11)	0.73197 (9)	0.0215 (3)
C16	0.7014 (2)	0.41920 (12)	0.69865 (9)	0.0240 (3)
C17	0.7507 (2)	0.29096 (12)	0.71734 (10)	0.0248 (3)
C18	0.6025 (2)	0.22840 (12)	0.77110 (9)	0.0244 (3)
C19	0.4035 (2)	0.29355 (13)	0.80581 (10)	0.0268 (3)
C20	0.3503 (2)	0.42037 (12)	0.78536 (10)	0.0247 (3)
H2	0.361 (3)	1.0290 (16)	0.6039 (12)	0.035 (4)*
H7	-0.153 (3)	0.6200 (16)	0.5199 (12)	0.035 (4)*
H8	-0.267 (2)	0.8096 (13)	0.4535 (11)	0.021 (3)*
H10	0.938 (2)	0.9233 (14)	0.7633 (11)	0.026 (3)*
H11	1.287 (3)	0.9694 (15)	0.8870 (12)	0.033 (4)*
H12	1.344 (3)	0.8484 (16)	1.0224 (12)	0.036 (4)*
H13	1.048 (2)	0.6786 (16)	1.0312 (13)	0.037 (4)*
H14	0.698 (3)	0.6366 (16)	0.9090 (12)	0.033 (4)*
H16	0.807 (2)	0.4646 (15)	0.6621 (12)	0.029 (3)*
H17	0.888 (2)	0.2451 (14)	0.6924 (11)	0.023 (3)*
H18	0.640 (2)	0.1387 (15)	0.7834 (11)	0.028 (3)*
H19	0.296 (2)	0.2499 (15)	0.8440 (12)	0.033 (4)*
H20	0.210 (3)	0.4660 (15)	0.8084 (12)	0.032 (4)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0212 (5)	0.0209 (5)	0.0245 (5)	0.0010 (4)	0.0015 (4)	0.0073 (4)
N2	0.0241 (5)	0.0195 (5)	0.0240 (5)	0.0010 (4)	0.0019 (4)	0.0070 (4)
C1	0.0227 (6)	0.0196 (6)	0.0205 (6)	0.0010 (5)	0.0037 (4)	0.0052 (4)
C2	0.0228 (6)	0.0193 (6)	0.0258 (6)	-0.0015 (5)	0.0015 (4)	0.0073 (5)
C3	0.0204 (6)	0.0208 (6)	0.0216 (6)	0.0006 (5)	0.0027 (4)	0.0049 (4)
C4	0.0219 (6)	0.0196 (5)	0.0231 (6)	0.0019 (4)	0.0047 (4)	0.0067 (4)
C5	0.0204 (5)	0.0208 (6)	0.0224 (6)	0.0010 (4)	0.0035 (4)	0.0060 (4)
C6	0.0243 (6)	0.0189 (5)	0.0217 (6)	0.0012 (5)	0.0038 (4)	0.0051 (4)
C7	0.0238 (6)	0.0199 (6)	0.0244 (6)	-0.0024 (5)	0.0005 (4)	0.0053 (4)
C8	0.0224 (6)	0.0217 (6)	0.0227 (6)	-0.0013 (5)	0.0003 (4)	0.0058 (4)
C9	0.0207 (6)	0.0187 (5)	0.0249 (6)	0.0033 (4)	0.0025 (4)	0.0034 (4)
C10	0.0247 (6)	0.0207 (6)	0.0260 (6)	0.0034 (5)	0.0037 (5)	0.0057 (5)
C11	0.0227 (6)	0.0256 (6)	0.0302 (7)	-0.0002 (5)	0.0041 (5)	0.0036 (5)
C12	0.0224 (6)	0.0343 (7)	0.0261 (6)	0.0014 (5)	-0.0025 (5)	0.0042 (5)
C13	0.0279 (6)	0.0295 (6)	0.0270 (6)	0.0038 (5)	0.0002 (5)	0.0087 (5)
C14	0.0248 (6)	0.0235 (6)	0.0262 (6)	0.0008 (5)	0.0015 (5)	0.0058 (5)
C15	0.0218 (6)	0.0185 (5)	0.0225 (6)	-0.0011 (4)	-0.0023 (4)	0.0050 (4)
C16	0.0240 (6)	0.0229 (6)	0.0258 (6)	0.0002 (5)	0.0034 (5)	0.0077 (5)
C17	0.0238 (6)	0.0225 (6)	0.0264 (6)	0.0041 (5)	0.0008 (5)	0.0045 (5)
C18	0.0285 (6)	0.0180 (5)	0.0253 (6)	0.0009 (5)	-0.0048 (5)	0.0069 (4)
C19	0.0260 (6)	0.0259 (6)	0.0310 (7)	-0.0011 (5)	0.0017 (5)	0.0131 (5)
C20	0.0206 (6)	0.0248 (6)	0.0296 (6)	0.0023 (5)	0.0030 (5)	0.0089 (5)

*Geometric parameters (Å, °)*

N1—C4	1.3245 (15)	C10—H10	0.974 (14)
N1—C3	1.3613 (14)	C11—C12	1.3880 (18)
N2—C5	1.3166 (15)	C11—H11	0.966 (15)
N2—C6	1.3609 (15)	C12—C13	1.3899 (18)
C1—C2	1.3819 (16)	C12—H12	0.978 (15)
C1—C8	1.4301 (16)	C13—C14	1.3870 (17)
C1—C1 <sup>i</sup>	1.488 (2)	C13—H13	0.998 (15)
C2—C3	1.4141 (16)	C14—H14	0.988 (15)
C2—H2	0.976 (15)	C15—C16	1.3898 (17)
C3—C6	1.4129 (16)	C15—C20	1.3936 (16)
C4—C5	1.4484 (16)	C16—C17	1.3918 (16)
C4—C9	1.4900 (15)	C16—H16	0.976 (14)
C5—C15	1.4955 (15)	C17—C18	1.3865 (17)
C6—C7	1.4143 (16)	C17—H17	0.962 (14)
C7—C8	1.3604 (16)	C18—C19	1.3852 (18)
C7—H7	0.977 (15)	C18—H18	0.973 (14)
C8—H8	0.986 (13)	C19—C20	1.3922 (16)
C9—C14	1.3967 (17)	C19—H19	0.990 (15)
C9—C10	1.4010 (16)	C20—H20	0.971 (15)
C10—C11	1.3868 (17)		
C4—N1—C3	118.29 (10)	C9—C10—H10	118.5 (8)
C5—N2—C6	117.97 (10)	C12—C11—C10	120.12 (11)
C2—C1—C8	117.90 (10)	C12—C11—H11	120.4 (8)
C2—C1—C1 <sup>i</sup>	121.33 (13)	C10—C11—H11	119.4 (8)
C8—C1—C1 <sup>i</sup>	120.76 (12)	C11—C12—C13	119.59 (11)
C1—C2—C3	121.34 (11)	C11—C12—H12	120.8 (9)
C1—C2—H2	122.0 (8)	C13—C12—H12	119.6 (9)
C3—C2—H2	116.6 (9)	C14—C13—C12	120.35 (11)
N1—C3—C6	120.83 (10)	C14—C13—H13	118.8 (9)
N1—C3—C2	119.57 (10)	C12—C13—H13	120.9 (9)
C6—C3—C2	119.52 (10)	C13—C14—C9	120.67 (11)
N1—C4—C5	120.22 (10)	C13—C14—H14	119.5 (8)
N1—C4—C9	115.70 (10)	C9—C14—H14	119.8 (8)
C5—C4—C9	124.07 (10)	C16—C15—C20	119.65 (10)
N2—C5—C4	121.55 (10)	C16—C15—C5	120.82 (10)
N2—C5—C15	114.09 (10)	C20—C15—C5	119.38 (10)
C4—C5—C15	124.33 (10)	C15—C16—C17	119.93 (11)
N2—C6—C3	120.68 (10)	C15—C16—H16	119.3 (8)
N2—C6—C7	120.32 (10)	C17—C16—H16	120.8 (8)
C3—C6—C7	118.99 (10)	C18—C17—C16	120.38 (11)
C8—C7—C6	120.36 (11)	C18—C17—H17	119.8 (8)
C8—C7—H7	121.6 (9)	C16—C17—H17	119.8 (8)
C6—C7—H7	118.1 (9)	C19—C18—C17	119.78 (11)
C7—C8—C1	121.85 (11)	C19—C18—H18	121.0 (8)
C7—C8—H8	119.2 (7)	C17—C18—H18	119.2 (8)



C1—C8—H8	119.0 (7)	C18—C19—C20	120.18 (11)
C14—C9—C10	118.36 (11)	C18—C19—H19	120.6 (8)
C14—C9—C4	123.20 (10)	C20—C19—H19	119.2 (8)
C10—C9—C4	118.42 (10)	C15—C20—C19	120.05 (11)
C11—C10—C9	120.85 (11)	C15—C20—H20	119.3 (8)
C11—C10—H10	120.7 (8)	C19—C20—H20	120.7 (8)
C8—C1—C2—C3	-1.34 (17)	N1—C4—C9—C14	152.27 (11)
C1 <sup>i</sup> —C1—C2—C3	178.88 (12)	C5—C4—C9—C14	-26.95 (17)
C4—N1—C3—C6	2.79 (16)	N1—C4—C9—C10	-25.98 (15)
C4—N1—C3—C2	179.56 (10)	C5—C4—C9—C10	154.81 (11)
C1—C2—C3—N1	-174.44 (10)	C14—C9—C10—C11	2.26 (17)
C1—C2—C3—C6	2.37 (17)	C4—C9—C10—C11	-179.41 (10)
C3—N1—C4—C5	3.29 (16)	C9—C10—C11—C12	-0.35 (18)
C3—N1—C4—C9	-175.95 (9)	C10—C11—C12—C13	-1.57 (19)
C6—N2—C5—C4	3.88 (16)	C11—C12—C13—C14	1.55 (19)
C6—N2—C5—C15	-174.05 (9)	C12—C13—C14—C9	0.41 (18)
N1—C4—C5—N2	-6.97 (17)	C10—C9—C14—C13	-2.28 (17)
C9—C4—C5—N2	172.21 (10)	C4—C9—C14—C13	179.47 (10)
N1—C4—C5—C15	170.75 (10)	N2—C5—C15—C16	115.78 (12)
C9—C4—C5—C15	-10.07 (17)	C4—C5—C15—C16	-62.09 (15)
C5—N2—C6—C3	2.27 (16)	N2—C5—C15—C20	-59.79 (14)
C5—N2—C6—C7	-178.84 (10)	C4—C5—C15—C20	122.33 (12)
N1—C3—C6—N2	-5.86 (17)	C20—C15—C16—C17	0.92 (17)
C2—C3—C6—N2	177.37 (10)	C5—C15—C16—C17	-174.64 (10)
N1—C3—C6—C7	175.23 (10)	C15—C16—C17—C18	-1.36 (18)
C2—C3—C6—C7	-1.54 (16)	C16—C17—C18—C19	0.39 (17)
N2—C6—C7—C8	-179.17 (10)	C17—C18—C19—C20	1.01 (18)
C3—C6—C7—C8	-0.26 (17)	C16—C15—C20—C19	0.46 (18)
C6—C7—C8—C1	1.31 (18)	C5—C15—C20—C19	176.09 (10)
C2—C1—C8—C7	-0.51 (17)	C18—C19—C20—C15	-1.44 (18)
C1 <sup>i</sup> —C1—C8—C7	179.27 (13)		

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

#### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C2—H2 $\cdots$ C18 <sup>ii</sup>	0.977 (15)	2.722 (15)	3.5521 (16)	143.1 (12)
C7—H7 $\cdots$ N2 <sup>iii</sup>	0.979 (15)	2.593 (16)	3.3635 (15)	135.6 (11)
C10—H10 $\cdots$ C3 <sup>iv</sup>	0.974 (13)	2.935 (12)	3.2994 (15)	103.4 (8)
C10—H10 $\cdots$ C6 <sup>iv</sup>	0.974 (13)	2.974 (13)	3.1801 (15)	93.1 (8)
C11—H11 $\cdots$ N1 <sup>iv</sup>	0.963 (15)	2.893 (14)	3.3233 (14)	108.3 (10)
C12—H12 $\cdots$ C18 <sup>v</sup>	0.975 (15)	2.959 (15)	3.6146 (16)	125.6 (10)
C13—H13 $\cdots$ C18 <sup>v</sup>	0.999 (15)	2.978 (15)	3.6141 (16)	122.5 (10)
C16—H16 $\cdots$ N2 <sup>iv</sup>	0.978 (14)	2.815 (14)	3.7256 (14)	155.2 (11)
C17—H17 $\cdots$ C1 <sup>vi</sup>	0.969 (13)	2.993 (14)	3.7019 (16)	131.0 (9)

---

C17—H17···C8 <sup>vi</sup>	0.969 (13)	2.860 (14)	3.6338 (15)	137.5 (9)
C18—H18···N1 <sup>vii</sup>	0.975 (14)	2.808 (14)	3.6217 (14)	141.5 (10)

---

Symmetry codes: (ii)  $x, y+1, z$ ; (iii)  $-x, -y+1, -z+1$ ; (iv)  $x+1, y, z$ ; (v)  $-x+2, -y+1, -z+2$ ; (vi)  $-x+1, -y+1, -z+1$ ; (vii)  $x, y-1, z$ .