

## Bis(2,2'-bipyridyl- $\kappa^2N,N'$ )dichlorido-rhodium(III) perchlorate

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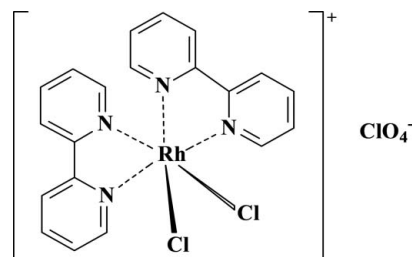
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å; disorder in solvent or counterion;  $R$  factor = 0.042;  $wR$  factor = 0.081; data-to-parameter ratio = 23.5.

The asymmetric unit of the title compound,  $[\text{RhCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\text{ClO}_4$ , consists of one unit of the cationic complex  $[\text{RhCl}_2(\text{bipy})_2]^+$  and one uncoordinated perchlorate anion. The  $\text{Rh}^{\text{III}}$  atom is coordinated by four N atoms from two bipyridyl ligands and two Cl atoms, forming a distorted octahedral environment. The Cl ligands are *cis*. Two intramolecular  $\text{C}-\text{H}\cdots\text{Cl}$  hydrogen bonds occur in the cationic complex. In the crystal, molecules are linked together by a hydrogen-bond network involving the H atoms of bipyridyl rings and perchlorate anions. An O atom of the perchlorate anion is disordered over two sites, with an occupancy-factor ratio of 0.78 (3):0.22 (3).

### Related literature

For potential applications of noble metal complexes of pyridyl ligands in biochemistry, catalysis and anticancer activity, see: Chifotides *et al.* (2004); Mbaye *et al.* (2003); Karidi *et al.* (2005); Tan *et al.* (2005). For their photochemical and photo-physical properties, see: Forster & Rund (2003); Arachchige *et al.* (2008) and for their electrochemical properties, see: Rasmussen *et al.* (1990). For related structures, see: Al-Noaimi & Haddad (2007); Andansen & Josephsen (1971); Choudhury *et al.* (2006); De Munno *et al.* (1993); Figgis *et al.* (1985); Fontaine (2001); Gao & Ng (2010); Kramer & Straehle (1986); Sofetis *et al.* (2006); Strenger *et al.* (2000). For similar structures with platinum group metals, see: Lahuerta *et al.* (1991); Kim *et al.* (2009); Helberg *et al.* (1996); Prajapati *et al.* (2008); Eggleston *et al.* (1985).



### Experimental

#### Crystal data

$[\text{RhCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\text{ClO}_4$	$V = 2202.33$ (8) Å <sup>3</sup>
$M_r = 585.63$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 11.0344$ (2) Å	$\mu = 1.18$ mm <sup>-1</sup>
$b = 11.6796$ (2) Å	$T = 293$ K
$c = 17.0884$ (3) Å	$0.19 \times 0.16 \times 0.12$ mm

#### Data collection

Agilent Xcalibur Ruby Gemini diffractometer	12115 measured reflections
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2011)	6922 independent reflections
$T_{\min} = 0.973$ , $T_{\max} = 1$	5569 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.026$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	$\Delta\rho_{\text{max}} = 0.75$ e Å <sup>-3</sup>
$wR(F^2) = 0.081$	$\Delta\rho_{\text{min}} = -0.25$ e Å <sup>-3</sup>
$S = 1.02$	Absolute structure: Flack (1983),
6922 reflections	2630 Friedel pairs
294 parameters	Flack parameter: 0.47 (3)
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

Rh1—N2	2.019 (2)	Rh1—N4	2.038 (3)
Rh1—N1	2.023 (2)	Rh1—Cl3	2.3291 (9)
Rh1—N3	2.037 (2)	Rh1—Cl2	2.3344 (9)
N1—Rh1—N3	174.22 (10)	Cl3—Rh1—Cl2	91.18 (4)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
C1—H1 $\cdots$ Cl2	0.93	2.70	3.301 (4)	123
C11—H11 $\cdots$ Cl3	0.93	2.76	3.358 (4)	123
C3—H3 $\cdots$ O1 <sup>i</sup>	0.93	2.29	3.192 (5)	164
C8—H8 $\cdots$ O1 <sup>ii</sup>	0.93	2.56	3.142 (6)	121
C9—H9 $\cdots$ O1 <sup>ii</sup>	0.93	2.58	3.154 (6)	120
C13—H13 $\cdots$ O4 <sup>iii</sup>	0.93	2.56	3.427 (6)	155

Symmetry codes: (i)  $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $x - \frac{1}{2}, -y - \frac{1}{2}, -z + 1$ ; (iii)  $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2059).

## References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Al-Noaimi, M. & Haddad, S. F. (2007). *Acta Cryst.* **E63**, m2332.
- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Andansen, P. & Josephsen, J. (1971). *Acta Chem. Scand.* **25**, 3255–3260.
- Arachchige, S. M., Brown, J. & Brewster, K. J. (2008). *J. Photochem. Photobiol. A*, **197**, 13–17.
- Chifotides, H. T., Fu, P. K. L., Dunbar, K. R. & Turro, C. (2004). *Inorg. Chem.* **43**, 1175–1183.
- Choudhury, S. R., Dutta, A., Mukhopadhyay, S., Lu, L.-P. & Zhu, M.-L. (2006). *Acta Cryst.* **E62**, m1489–m1491.
- De Munno, G., Nicolò, F. & Julve, M. (1993). *Acta Cryst.* **C49**, 1049–1052.
- Eggleston, D. S., Goldsby, K. A., Hodgson, D. J. & Meyer, T. J. (1985). *Inorg. Chem.* **24**, 4573–4580.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Figgis, B. N., Reynolds, P. A. & White, A. H. (1985). *Inorg. Chem.* **24**, 3762–3770.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Fontaine, F. G. (2001). *Acta Cryst.* **E57**, m270–m271.
- Forster, L. C. & Rund, J. V. (2003). *Inorg. Chem. Commun.* **6**, 78–81.
- Gao, S. & Ng, S. W. (2010). *Acta Cryst.* **E66**, m1692.
- Helberg, L. E., Orth, S. D., Sabat, M. & Harman, W. D. (1996). *Inorg. Chem.* **35**, 5584–5594.
- Karidi, K., Garoufis, A., Tsipis, A., Hadjiliadis, N., Dulk, H. & Reedijk, J. (2005). *Dalton Trans.* pp. 1176–1187.
- Kim, N.-H., Hwang, I.-C. & Ha, K. (2009). *Acta Cryst.* **E65**, m180.
- Kramer, T. & Straehle, J. (1986). *Z. Naturforsch. Teil B*, **41**, 692–696.
- Lahuerta, P., Latorre, J., Martínez-Máñez, R., García-Granda, S. & Gómez-Beltrán, F. (1991). *Acta Cryst.* **C47**, 519–522.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Mbaye, M. D., Demersement, B., Reneaud, J.-L., Toupet, L. & Bruneau, C. (2003). *Angew. Chem. Int. Ed.* **42**, 5066–5068.
- Prajapati, R., Yadan, V. K., Dubey, S. K., Durham, B. & Mishra, L. (2008). *Indian J. Chem. Sect. A*, **47**, 1780–1787.
- Rasmussen, S. C., Richter, M. M., Yi, E., Place, H. & Brewer, K. J. (1990). *Inorg. Chem.* **29**, 3926–3932.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sofetis, A., Raptopoulou, C. P., Terzis, A. & Zafiroopoulos, T. F. (2006). *Inorg. Chim. Acta*, **359**, 3389–3395.
- Strenger, I., Rosu, T. & Negoiu, M. (2000). *Z. Kristallogr. New Cryst. Struct.* **215**, 489–490.
- Tan, L. F., Chao, H., Li, H., Liu, Y. J., Sun, B., Wei, W. & Ji, L. N. (2005). *J. Inorg. Biochem.* **99**, 513–520.

## supporting information

*Acta Cryst.* (2012). E68, m713–m714 [doi:10.1107/S1600536812018685]

**Bis(2,2'-bipyridyl- $\kappa^2N,N'$ )dichloridorhodium(III) perchlorate**

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**S1. Comment**

In recent years, noble metal complexes of pyridyl ligands have received much attention because of their rich electrochemical (Rasmussen *et al.*, 1990, photophysical (Forster & Rund, 2003) and photochemical (Arachchige *et al.*, 2008) properties, and their potential applications in catalysis (Mbaye *et al.*, 2003), biochemistry (Tan *et al.*, 2005; Chifotides *et al.*, 2004) and anticancer activity (Karidi *et al.*, 2005). Bipyridine (bipy) is one of the most commonly used bidentate ligand of this type in the formation of wide variety of transition metal complexes with a general formula of  $[M^II(bipy)_2X_2]$  ( $M = Co, Ni, Mn, Fe$ ) in which  $X$  is an coordinated anionic ligand such as CN, SCN and chloride (De Munno *et al.*, 1993; Eggleston *et al.*, 1985; Kramer & Straehle, 1986; Al-Noaimi and Haddad, 2007; Fontaine, 2001; Choudhury *et al.*, 2006; Gao & Ng, 2010) and complexes with cationic part  $[M^III Cl_2(bipy)_2]^+$  ( $M = Re, Ru, Co, Ga$ ) and any counter anion like Cl<sup>-</sup>, PF<sub>6</sub><sup>-</sup> (Figgis *et al.*, 1985; Sofetis *et al.*, 2006; Andansen & Josephsen, 1971; Strenger *et al.*, 2000; Kim *et al.*, 2009; Prajapati *et al.*, 2008; Helberg *et al.*, 1996). The complex  $[RhCl_2(bipy)_2]Cl \cdot 2H_2O$  has also been obtained and crystallographically determined by Lahuerta *et al.*, 1991. Yet, no crystal structure has been reported for the cationic complex  $cis-[Rh(bipy)_2Cl_2]^+$  in its perchlorate form as counter anion, therefore, we report the crystal structure of compound **(I)**.

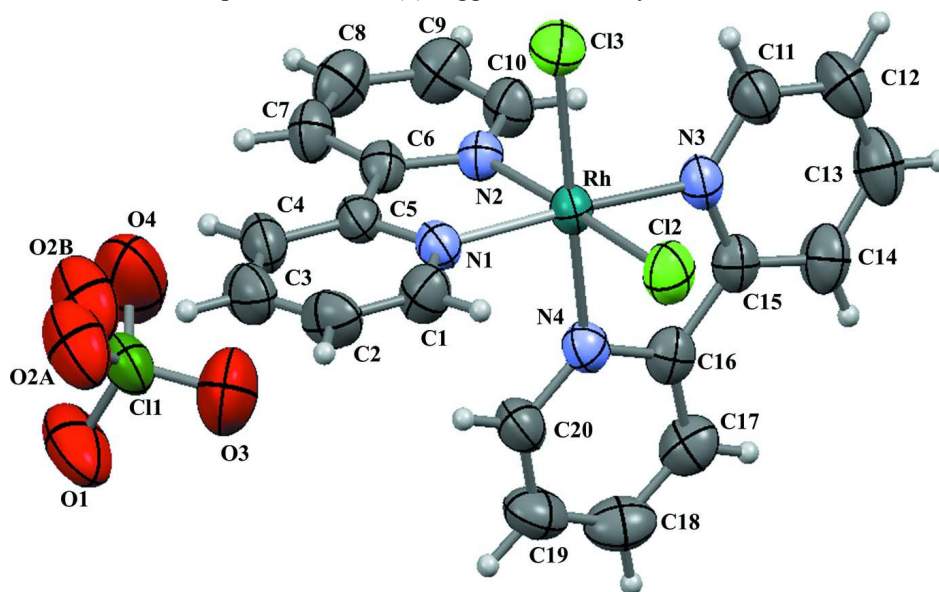
Complex **(I)** crystallizes in the orthorhombic space group  $P2_12_12_1$ . The molecular structure of **(I)** depicted in Figure 1. It has a distorted octahedral geometry with the two chloride ions in *cis* positions. Selected bond lengths for the complex are given in Table 1. The Rh–N axial bond distance (2.038 (3) Å) is slightly longer than Rh–N equatorial bonds (average 2.026 (5) Å). Its may be well compared with the negligible difference between equatorial and axial M–N bonds distances seen in the analogous complexes of platinum metal group (Lahuerta *et al.*, 1991; Kim *et al.*, 2009; Helberg *et al.*, 1996; Prajapati *et al.*, 2008; Eggleston *et al.*, 1985), but greater distortion observed in majority of transition metal complexes (Strenger *et al.*, 2000; Fontaine, 2001; Kramer & Straehle, 1986; Sofetis *et al.*, 2006; Figgis *et al.*, 1985; Choudhury *et al.*, 2006; Gao & Ng, 2010). The Rh–Cl bond distances in **(I)** are 2.3291 (9) (equatorial) and 2.3344 (9) Å (axial). The N<sub>eq</sub>–Rh–N<sub>eq</sub> angle is 174.22 (10)° and its distorted from linearity by approximately 6°. Also Cl<sub>eq</sub>–Rh–Cl<sub>ax</sub> angle (91.18 (4)°) is nearly octahedral. The *cis* isomerization of cationic complex  $[RhCl_2(bipy)_2]^+$  is stabilized by short contacts. Coordinated chlorides which situated in *cis* position to respect to each other make up short contact Cl2...H1 and Cl3...H11 with distances 2.7 and 2.76 Å, respectively (Tabl. 2). The crystal lattice of **(I)** is made up of well separated ClO<sub>4</sub><sup>-</sup> anions and  $[RhCl_2(bipy)_2]^+$  cations. The perchlorate anion contribute to forming extensive hydrogen bonding net linked together cationic and anionic parts of the structure. The interactions involve the hydrogen bonding between H atoms of bipy ring with oxygen atoms uncoordinated perchlorate anion. Each oxygen atoms participates in four hydrogen bonds to H atoms of aryl groups (Tabl. 2). The atom O2 of the perchlorate anion is disordered and splitted over two sites with refined occupancy ratio of 0.78 (3):0.22 (3).

## S2. Experimental

To a solution of  $\text{RhCl}_3 \cdot x\text{H}_2\text{O}$  (0.05 g, 0.231 mmol) and  $\text{KClO}_4$  (0.09 g, 0.693 mmol) in  $\text{H}_2\text{O}$  (10 ml) was added 2,2'-bipyridine (0.04 g, 0.462 mmol) in  $\text{CH}_3\text{OH}$  (10 ml), and was stirred for 2 h to yellow solution resulted. Yellowish rhombohedral crystals suitable for X-ray analysis were obtained by slow evaporation during two weeks.  $\text{RhCl}_3 \cdot x\text{H}_2\text{O}$  purchased from Johnson Matthey, all other reagents was obtained commercially from Sigma-Aldrich and used without further purification.

## S3. Refinement

H atoms were positioned geometrically and allowed to ride on their respective parent atoms [ $\text{C}-\text{H} = 0.93 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ]. The value of the Flack parameter, 0.47 (3) suggests that the crystal is a racemic twin.



**Figure 1**

A view of  $[\text{RhCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\text{ClO}_4$  asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

### Bis(2,2'-bipyridyl- $\kappa^2N,N'$ )dichloridorhodium(III) perchlorate

#### Crystal data

$[\text{RhCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\text{ClO}_4$

$M_r = 585.63$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 11.0344 (2) \text{ \AA}$

$b = 11.6796 (2) \text{ \AA}$

$c = 17.0884 (3) \text{ \AA}$

$V = 2202.33 (8) \text{ \AA}^3$

$Z = 4$

$F(000) = 1168$

$D_x = 1.766 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.7107 \text{ \AA}$

Cell parameters from 4926 reflections

$\theta = 3.5\text{--}32.1^\circ$

$\mu = 1.18 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Rhombohedron, yellow

$0.19 \times 0.16 \times 0.12 \text{ mm}$

#### Data collection

Agilent Xcalibur Ruby Gemini  
diffractometer

Radiation source: Enhance (Mo) X-ray Source  
Graphite monochromator

Detector resolution:  $10.2673 \text{ pixels mm}^{-1}$   
 $\omega$  scans

Absorption correction: multi-scan  
(*CrysAlis PRO*; Agilent, 2011)

$T_{\min} = 0.973$ ,  $T_{\max} = 1$   
 12115 measured reflections  
 6922 independent reflections  
 5569 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$

$\theta_{\max} = 32.1^\circ$ ,  $\theta_{\min} = 3.5^\circ$   
 $h = -8 \rightarrow 16$   
 $k = -17 \rightarrow 10$   
 $l = -22 \rightarrow 25$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.081$   
 $S = 1.02$   
 6922 reflections  
 294 parameters  
 0 restraints  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0292P)^2 + 0.1669P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.75 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 2630 Friedel  
 pairs  
 Absolute structure parameter: 0.47 (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>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Rh1	0.98325 (2)	0.241474 (19)	0.367163 (12)	0.04095 (7)	
Cl1	0.97063 (10)	-0.18258 (8)	0.63515 (5)	0.0629 (2)	
Cl2	1.10302 (9)	0.40548 (7)	0.35644 (6)	0.0609 (2)	
Cl3	0.80775 (9)	0.35183 (8)	0.37395 (6)	0.0603 (2)	
N1	0.9798 (2)	0.2369 (2)	0.48547 (13)	0.0439 (5)	
N2	0.8794 (2)	0.1010 (2)	0.38286 (14)	0.0426 (6)	
N3	0.9942 (3)	0.2300 (2)	0.24844 (13)	0.0467 (6)	
N4	1.1338 (3)	0.1414 (2)	0.35733 (15)	0.0455 (6)	
O1	1.0370 (4)	-0.2722 (3)	0.6687 (2)	0.1228 (15)	
O2A	0.9396 (14)	-0.1068 (7)	0.6981 (7)	0.102 (3)	0.78 (3)
O2B	0.891 (4)	-0.123 (3)	0.6744 (18)	0.102 (3)	0.22 (3)
O3	1.0402 (4)	-0.1234 (3)	0.57914 (19)	0.1075 (12)	
O4	0.8704 (4)	-0.2320 (5)	0.5957 (2)	0.1495 (17)	
C1	1.0374 (3)	0.3097 (3)	0.5332 (2)	0.0557 (9)	
H1	1.0805	0.3705	0.5117	0.067*	
C2	1.0344 (4)	0.2968 (4)	0.6128 (2)	0.0654 (11)	
H2	1.0755	0.3481	0.6448	0.078*	
C3	0.9706 (4)	0.2082 (4)	0.6450 (2)	0.0672 (11)	
H3	0.968	0.1985	0.699	0.081*	
C4	0.9098 (4)	0.1331 (3)	0.59588 (19)	0.0586 (9)	
H4	0.8657	0.0725	0.6166	0.07*	

C5	0.9153 (3)	0.1492 (3)	0.51615 (17)	0.0446 (7)
C6	0.8570 (3)	0.0741 (2)	0.45826 (17)	0.0442 (7)
C7	0.7849 (4)	-0.0179 (3)	0.4774 (2)	0.0546 (9)
H7	0.7708	-0.036	0.5297	0.065*
C8	0.7341 (4)	-0.0825 (3)	0.4193 (2)	0.0665 (11)
H8	0.6858	-0.1452	0.4316	0.08*
C9	0.7550 (4)	-0.0539 (3)	0.3429 (2)	0.0637 (10)
H9	0.72	-0.0963	0.3028	0.076*
C10	0.8279 (3)	0.0380 (3)	0.3260 (2)	0.0535 (9)
H10	0.8421	0.0572	0.274	0.064*
C11	0.9169 (4)	0.2771 (3)	0.1977 (2)	0.0629 (10)
H11	0.8496	0.3163	0.2166	0.075*
C12	0.9344 (4)	0.2689 (4)	0.1183 (2)	0.0719 (11)
H12	0.8797	0.3022	0.0837	0.086*
C13	1.0325 (5)	0.2118 (3)	0.0909 (2)	0.0726 (13)
H13	1.0453	0.2058	0.0373	0.087*
C14	1.1141 (4)	0.1621 (3)	0.1429 (2)	0.0632 (10)
H14	1.182	0.1231	0.1249	0.076*
C15	1.0912 (3)	0.1724 (3)	0.22194 (18)	0.0461 (8)
C16	1.1686 (3)	0.1207 (3)	0.28234 (18)	0.0464 (8)
C17	1.2693 (4)	0.0539 (3)	0.2680 (2)	0.0595 (9)
H17	1.2956	0.0422	0.2169	0.071*
C18	1.3302 (4)	0.0049 (3)	0.3289 (3)	0.0698 (12)
H18	1.3968	-0.042	0.3196	0.084*
C19	1.2925 (4)	0.0256 (3)	0.4035 (3)	0.0686 (11)
H19	1.3331	-0.0074	0.4455	0.082*
C20	1.1956 (4)	0.0945 (3)	0.4161 (2)	0.0563 (9)
H20	1.1715	0.1094	0.4673	0.068*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Rh1	0.04542 (12)	0.04254 (12)	0.03489 (10)	-0.00107 (11)	-0.00011 (10)	0.00272 (10)
Cl1	0.0798 (6)	0.0651 (5)	0.0438 (4)	0.0109 (5)	-0.0048 (5)	0.0050 (4)
Cl2	0.0664 (6)	0.0557 (5)	0.0606 (5)	-0.0147 (4)	0.0015 (5)	0.0072 (4)
Cl3	0.0580 (5)	0.0612 (5)	0.0618 (5)	0.0098 (4)	0.0026 (5)	0.0058 (5)
N1	0.0474 (14)	0.0471 (13)	0.0370 (11)	0.0040 (18)	-0.0028 (10)	0.0026 (10)
N2	0.0443 (15)	0.0422 (13)	0.0414 (15)	-0.0005 (11)	0.0015 (12)	0.0017 (11)
N3	0.0537 (16)	0.0491 (14)	0.0375 (12)	-0.0050 (17)	-0.0008 (11)	0.0049 (11)
N4	0.0483 (16)	0.0430 (13)	0.0451 (15)	-0.0016 (12)	-0.0021 (13)	0.0042 (12)
O1	0.157 (4)	0.120 (3)	0.091 (2)	0.073 (3)	0.009 (2)	0.032 (2)
O2A	0.160 (8)	0.084 (3)	0.063 (4)	0.040 (4)	0.005 (4)	-0.011 (3)
O2B	0.160 (8)	0.084 (3)	0.063 (4)	0.040 (4)	0.005 (4)	-0.011 (3)
O3	0.123 (3)	0.108 (3)	0.092 (2)	-0.013 (2)	0.025 (2)	0.0163 (19)
O4	0.137 (4)	0.207 (5)	0.104 (3)	-0.053 (4)	-0.027 (3)	0.028 (3)
C1	0.057 (2)	0.062 (2)	0.0480 (18)	-0.0090 (18)	0.0027 (17)	-0.0091 (15)
C2	0.063 (3)	0.085 (3)	0.048 (2)	0.002 (2)	-0.0115 (18)	-0.0183 (19)
C3	0.070 (3)	0.090 (3)	0.0414 (18)	0.009 (2)	-0.0019 (19)	-0.0028 (17)

C4	0.069 (3)	0.065 (2)	0.0419 (18)	0.008 (2)	0.0038 (18)	0.0098 (17)
C5	0.0456 (19)	0.0481 (17)	0.0401 (16)	0.0076 (15)	0.0025 (14)	0.0040 (14)
C6	0.051 (2)	0.0390 (16)	0.0432 (16)	0.0056 (15)	0.0066 (15)	0.0031 (13)
C7	0.067 (3)	0.0458 (19)	0.0512 (18)	-0.0012 (17)	0.0086 (18)	0.0064 (16)
C8	0.071 (3)	0.049 (2)	0.079 (3)	-0.0105 (19)	0.011 (2)	0.0006 (19)
C9	0.069 (3)	0.058 (2)	0.063 (2)	-0.009 (2)	0.0035 (19)	-0.0161 (19)
C10	0.060 (2)	0.056 (2)	0.0453 (18)	-0.0061 (18)	0.0009 (17)	-0.0073 (16)
C11	0.071 (3)	0.069 (2)	0.0481 (19)	0.004 (2)	-0.0041 (18)	0.0063 (17)
C12	0.090 (3)	0.079 (3)	0.046 (2)	0.004 (2)	-0.0136 (19)	0.007 (2)
C13	0.108 (4)	0.070 (2)	0.0402 (19)	-0.006 (2)	0.002 (2)	-0.0029 (17)
C14	0.081 (3)	0.062 (2)	0.047 (2)	-0.004 (2)	0.011 (2)	-0.0068 (17)
C15	0.056 (2)	0.0385 (16)	0.0441 (17)	-0.0077 (15)	0.0018 (16)	-0.0020 (13)
C16	0.054 (2)	0.0384 (16)	0.0467 (18)	-0.0089 (15)	0.0023 (15)	-0.0056 (13)
C17	0.060 (2)	0.055 (2)	0.063 (2)	-0.0002 (19)	0.0051 (19)	-0.0151 (18)
C18	0.057 (3)	0.054 (2)	0.098 (3)	0.0120 (19)	-0.003 (2)	-0.013 (2)
C19	0.061 (3)	0.061 (2)	0.084 (3)	0.012 (2)	-0.014 (2)	0.011 (2)
C20	0.060 (2)	0.059 (2)	0.050 (2)	0.0023 (18)	-0.0066 (18)	0.0101 (17)

*Geometric parameters (Å, °)*

Rh1—N2	2.019 (2)	C4—H4	0.93
Rh1—N1	2.023 (2)	C5—C6	1.470 (4)
Rh1—N3	2.037 (2)	C6—C7	1.377 (5)
Rh1—N4	2.038 (3)	C7—C8	1.369 (5)
Rh1—C13	2.3291 (9)	C7—H7	0.93
Rh1—C13	2.3291 (9)	C8—C9	1.366 (5)
Rh1—C12	2.3344 (9)	C8—H8	0.93
Rh1—C12	2.3344 (9)	C9—C10	1.373 (5)
Cl1—O2B	1.31 (3)	C9—H9	0.93
Cl1—O1	1.401 (3)	C10—H10	0.93
Cl1—O3	1.408 (3)	C11—C12	1.375 (5)
Cl1—O4	1.418 (4)	C11—H11	0.93
Cl1—O2A	1.434 (9)	C12—C13	1.354 (6)
N1—C1	1.338 (4)	C12—H12	0.93
N1—C5	1.353 (4)	C13—C14	1.392 (6)
N2—C10	1.344 (4)	C13—H13	0.93
N2—C6	1.349 (4)	C14—C15	1.379 (5)
N3—C11	1.335 (4)	C14—H14	0.93
N3—C15	1.343 (4)	C15—C16	1.469 (5)
N4—C20	1.332 (4)	C16—C17	1.380 (5)
N4—C16	1.359 (4)	C17—C18	1.365 (5)
C1—C2	1.369 (5)	C17—H17	0.93
C1—H1	0.93	C18—C19	1.362 (6)
C2—C3	1.367 (6)	C18—H18	0.93
C2—H2	0.93	C19—C20	1.356 (5)
C3—C4	1.387 (5)	C19—H19	0.93
C3—H3	0.93	C20—H20	0.93
C4—C5	1.377 (4)		

N2—Rh1—N1	80.50 (10)	C2—C3—C4	118.9 (3)
N2—Rh1—N3	96.45 (10)	C2—C3—H3	120.5
N1—Rh1—N3	174.22 (10)	C4—C3—H3	120.5
N2—Rh1—N4	90.41 (11)	C5—C4—C3	119.5 (4)
N1—Rh1—N4	94.74 (10)	C5—C4—H4	120.3
N3—Rh1—N4	80.32 (11)	C3—C4—H4	120.3
N2—Rh1—Cl3	88.36 (8)	N1—C5—C4	120.6 (3)
N1—Rh1—Cl3	87.08 (7)	N1—C5—C6	114.9 (3)
N3—Rh1—Cl3	97.78 (8)	C4—C5—C6	124.4 (3)
N4—Rh1—Cl3	177.61 (8)	N2—C6—C7	121.0 (3)
N2—Rh1—Cl3	88.36 (8)	N2—C6—C5	115.1 (3)
N1—Rh1—Cl3	87.08 (7)	C7—C6—C5	123.9 (3)
N3—Rh1—Cl3	97.78 (8)	C8—C7—C6	119.6 (3)
N4—Rh1—Cl3	177.61 (8)	C8—C7—H7	120.2
Cl3—Rh1—Cl3	0.00 (5)	C6—C7—H7	120.2
N2—Rh1—Cl2	176.85 (7)	C9—C8—C7	119.3 (4)
N1—Rh1—Cl2	96.36 (8)	C9—C8—H8	120.3
N3—Rh1—Cl2	86.69 (7)	C7—C8—H8	120.3
N4—Rh1—Cl2	90.16 (8)	C8—C9—C10	119.4 (4)
Cl3—Rh1—Cl2	91.18 (4)	C8—C9—H9	120.3
Cl3—Rh1—Cl2	91.18 (4)	C10—C9—H9	120.3
N2—Rh1—Cl2	176.85 (7)	N2—C10—C9	121.5 (3)
N1—Rh1—Cl2	96.36 (8)	N2—C10—H10	119.2
N3—Rh1—Cl2	86.69 (7)	C9—C10—H10	119.2
N4—Rh1—Cl2	90.16 (8)	N3—C11—C12	121.5 (4)
Cl3—Rh1—Cl2	91.18 (4)	N3—C11—H11	119.2
Cl3—Rh1—Cl2	91.18 (4)	C12—C11—H11	119.2
Cl2—Rh1—Cl2	0.00 (5)	C13—C12—C11	119.2 (4)
O2B—C11—O1	122.6 (14)	C13—C12—H12	120.4
O2B—C11—O3	117.0 (14)	C11—C12—H12	120.4
O1—C11—O3	111.1 (2)	C12—C13—C14	120.1 (3)
O2B—C11—O4	86 (2)	C12—C13—H13	119.9
O1—C11—O4	107.4 (3)	C14—C13—H13	119.9
O3—C11—O4	107.5 (2)	C15—C14—C13	118.1 (4)
O1—C11—O2A	106.2 (5)	C15—C14—H14	121
O3—C11—O2A	109.7 (4)	C13—C14—H14	121
O4—C11—O2A	115.0 (7)	N3—C15—C14	121.3 (3)
C1—N1—C5	119.7 (3)	N3—C15—C16	115.6 (3)
C1—N1—Rh1	125.7 (2)	C14—C15—C16	123.1 (3)
C5—N1—Rh1	114.7 (2)	N4—C16—C17	119.7 (3)
C10—N2—C6	119.1 (3)	N4—C16—C15	115.2 (3)
C10—N2—Rh1	126.0 (2)	C17—C16—C15	125.1 (3)
C6—N2—Rh1	114.8 (2)	C18—C17—C16	119.9 (4)
C11—N3—C15	119.8 (3)	C18—C17—H17	120.1
C11—N3—Rh1	125.6 (2)	C16—C17—H17	120.1
C15—N3—Rh1	114.6 (2)	C19—C18—C17	119.3 (4)
C20—N4—C16	119.6 (3)	C19—C18—H18	120.4



C20—N4—Rh1	126.2 (2)	C17—C18—H18	120.4
C16—N4—Rh1	114.2 (2)	C20—C19—C18	119.7 (4)
N1—C1—C2	121.6 (3)	C20—C19—H19	120.1
N1—C1—H1	119.2	C18—C19—H19	120.1
C2—C1—H1	119.2	N4—C20—C19	121.8 (4)
C3—C2—C1	119.8 (4)	N4—C20—H20	119.1
C3—C2—H2	120.1	C19—C20—H20	119.1
C1—C2—H2	120.1		
N1—Rh1—C12—C12	0E1 (8)	C12—Rh1—N4—C16	-84.4 (2)
N3—Rh1—C12—C12	0.00 (2)	C5—N1—C1—C2	1.0 (5)
N4—Rh1—C12—C12	0.00 (2)	Rh1—N1—C1—C2	-176.9 (3)
C13—Rh1—C12—C12	0.00 (2)	N1—C1—C2—C3	-0.4 (6)
C13—Rh1—C12—C12	0.00 (2)	C1—C2—C3—C4	-0.1 (6)
N2—Rh1—C13—C13	0.00 (3)	C2—C3—C4—C5	0.2 (6)
N1—Rh1—C13—C13	0.00 (3)	C1—N1—C5—C4	-0.9 (5)
N3—Rh1—C13—C13	0.00 (3)	Rh1—N1—C5—C4	177.2 (3)
C12—Rh1—C13—C13	0.00 (3)	C1—N1—C5—C6	-179.4 (3)
C12—Rh1—C13—C13	0.00 (3)	Rh1—N1—C5—C6	-1.3 (3)
N2—Rh1—N1—C1	178.2 (3)	C3—C4—C5—N1	0.3 (5)
N4—Rh1—N1—C1	88.5 (3)	C3—C4—C5—C6	178.7 (3)
C13—Rh1—N1—C1	-93.0 (3)	C10—N2—C6—C7	1.5 (5)
C13—Rh1—N1—C1	-93.0 (3)	Rh1—N2—C6—C7	178.3 (3)
C12—Rh1—N1—C1	-2.2 (3)	C10—N2—C6—C5	-178.8 (3)
C12—Rh1—N1—C1	-2.2 (3)	Rh1—N2—C6—C5	-2.0 (4)
N2—Rh1—N1—C5	0.2 (2)	N1—C5—C6—N2	2.2 (4)
N4—Rh1—N1—C5	-89.4 (2)	C4—C5—C6—N2	-176.2 (3)
C13—Rh1—N1—C5	89.0 (2)	N1—C5—C6—C7	-178.2 (3)
C13—Rh1—N1—C5	89.0 (2)	C4—C5—C6—C7	3.4 (5)
C12—Rh1—N1—C5	179.9 (2)	N2—C6—C7—C8	-0.7 (6)
C12—Rh1—N1—C5	179.9 (2)	C5—C6—C7—C8	179.7 (3)
N1—Rh1—N2—C10	177.6 (3)	C6—C7—C8—C9	-0.6 (6)
N3—Rh1—N2—C10	-7.4 (3)	C7—C8—C9—C10	1.0 (6)
N4—Rh1—N2—C10	-87.7 (3)	C6—N2—C10—C9	-1.1 (5)
C13—Rh1—N2—C10	90.3 (3)	Rh1—N2—C10—C9	-177.5 (3)
C13—Rh1—N2—C10	90.3 (3)	C8—C9—C10—N2	-0.1 (6)
N1—Rh1—N2—C6	1.1 (2)	C15—N3—C11—C12	-0.5 (5)
N3—Rh1—N2—C6	176.1 (2)	Rh1—N3—C11—C12	177.1 (3)
N4—Rh1—N2—C6	95.8 (2)	N3—C11—C12—C13	0.0 (6)
C13—Rh1—N2—C6	-86.3 (2)	C11—C12—C13—C14	0.0 (6)
C13—Rh1—N2—C6	-86.3 (2)	C12—C13—C14—C15	0.5 (6)
N2—Rh1—N3—C11	89.5 (3)	C11—N3—C15—C14	1.0 (5)
N4—Rh1—N3—C11	178.8 (3)	Rh1—N3—C15—C14	-176.9 (3)
C13—Rh1—N3—C11	0.3 (3)	C11—N3—C15—C16	-178.1 (3)
C13—Rh1—N3—C11	0.3 (3)	Rh1—N3—C15—C16	4.0 (3)
C12—Rh1—N3—C11	-90.4 (3)	C13—C14—C15—N3	-1.0 (5)
C12—Rh1—N3—C11	-90.4 (3)	C13—C14—C15—C16	178.1 (3)
N2—Rh1—N3—C15	-92.7 (2)	C20—N4—C16—C17	-1.7 (5)

N4—Rh1—N3—C15	-3.4 (2)	Rh1—N4—C16—C17	-179.9 (2)
Cl3—Rh1—N3—C15	178.0 (2)	C20—N4—C16—C15	177.4 (3)
Cl3—Rh1—N3—C15	178.0 (2)	Rh1—N4—C16—C15	-0.8 (3)
Cl2—Rh1—N3—C15	87.3 (2)	N3—C15—C16—N4	-2.1 (4)
Cl2—Rh1—N3—C15	87.3 (2)	C14—C15—C16—N4	178.8 (3)
N2—Rh1—N4—C20	-79.4 (3)	N3—C15—C16—C17	177.0 (3)
N1—Rh1—N4—C20	1.1 (3)	C14—C15—C16—C17	-2.2 (5)
N3—Rh1—N4—C20	-175.9 (3)	N4—C16—C17—C18	2.8 (5)
Cl2—Rh1—N4—C20	97.5 (3)	C15—C16—C17—C18	-176.2 (3)
Cl2—Rh1—N4—C20	97.5 (3)	C16—C17—C18—C19	-1.8 (6)
N2—Rh1—N4—C16	98.7 (2)	C17—C18—C19—C20	-0.2 (7)
N1—Rh1—N4—C16	179.2 (2)	C16—N4—C20—C19	-0.4 (5)
N3—Rh1—N4—C16	2.3 (2)	Rh1—N4—C20—C19	177.6 (3)
Cl2—Rh1—N4—C16	-84.4 (2)	C18—C19—C20—N4	1.4 (6)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1...Cl2	0.93	2.7	3.301 (4)	123
C11—H11...Cl3	0.93	2.76	3.358 (4)	123
C3—H3...O1 <sup>i</sup>	0.93	2.29	3.192 (5)	164
C8—H8...O1 <sup>ii</sup>	0.93	2.56	3.142 (6)	121
C9—H9...O1 <sup>ii</sup>	0.93	2.58	3.154 (6)	120
C13—H13...O4 <sup>iii</sup>	0.93	2.56	3.427 (6)	155

Symmetry codes: (i)  $-x+2, y+1/2, -z+3/2$ ; (ii)  $x-1/2, -y-1/2, -z+1$ ; (iii)  $-x+2, y+1/2, -z+1/2$ .