

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

A nearly planar arrangement of ions in 4,4'-bipiperidinium tetracyanido-platinate(II) monohydrate

Branson A. Maynard and Richard E. Sykora*

Department of Chemistry, University of South Alabama, Mobile AL 36688-0002, USA

Correspondence e-mail: rsykora@jaguar1.usouthal.edu

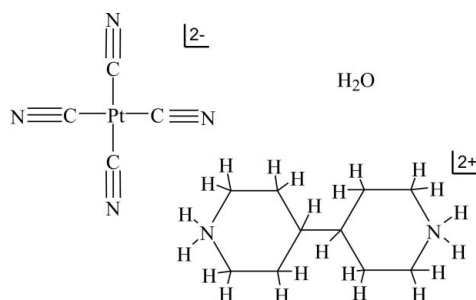
Received 15 November 2007; accepted 30 November 2007

Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.013$ Å; R factor = 0.032; wR factor = 0.088; data-to-parameter ratio = 16.1.

The title compound, $(\text{C}_{10}\text{H}_{22}\text{N}_2)[\text{Pt}(\text{CN})_4]\cdot\text{H}_2\text{O}$, was isolated from solution as a molecular salt. The compound contains discrete 4,4'-bipiperidinium cations and tetracyanoplatinate(II) anions that are involved in a hydrogen-bonding network with one water molecule of hydration. The structure differs from that of the similar acetonitrile solvate, $(\text{C}_{10}\text{H}_{22}\text{N}_2)[\text{Pt}(\text{CN})_4]\cdot 2\text{CH}_3\text{CN}$, in the orientation of the ions relative to one another. The hydrate reported here contains layers of nearly parallel cations and anions with an angle between their mean planes of only 4.35 (11°), while in the acetonitrile solvate the cations and anions are nearly perpendicular to one another (86.1° between mean planes). The crystal showed partial inversion twinning.

Related literature

Organic dications such as 4,4'-bipyridinium and 4,4'-bipiperidinium have been shown to be successful in crystallizing a number of square-planar metallate anions, and a large number of salts containing these two ions have been reported (Lewis & Orpen, 1998; Angeloni & Orpen, 2001; Crawford *et al.*, 2004). For the acetonitrile solvate, with a contrasting arrangement of the ions, see Crawford *et al.* (2004).



Experimental

Crystal data

$(\text{C}_{10}\text{H}_{22}\text{N}_2)[\text{Pt}(\text{CN})_4]\cdot\text{H}_2\text{O}$
 $M_r = 487.48$
 Orthorhombic, $P2_12_12_1$
 $a = 9.5246$ (13) Å
 $b = 11.966$ (3) Å
 $c = 15.411$ (3) Å
 $V = 1756.4$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 8.00$ mm⁻¹
 $T = 290$ (2) K
 $0.63 \times 0.60 \times 0.40$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: numerical (*XPREP* in *SHELXTL*; Bruker, 1998)
 $T_{\min} = 0.014$, $T_{\max} = 0.104$
 3233 independent reflections
 3030 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 3 standard reflections
 frequency: 120 min
 intensity decay: none
 3233 reflections
 201 parameters
 H-atom parameters constrained

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.088$
 $S = 1.10$
 $\Delta\rho_{\max} = 1.74$ e Å⁻³
 $\Delta\rho_{\min} = -0.69$ e Å⁻³
 Absolute structure: (Flack, 1983),
 1371 Friedel pairs
 Flack parameter: 0.39 (10)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N5}-\text{H5A}\cdots\text{O1}^{\text{i}}$	0.90	1.92	2.809 (12)	172
$\text{N5}-\text{H5B}\cdots\text{N2}^{\text{ii}}$	0.90	2.17	2.940 (12)	143
$\text{N5}-\text{H5B}\cdots\text{N4}^{\text{iii}}$	0.90	2.44	3.008 (13)	121
$\text{N6}-\text{H6A}\cdots\text{N4}$	0.90	2.25	2.976 (13)	137
$\text{N6}-\text{H6A}\cdots\text{N2}^{\text{iv}}$	0.90	2.42	3.021 (12)	125
$\text{N6}-\text{H6B}\cdots\text{N3}^{\text{v}}$	0.90	2.13	3.026 (12)	179
$\text{O1}-\text{H1A}\cdots\text{N1}^{\text{vi}}$	0.85	2.10	2.946 (11)	179
$\text{O1}-\text{H1B}\cdots\text{N3}^{\text{v}}$	0.85	2.27	3.125 (10)	180

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *pubCIF* (Westrip, 2007).

The authors gratefully acknowledge the Department of Energy and Oak Ridge National Laboratory for the loan of an Enraf–Nonius CAD-4 X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2073).

References

- Angeloni, A. & Orpen, A. G. (2001). *Chem. Commun.* pp. 343–344.
 Bruker (1998). *SHELXTL*. Version 5.1 for Windows. Bruker AXS Inc., Madison, Wisconsin, USA.
 Crawford, P. C., Gillon, A. L., Green, J., Orpen, A. G., Podesta, T. J. & Pritchard, S. V. (2004). *CrystEngComm*, **6**, 419–428.
 Enraf–Nonius (1993). *CAD-4-PC Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.

Harms, K. & Wocadlo, S. (1996). *XCAD4*. University of Marburg, Germany.

Lewis, G. R. & Orpen, A. G. (1998). *Chem. Commun.* pp.1873–1874.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Westrip, S. P. (2007). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2008). E64, m138-m139 [doi:10.1107/S1600536807064835]

A nearly planar arrangement of ions in 4,4'-bipiperidinium tetracyanidoplatinate(II) monohydrate

B. A. Maynard and R. E. Sykora

Comment

The title compound, $(C_{10}H_{22}N_2)Pt(CN)_4 \cdot H_2O$, was obtained as an unexpected product during a reaction that attempted to prepare a praseodymium tetracyanoplatinate incorporating 4,4'-bipiperidine.

The structure of (**I**) consists of separated 4,4'-bipiperidinium dications and tetracyanoplatinate anions, additionally one water molecule of crystallization is also present. Fig. 1 shows an illustration of the units of the structure along with the atomic labeling scheme. The 4,4'-bipiperidinium cations and tetracyanoplatinate anions lie in approximately the *ab* crystallographic planes and contain multiple hydrogen bonding interactions as can be seen in Fig. 2. Each of the approximately square planar anions is hydrogen bonded to four cations and each cation is also hydrogen bonded to four anions. See Table 1 for bond distances and angles of these hydrogen bonding interactions. The mean plane that passes through the 4,4'-bipiperidinium cation makes an angle of $4.35(11)^\circ$ with the mean plane of the tetracyanoplatinate anion in the structure, illustrating the nearly parallel nature of these groups relative to one another. Small cavities in these two dimensional planes are filled with water molecules that hydrogen bond within the plane to N1 and N3 atoms of the tetracyanoplatinate anions. Additional hydrogen bonding interactions are also present between the water molecules in one plane and H5A atoms of neighboring planes. See Table 1 for details of these H-bonding interactions.

Several major structural differences exist between **I** and the previously reported $(C_{10}H_{22}N_2)Pt(CN)_4 \cdot 2CH_3CN$, **II** (Crawford *et al.*, 2004). While compound **I** contains a nearly parallel arrangement of the cations and anions, the 4,4'-bipiperidinium cations in **II** are nearly perpendicular to the tetracyanoplatinate anions. The angle between the mean planes formed by the two groups in **II** is 86.1° . This packing arrangement of the cations and anions in **II** leaves relatively large holes in the structure that accommodate acetonitrile solvate molecules. In **I**, the smaller cavities contain water molecules.

Experimental

$K_2Pt(CN)_4 \cdot 3H_2O$ (Alfa Aesar, 99.9%), 4,4'-bipiperidine dihydrochloride (Aldrich, 97%), and $Pr(NO_3)_3 \cdot 6H_2O$ (Strem Chemicals, 99.9%) were used as received without further purification. $K_2Pt(CN)_4 \cdot 3H_2O$ (1 ml, 0.14 M) in 90%:10% $CH_3CN:H_2O$ was added to an CH_3CN solution of $Pr(NO_3)_3 \cdot 6H_2O$ (1 ml, 0.10 M). 4,4'-bipiperidine dihydrochloride (1 ml, 0.077 M) in CH_3CN was then layered on this solution. Slow evaporation of the solvents over a period of several days resulted in colorless, prismatic crystals of the title compound.

Refinement

H atoms of the 4,4'-bipiperidinium cation were placed in calculated positions and allowed to ride during subsequent refinement, with $U_{iso}(H) = 1.2U_{eq}(C)$ and C—H distances of 0.97 Å for H atoms bonded to the C atoms and $U_{iso}(H) = 1.2U_{eq}(N)$ and N—H distances of 0.90 Å for the H atoms bonded to the N atoms. The H atoms on the water molecule were not located in the difference map, but were placed in calculated positions with O—H distances of 0.85 Å and $U_{iso}(H) = 1.2U_{eq}(O)$. The

supplementary materials

H atoms were not allowed to move during refinement. The crystal of **I** that was used for the diffraction study was found to be a racemic twin and therefore the refinement was carried out taking into account the inverted component.

Figures

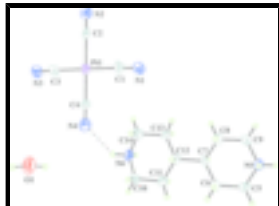


Fig. 1. The molecular structure of (**I**), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

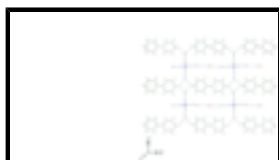


Fig. 2. A representation of two-dimensional layers of 4,4'-bipiperidinium cations, tetracyanoplatinate anions and water molecules found in the *ab* plane of (**I**).

4,4'-bipiperidinium tetracyanidoplatinate monohydrate

Crystal data

(C₁₀H₂₂N₂)[Pt(CN)₄]·H₂O

M_r = 487.48

Orthorhombic, *P*2₁2₁2₁

Hall symbol: P 2ac 2ab

a = 9.5246 (13) Å

b = 11.966 (3) Å

c = 15.411 (3) Å

V = 1756.4 (6) Å³

Z = 4

*F*₀₀₀ = 944

D_x = 1.843 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 8.2–11.7°

μ = 8.00 mm⁻¹

T = 290 (2) K

Rectangular prism, colorless

0.63 × 0.60 × 0.40 mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 290(2) K

θ/2θ scans

Absorption correction: analytical
(XPREP; Bruker, 1998)

*T*_{min} = 0.014, *T*_{max} = 0.104

3591 measured reflections

3233 independent reflections

3030 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.025

θ_{max} = 25.4°

θ_{min} = 2.2°

h = 0 → 11

k = 0 → 14

l = -18 → 18

3 standard reflections

every 120 min

intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 1.1497P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.10$	$\Delta\rho_{\max} = 1.74 \text{ e } \text{\AA}^{-3}$
3233 reflections	$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$
201 parameters	Extinction correction: SHELXL97,
Primary atom site location: structure-invariant direct methods	$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0087 (5)
Hydrogen site location: inferred from neighbouring sites	Absolute structure: (Flack, 1983), 1371 Friedel pairs
	Flack parameter: 0.39 (10)

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pt1	0.78147 (3)	0.08308 (3)	0.143832 (18)	0.03168 (13)
C1	0.7890 (11)	0.2485 (8)	0.1344 (5)	0.0431 (18)
C2	0.9911 (9)	0.0805 (8)	0.1605 (6)	0.042 (2)
C3	0.7736 (10)	-0.0821 (7)	0.1573 (5)	0.0381 (17)
C4	0.5765 (8)	0.0854 (8)	0.1287 (6)	0.0378 (18)
C5	0.1305 (10)	0.8253 (9)	0.1135 (7)	0.044 (2)
H5C	0.0523	0.8616	0.0846	0.053*
H5D	0.1119	0.8247	0.1754	0.053*
C6	0.1454 (11)	0.7054 (7)	0.0804 (6)	0.038 (2)
H6C	0.0598	0.6646	0.0928	0.046*
H6D	0.1580	0.7066	0.0179	0.046*
C7	0.2665 (10)	0.6462 (6)	0.1210 (6)	0.0330 (18)
H7A	0.2521	0.6462	0.1840	0.040*
C8	0.3971 (12)	0.7103 (8)	0.1024 (8)	0.048 (3)
H8A	0.4145	0.7101	0.0404	0.058*

supplementary materials

H8B	0.4760	0.6740	0.1306	0.058*
C9	0.3868 (11)	0.8308 (8)	0.1341 (9)	0.057 (3)
H9A	0.3787	0.8315	0.1969	0.068*
H9B	0.4718	0.8707	0.1185	0.068*
C10	0.1598 (12)	0.3338 (8)	0.0928 (6)	0.037 (2)
H10A	0.0749	0.2943	0.1091	0.045*
H10B	0.1741	0.3237	0.0309	0.045*
C11	0.1436 (11)	0.4557 (7)	0.1125 (6)	0.035 (2)
H11A	0.1209	0.4648	0.1735	0.042*
H11B	0.0663	0.4857	0.0789	0.042*
C12	0.2812 (11)	0.5236 (7)	0.0912 (6)	0.0336 (17)
H12A	0.2954	0.5228	0.0283	0.040*
C13	0.4053 (10)	0.4656 (8)	0.1339 (7)	0.039 (2)
H13A	0.4914	0.5003	0.1138	0.046*
H13B	0.3997	0.4765	0.1962	0.046*
C14	0.4117 (12)	0.3420 (9)	0.1151 (7)	0.045 (3)
H14A	0.4288	0.3303	0.0537	0.053*
H14B	0.4887	0.3087	0.1472	0.053*
N1	0.7856 (10)	0.3441 (7)	0.1291 (6)	0.059 (2)
N2	1.1070 (9)	0.0788 (8)	0.1678 (8)	0.070 (3)
N3	0.7651 (10)	-0.1777 (6)	0.1673 (5)	0.0474 (19)
N4	0.4565 (10)	0.0854 (9)	0.1178 (7)	0.066 (3)
N5	0.2634 (9)	0.8885 (5)	0.0956 (6)	0.0413 (18)
H5A	0.2755	0.8949	0.0379	0.050*
H5B	0.2565	0.9578	0.1179	0.050*
N6	0.2788 (8)	0.2880 (6)	0.1401 (5)	0.0383 (15)
H6A	0.2849	0.2142	0.1296	0.046*
H6B	0.2648	0.2973	0.1974	0.046*
O1	0.2238 (9)	0.0836 (6)	0.4148 (5)	0.0655 (19)
H1A	0.2210	0.0146	0.4017	0.079*
H1B	0.2269	0.1486	0.3921	0.079*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.03236 (18)	0.02454 (17)	0.03815 (18)	-0.00054 (13)	0.00362 (13)	0.00082 (13)
C1	0.051 (5)	0.028 (4)	0.050 (4)	-0.005 (4)	-0.002 (5)	-0.002 (4)
C2	0.041 (5)	0.009 (3)	0.075 (6)	0.006 (4)	0.009 (4)	0.001 (5)
C3	0.042 (4)	0.037 (5)	0.035 (3)	-0.007 (5)	-0.003 (4)	-0.002 (4)
C4	0.027 (4)	0.019 (3)	0.067 (5)	0.002 (4)	0.002 (4)	0.010 (5)
C5	0.021 (4)	0.037 (5)	0.075 (6)	0.003 (4)	0.003 (4)	0.001 (4)
C6	0.034 (5)	0.022 (4)	0.058 (5)	0.001 (4)	-0.006 (5)	-0.005 (4)
C7	0.034 (5)	0.015 (4)	0.050 (4)	-0.004 (3)	-0.002 (4)	-0.004 (3)
C8	0.036 (5)	0.027 (5)	0.082 (7)	0.003 (4)	-0.002 (5)	-0.005 (4)
C9	0.032 (5)	0.031 (5)	0.107 (9)	-0.002 (4)	-0.017 (6)	-0.007 (6)
C10	0.037 (5)	0.024 (4)	0.051 (5)	-0.007 (4)	-0.009 (4)	0.005 (4)
C11	0.032 (4)	0.024 (4)	0.050 (5)	-0.002 (4)	-0.002 (4)	0.010 (3)
C12	0.031 (4)	0.027 (4)	0.042 (4)	0.000 (4)	-0.001 (4)	-0.002 (3)

C13	0.024 (4)	0.028 (4)	0.064 (6)	0.002 (3)	-0.002 (5)	-0.001 (5)
C14	0.032 (5)	0.029 (5)	0.072 (7)	-0.004 (4)	0.006 (4)	0.000 (4)
N1	0.043 (4)	0.035 (5)	0.097 (7)	-0.008 (4)	0.005 (6)	0.004 (4)
N2	0.035 (5)	0.034 (4)	0.140 (9)	0.002 (4)	0.007 (5)	-0.003 (6)
N3	0.051 (5)	0.030 (4)	0.061 (5)	-0.006 (4)	-0.001 (4)	0.007 (3)
N4	0.049 (5)	0.030 (4)	0.118 (8)	0.000 (5)	-0.005 (5)	0.006 (6)
N5	0.036 (4)	0.021 (4)	0.067 (5)	0.002 (3)	-0.004 (4)	0.000 (3)
N6	0.040 (4)	0.021 (3)	0.054 (4)	0.003 (3)	0.005 (5)	0.000 (3)
O1	0.085 (5)	0.037 (3)	0.074 (5)	0.008 (5)	-0.007 (4)	-0.005 (4)

Geometric parameters (Å, °)

Pt1—C4	1.967 (8)	C9—H9B	0.9700
Pt1—C1	1.986 (9)	C10—N6	1.455 (12)
Pt1—C3	1.989 (8)	C10—C11	1.498 (14)
Pt1—C2	2.013 (9)	C10—H10A	0.9700
C1—N1	1.147 (12)	C10—H10B	0.9700
C2—N2	1.110 (13)	C11—C12	1.577 (14)
C3—N3	1.157 (10)	C11—H11A	0.9700
C4—N4	1.155 (12)	C11—H11B	0.9700
C5—N5	1.500 (12)	C12—C13	1.520 (14)
C5—C6	1.529 (13)	C12—H12A	0.9800
C5—H5C	0.9700	C13—C14	1.508 (13)
C5—H5D	0.9700	C13—H13A	0.9700
C6—C7	1.491 (13)	C13—H13B	0.9700
C6—H6C	0.9700	C14—N6	1.472 (13)
C6—H6D	0.9700	C14—H14A	0.9700
C7—C8	1.490 (14)	C14—H14B	0.9700
C7—C12	1.543 (10)	N5—H5A	0.9000
C7—H7A	0.9800	N5—H5B	0.9000
C8—C9	1.526 (14)	N6—H6A	0.9000
C8—H8A	0.9700	N6—H6B	0.9000
C8—H8B	0.9700	O1—H1A	0.8503
C9—N5	1.487 (13)	O1—H1B	0.8532
C9—H9A	0.9700		
C4—Pt1—C1	90.7 (4)	C11—C10—H10A	109.6
C4—Pt1—C3	89.4 (4)	N6—C10—H10B	109.6
C1—Pt1—C3	178.2 (3)	C11—C10—H10B	109.6
C4—Pt1—C2	179.5 (4)	H10A—C10—H10B	108.1
C1—Pt1—C2	89.4 (4)	C10—C11—C12	112.0 (9)
C3—Pt1—C2	90.5 (4)	C10—C11—H11A	109.2
N1—C1—Pt1	176.3 (10)	C12—C11—H11A	109.2
N2—C2—Pt1	178.5 (10)	C10—C11—H11B	109.2
N3—C3—Pt1	177.5 (9)	C12—C11—H11B	109.2
N4—C4—Pt1	178.3 (10)	H11A—C11—H11B	107.9
N5—C5—C6	109.5 (8)	C13—C12—C7	112.1 (8)
N5—C5—H5C	109.8	C13—C12—C11	108.7 (7)
C6—C5—H5C	109.8	C7—C12—C11	110.7 (8)
N5—C5—H5D	109.8	C13—C12—H12A	108.4

supplementary materials

C6—C5—H5D	109.8	C7—C12—H12A	108.4
H5C—C5—H5D	108.2	C11—C12—H12A	108.4
C7—C6—C5	112.2 (8)	C14—C13—C12	113.4 (9)
C7—C6—H6C	109.2	C14—C13—H13A	108.9
C5—C6—H6C	109.2	C12—C13—H13A	108.9
C7—C6—H6D	109.2	C14—C13—H13B	108.9
C5—C6—H6D	109.2	C12—C13—H13B	108.9
H6C—C6—H6D	107.9	H13A—C13—H13B	107.7
C8—C7—C6	108.7 (7)	N6—C14—C13	110.2 (9)
C8—C7—C12	110.9 (8)	N6—C14—H14A	109.6
C6—C7—C12	113.4 (8)	C13—C14—H14A	109.6
C8—C7—H7A	107.9	N6—C14—H14B	109.6
C6—C7—H7A	107.9	C13—C14—H14B	109.6
C12—C7—H7A	107.9	H14A—C14—H14B	108.1
C7—C8—C9	111.8 (9)	C9—N5—C5	111.1 (7)
C7—C8—H8A	109.3	C9—N5—H5A	109.4
C9—C8—H8A	109.3	C5—N5—H5A	109.4
C7—C8—H8B	109.3	C9—N5—H5B	109.4
C9—C8—H8B	109.3	C5—N5—H5B	109.4
H8A—C8—H8B	107.9	H5A—N5—H5B	108.0
N5—C9—C8	111.2 (9)	C10—N6—C14	111.9 (7)
N5—C9—H9A	109.4	C10—N6—H6A	109.2
C8—C9—H9A	109.4	C14—N6—H6A	109.2
N5—C9—H9B	109.4	C10—N6—H6B	109.2
C8—C9—H9B	109.4	C14—N6—H6B	109.2
H9A—C9—H9B	108.0	H6A—N6—H6B	107.9
N6—C10—C11	110.2 (8)	H1A—O1—H1B	142.2
N6—C10—H10A	109.6		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N5—H5A \cdots O1 ⁱ	0.90	1.92	2.809 (12)	172
N5—H5B \cdots N2 ⁱⁱ	0.90	2.17	2.940 (12)	143
N5—H5B \cdots N4 ⁱⁱⁱ	0.90	2.44	3.008 (13)	121
N6—H6A \cdots N4	0.90	2.25	2.976 (13)	137
N6—H6A \cdots N2 ^{iv}	0.90	2.42	3.021 (12)	125
N6—H6B \cdots N3 ^v	0.90	2.13	3.026 (12)	179
O1—H1A \cdots N1 ^{vi}	0.85	2.10	2.946 (11)	179.4
O1—H1B \cdots N3 ^v	0.85	2.27	3.125 (10)	179.6

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

Fig. 2

