Crystal structure of *trans*-bis{4-bromo-*N*-[(pyridin-2-yl)methylidene]aniline-$\kappa^2 N,N'$}dichlorido-ruthenium(II)

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In the title complex, [RuCl$_2$(C$_{12}$H$_9$BrN$_2$)$_2$] or [RuCl$_2$(PM-BrA)$_2$] (PM-BrA = 4-bromo-*N*-(2′-pyridylmethylene)aniline), the Ru^{II} cation is located on a centre of inversion and is surrounded by four N atoms of two PM-BrA ligands in the equatorial plane and by two Cl atoms in a *trans* axial arrangement, displaying a distorted octahedral coordination environment. Two C atoms in the benzene ring of the PM-BrA ligand are equally disordered over two sets of sites. The benzene and pyridine rings of the PM-BrA ligand are oriented at dihedral angles of 62.1 (10) and 73.7 (11)° under consideration of the two orientations of the disordered benzene ring. In the crystal, the complex molecules are connected *via* C—H⋯Cl hydrogen-bonding interactions into a layered arrangement parallel (100). C—H⋯Br hydrogen bonding and weak aromatic π–π stacking interactions complete a three-dimensional supramolecular network.

1. Chemical context

Bidentate Schiff bases are one of the most widely used ligands in coordination chemistry. Their complexes have found utility in a wide range of applications (Rezaeivala & Keypour, 2014; Gupta & Sutar, 2008). In particular, ruthenium(II) complexes of Schiff bases have been shown to display a variety of structural features and exhibit interesting biological and catalytic reactivities (Li *et al.*, 2015; Wang *et al.*, 2015; Drozdzak *et al.*, 2005). Herein, we report the synthesis and crystal structure of a ruthenium(II) complex with the bidentate Schiff base ligand of 4-bromo-*N*-(2′-pyridylmethylene)aniline (PM-BrA), [RuCl$_2$(C$_{12}$H$_9$BrN$_2$)$_2$], (I).
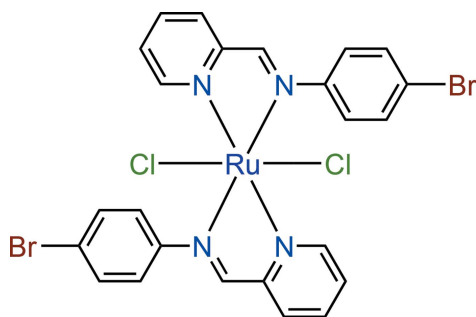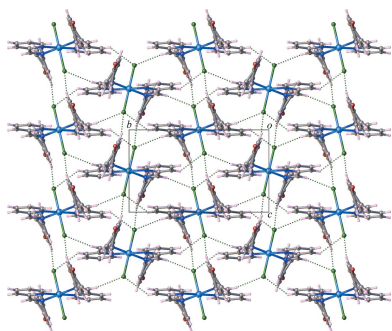
2. Structural commentary

The asymmetric unit of compound (I) contains one half of the complex molecule with the Ru^{II} cation lying on an inversion centre (Fig. 1). The coordination environment around Ru^{II} is a distorted [Cl$_2$N$_4$] octahedron, whereby the metal is chelated by two PM-BrA ligands in the equatorial plane and by two Cl
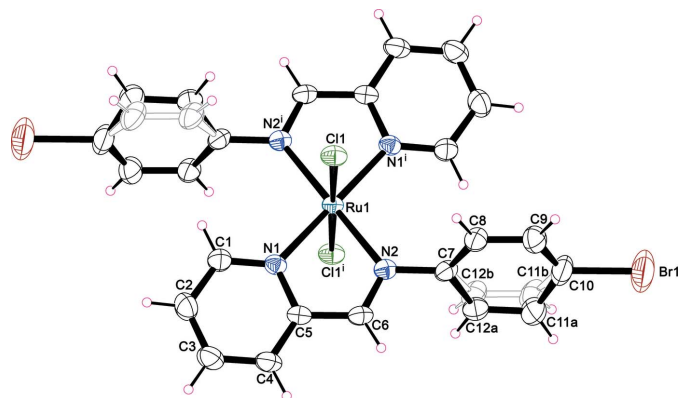
Figure 1
The molecular structure of complex (I), showing displacement ellipsoids at the 50% probability level. Disorder is displayed for the C11 and C12 atoms of the benzene ring. [Symmetry operator: (i) $-x + 1, -y + 1, -z$.]

atoms in a *trans* axial arrangement. The ligand exhibits an N1$\cdots$N2 bite distance of 2.585 (7) Å with an N1—Ru1—N2 bite angle of 76.9 (1)°. The reduced bite angle of the chelating ligand is one of the main factors accounting for the distortion from the ideal octahedral geometry of the coordination polyhedron, with the the largest *cis* angle being 103.1 (2)°. The Ru—N bond lengths are 2.073 (5) and 2.084 (5) Å, and the Ru—Cl bond length is 2.3908 (14) Å, in agreement with those observed in the structures of similar compounds (Roy *et al.*, 2012). Two C atoms in the benzene ring of the PM-BrA ligand are equally disordered over two sets of sites. The dihedral angle between the least-square planes of the benzene and pyridine rings in the PM-BrA ligand are 62.1 (10) and 73.7 (11)° under consideration of the two orientations of the disordered benzene ring.
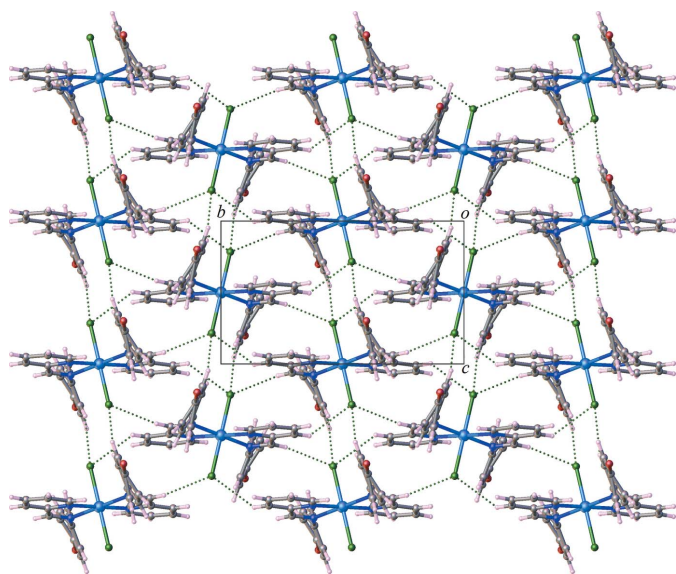
Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$\cdots$Cl1^{i}	0.93	2.79	3.472 (7)	132
C6—H6$\cdots$Cl1^{ii}	0.93	2.83	3.673 (7)	151
C3—H3$\cdots$Br1^{iii}	0.93	3.13	3.797 (8)	131
C4—H4$\cdots$Cl1^{iv}	0.93	2.94	3.529 (7)	122

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

3. Supramolecular features

In the crystal, weak intermolecular C—H$\cdots$Cl hydrogen-bonding interactions between the C atoms of the benzene ring and the Cl atoms connect the complex molecules into a supramolecular layered arrangement parallel to (100) (Fig. 2). As shown in Fig. 3, a C—H$\cdots$Br hydrogen bond between the phenyl C atoms and the Br atoms, along with weak aromatic π–π stacking interactions [centroid-to-centroid distance = 4.107 (4) Å, dihedral angle = 0.7 (3)°] complete a three-dimensional supramolecular network. Numerical values of C—H$\cdots X$ (X = Cl, Br) interactions are compiled in Table 1.

4. Database survey

The structure of *trans*-[RuCl2(Hpyrimol)2] (Hpyrimol = 4-methyl-2-*N*-(2-pyridylmethylene)aminophenol) with a closely related Schiff base N2 donor set for each ligand has been reported (Roy *et al.*, 2012). The bond lengths and bond angles in this complex are in agreement with those in the structure of

Figure 2
Crystal packing of complex (I) in a view along [100]. C—H$\cdots$Cl hydrogen-bonding interactions are shown as dashed lines.
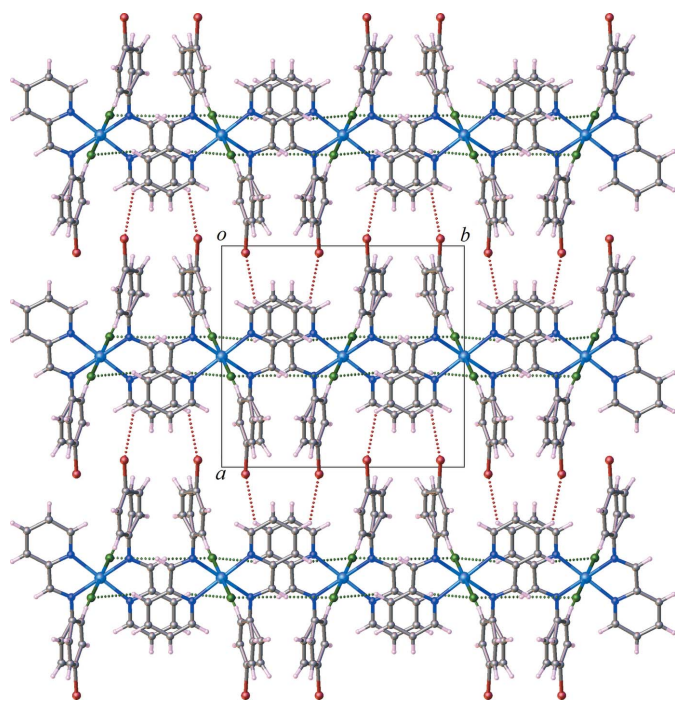

Figure 3
Crystal packing and C—H$\cdots$Br and C—H$\cdots$Cl hydrogen-bonding interactions (dashed lines) in complex (I), viewed along [001].

(I). A search of the Cambridge Structural Database (Version 5.36, last update February 2015; Groom & Allen, 2014) gave 12 hits for complexes involving transition metals and the ligand PM-BrA (KISZIX, KISZOD, KISZUJ, Davies *et al.*, 2014; XEDCUG, Khalaji *et al.*, 2012; UNIZOH, Harding *et al.*, 2011; SUYDAS, Harding *et al.*, 2010; FOWBOJ, Khalaj *et al.*, 2009; FOWBID, Mahmoudi *et al.*, 2009; MOYDUA, Dehghanpour *et al.*, 2009; TULKIV, Gao *et al.*, 2009; YOCZAS, Khalaj *et al.*, 2008; YOCZEW, Mahmoudi *et al.*, 2008).

5. Synthesis and crystallization

A solution of the ligand 4-bromo-N-(2′-pyridylmethylene)-aniline (104.4 mg, 0.4 mmol) in dry methanol (5 ml) was placed in a test tube. A solution of $RuCl_3$ (41.5 mg, 0.2 mmol) in dry methanol (5 ml) was then carefully layered on the top of a methanolic solution. After slow diffusion at room temperature for three days, pale-green plate- or block-like crystals of complex (I) were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were positioned with idealized geometry and refined with $U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model with C—H = 0.95 Å. C atoms C11 and C12 and attached H atoms in the benzene ring are disordered over two set of sites and were refined using a split model with equal occupancy.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$[RuCl_2(C_{12}H_9BrN_2)]$
M_r	694.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	12.3270 (7), 13.3114 (7), 7.9673 (4)
β (°)	100.091 (2)
V (Å^3)	1287.13 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.94
Crystal size (mm)	$0.26 \times 0.20 \times 0.18$
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.549, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15951, 2391, 1844
R_{int}	0.054
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.607
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.054, 0.152, 1.04
No. of reflections	2391
No. of parameters	170
No. of restraints	73
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.96, −1.29

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2007* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

supporting information

Crystal structure of *trans*-bis{4-bromo-*N*-[(pyridin-2-yl)methylidene]aniline-*κ²N,N′*}dichloridoruthenium(II)

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2007* (Sheldrick, 2015*b*); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

***trans*-Bis{4-bromo-*N*-[(pyridin-2-yl)methylidene]aniline-\ *κ²N,N′*}dichloridoruthenium(II)**

Crystal data

$[RuCl_2(C_{12}H_9BrN_2)]$	$F(000) = 676$
$M_r = 694.21$	$D_x = 1.791$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 12.3270$ (7) Å	Cell parameters from 4475 reflections
$b = 13.3114$ (7) Å	$\theta = 3.0$–25.4°
$c = 7.9673$ (4) Å	$\mu = 3.94$ mm^{-1}
$\beta = 100.091$ (2)°	$T = 296$ K
$V = 1287.13$ (12) Å^3	Block, green
$Z = 2$	$0.26 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII CCD diffractometer	2391 independent reflections
φ and ω scans	1844 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)	$R_{int} = 0.054$
	$\theta_{max} = 25.6°, \theta_{min} = 3.0°$
$T_{min} = 0.549, T_{max} = 0.745$	$h = -14 \rightarrow 14$
15951 measured reflections	$k = -16 \rightarrow 16$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H-atom parameters constrained
$wR(F^2) = 0.152$	$w = 1/[\sigma^2(F_o^2) + (0.0854P)^2 + 2.577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	
2391 reflections	$(\Delta/\sigma)_{max} < 0.001$
170 parameters	$\Delta\rho_{max} = 0.96$ e Å^{-3}
73 restraints	$\Delta\rho_{min} = -1.29$ e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ru1	0.5000	0.5000	0.0000	0.0438 (2)	
Cl1	0.58971 (14)	0.46179 (12)	0.28373 (18)	0.0584 (4)	
Br1	−0.03134 (9)	0.59834 (15)	0.3013 (2)	0.1661 (8)	
N1	0.6046 (4)	0.6219 (4)	−0.0113 (6)	0.0497 (11)	
N2	0.4128 (4)	0.6192 (4)	0.0781 (6)	0.0503 (11)	
C7	0.3075 (5)	0.6159 (5)	0.1302 (8)	0.0563 (14)	
C8	0.3001 (6)	0.5804 (5)	0.2904 (8)	0.0631 (16)	
H8	0.3635	0.5596	0.3632	0.076*	
C6	0.4524 (6)	0.7073 (5)	0.0614 (8)	0.0600 (16)	
H6	0.4143	0.7650	0.0817	0.072*	
C5	0.5584 (5)	0.7125 (4)	0.0100 (8)	0.0555 (14)	
C9	0.2001 (7)	0.5755 (6)	0.3439 (10)	0.079 (2)	
H9	0.1950	0.5530	0.4528	0.094*	
C3	0.7127 (7)	0.8027 (6)	−0.0497 (11)	0.080 (2)	
H3	0.7478	0.8626	−0.0679	0.096*	
C4	0.6092 (7)	0.8027 (5)	−0.0099 (10)	0.075 (2)	
H4	0.5736	0.8630	0.0035	0.090*	
C1	0.7073 (6)	0.6224 (5)	−0.0410 (10)	0.0713 (19)	
H1	0.7433	0.5616	−0.0482	0.086*	
C10	0.1078 (7)	0.6047 (8)	0.2316 (13)	0.095 (3)	
C11B	0.113 (3)	0.624 (4)	0.061 (3)	0.087 (7)	0.50 (9)
H11B	0.0484	0.6332	−0.0171	0.105*	0.50 (9)
C2	0.7629 (7)	0.7123 (6)	−0.0620 (11)	0.082 (2)	
H2	0.8344	0.7102	−0.0844	0.099*	
C12B	0.2127 (19)	0.630 (4)	0.010 (4)	0.078 (7)	0.50 (9)
H12B	0.2170	0.6422	−0.1035	0.093*	0.50 (9)
C12A	0.2157 (19)	0.661 (3)	0.035 (6)	0.077 (7)	0.50 (9)
H12A	0.2222	0.6925	−0.0674	0.092*	0.50 (9)
C11A	0.116 (3)	0.660 (4)	0.087 (5)	0.095 (8)	0.50 (9)
H11A	0.0561	0.6956	0.0276	0.114*	0.50 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.0540 (4)	0.0368 (3)	0.0405 (4)	0.0017 (3)	0.0078 (3)	0.0008 (3)
Cl1	0.0767 (10)	0.0531 (8)	0.0423 (7)	0.0036 (8)	0.0016 (6)	0.0044 (6)
Br1	0.0811 (7)	0.2433 (19)	0.1885 (15)	0.0388 (9)	0.0645 (8)	0.0555 (13)
N1	0.061 (3)	0.043 (3)	0.046 (3)	−0.002 (2)	0.011 (2)	−0.002 (2)
N2	0.061 (3)	0.048 (3)	0.041 (2)	0.004 (2)	0.008 (2)	0.000 (2)

C7	0.063 (3)	0.053 (3)	0.054 (3)	0.015 (3)	0.012 (3)	0.001 (3)
C8	0.063 (4)	0.074 (4)	0.052 (4)	0.011 (3)	0.010 (3)	0.008 (3)
C6	0.073 (4)	0.041 (3)	0.065 (4)	0.007 (3)	0.011 (3)	−0.001 (3)
C5	0.067 (4)	0.041 (3)	0.057 (3)	0.000 (3)	0.006 (3)	0.005 (3)
C9	0.078 (5)	0.090 (5)	0.072 (5)	0.005 (4)	0.026 (4)	0.007 (4)
C3	0.080 (5)	0.057 (4)	0.102 (6)	−0.015 (4)	0.013 (4)	0.004 (4)
C4	0.081 (5)	0.046 (4)	0.097 (6)	−0.003 (4)	0.014 (4)	0.010 (3)
C1	0.069 (4)	0.051 (4)	0.097 (5)	−0.001 (3)	0.024 (4)	−0.006 (3)
C10	0.063 (4)	0.123 (7)	0.106 (6)	0.029 (5)	0.033 (4)	0.023 (5)
C11B	0.064 (7)	0.112 (18)	0.084 (8)	0.034 (12)	0.009 (8)	0.014 (10)
C2	0.067 (4)	0.079 (5)	0.105 (6)	−0.018 (4)	0.026 (4)	−0.006 (4)
C12B	0.069 (8)	0.099 (19)	0.064 (9)	0.028 (10)	0.010 (5)	0.019 (11)
C12A	0.071 (8)	0.077 (16)	0.081 (12)	0.015 (9)	0.010 (7)	0.027 (11)
C11A	0.061 (7)	0.111 (19)	0.111 (12)	0.016 (12)	0.009 (10)	0.037 (12)

Geometric parameters (Å, º)

Ru1—Cl1	2.3908 (14)	C5—C4	1.376 (9)
Ru1—Cl1^i	2.3907 (14)	C9—H9	0.9300
Ru1—N1	2.084 (5)	C9—C10	1.374 (12)
Ru1—N1^i	2.084 (5)	C3—H3	0.9300
Ru1—N2^i	2.073 (5)	C3—C4	1.367 (11)
Ru1—N2	2.073 (5)	C3—C2	1.365 (11)
Br1—C10	1.895 (8)	C4—H4	0.9300
N1—C5	1.356 (8)	C1—H1	0.9300
N1—C1	1.328 (8)	C1—C2	1.403 (10)
N2—C7	1.432 (8)	C10—C11B	1.392 (17)
N2—C6	1.286 (8)	C10—C11A	1.391 (17)
C7—C8	1.379 (9)	C11B—H11B	0.9300
C7—C12B	1.387 (15)	C11B—C12B	1.365 (17)
C7—C12A	1.385 (15)	C2—H2	0.9300
C8—H8	0.9300	C12B—H12B	0.9300
C8—C9	1.374 (10)	C12A—H12A	0.9300
C6—H6	0.9300	C12A—C11A	1.364 (17)
C6—C5	1.438 (9)	C11A—H11A	0.9300
Cl1^i—Ru1—Cl1	180.0	C4—C5—C6	122.0 (6)
N1—Ru1—Cl1	91.11 (14)	C8—C9—H9	121.0
N1^i—Ru1—Cl1	88.89 (14)	C10—C9—C8	118.0 (7)
N1^i—Ru1—Cl1^i	91.11 (14)	C10—C9—H9	121.0
N1—Ru1—Cl1^i	88.89 (14)	C4—C3—H3	121.0
N1—Ru1—N1^i	180.0 (2)	C2—C3—H3	121.0
N2^i—Ru1—Cl1^i	93.28 (13)	C2—C3—C4	118.0 (7)
N2^i—Ru1—Cl1	86.71 (13)	C5—C4—H4	120.4
N2—Ru1—Cl1^i	86.72 (13)	C3—C4—C5	119.3 (7)
N2—Ru1—Cl1	93.29 (13)	C3—C4—H4	120.4
N2^i—Ru1—N1^i	76.9 (2)	N1—C1—H1	119.1
N2—Ru1—N1	76.9 (2)	N1—C1—C2	121.7 (7)

N2^i—Ru1—N1	103.1 (2)	C2—C1—H1	119.1
N2—Ru1—N1^i	103.1 (2)	C9—C10—Br1	119.0 (7)
N2—Ru1—N2^i	180.0	C9—C10—C11B	120.9 (15)
C5—N1—Ru1	114.3 (4)	C9—C10—C11A	121.3 (16)
C1—N1—Ru1	128.9 (4)	C11B—C10—Br1	119.4 (15)
C1—N1—C5	116.9 (5)	C11A—C10—Br1	117.9 (17)
C7—N2—Ru1	127.4 (4)	C10—C11B—H11B	120.0
C6—N2—Ru1	116.1 (4)	C12B—C11B—C10	120 (3)
C6—N2—C7	116.0 (5)	C12B—C11B—H11B	120.0
C8—C7—N2	119.3 (5)	C3—C2—C1	120.4 (7)
C8—C7—C12B	120.0 (17)	C3—C2—H2	119.8
C8—C7—C12A	118.5 (18)	C1—C2—H2	119.8
C12B—C7—N2	119.5 (15)	C7—C12B—H12B	120.7
C12A—C7—N2	121.5 (17)	C11B—C12B—C7	119 (3)
C7—C8—H8	119.6	C11B—C12B—H12B	120.7
C9—C8—C7	120.8 (6)	C7—C12A—H12A	119.3
C9—C8—H8	119.6	C11A—C12A—C7	121 (3)
N2—C6—H6	121.5	C11A—C12A—H12A	119.3
N2—C6—C5	117.0 (6)	C10—C11A—H11A	121.4
C5—C6—H6	121.5	C12A—C11A—C10	117 (3)
N1—C5—C6	114.5 (5)	C12A—C11A—H11A	121.4
N1—C5—C4	123.5 (6)		
Ru1—N1—C5—C6	8.8 (7)	C8—C7—C12B—C11B	11 (4)
Ru1—N1—C5—C4	−173.2 (6)	C8—C7—C12A—C11A	−8 (4)
Ru1—N1—C1—C2	173.2 (6)	C8—C9—C10—Br1	179.8 (7)
Ru1—N2—C7—C8	76.6 (7)	C8—C9—C10—C11B	10 (3)
Ru1—N2—C7—C12B	−91 (3)	C8—C9—C10—C11A	−16 (3)
Ru1—N2—C7—C12A	−113 (3)	C6—N2—C7—C8	−111.0 (7)
Ru1—N2—C6—C5	−7.1 (8)	C6—N2—C7—C12B	81 (3)
Br1—C10—C11B—C12B	179.5 (19)	C6—N2—C7—C12A	59 (3)
Br1—C10—C11A—C12A	−178 (2)	C6—C5—C4—C3	176.3 (7)
N1—C5—C4—C3	−1.4 (11)	C5—N1—C1—C2	−4.5 (11)
N1—C1—C2—C3	1.0 (13)	C9—C10—C11B—C12B	−10 (4)
N2—C7—C8—C9	−179.5 (7)	C9—C10—C11A—C12A	18 (5)
N2—C7—C12B—C11B	178.6 (18)	C4—C3—C2—C1	2.6 (13)
N2—C7—C12A—C11A	−178 (2)	C1—N1—C5—C6	−173.1 (6)
N2—C6—C5—N1	−1.3 (9)	C1—N1—C5—C4	4.8 (10)
N2—C6—C5—C4	−179.3 (6)	C10—C11B—C12B—C7	0 (4)
C7—N2—C6—C5	179.7 (6)	C2—C3—C4—C5	−2.3 (12)
C7—C8—C9—C10	1.5 (12)	C12B—C7—C8—C9	−12 (3)
C7—C12A—C11A—C10	−6 (4)	C12A—C7—C8—C9	10 (3)

Symmetry code: (i) −x+1, −y+1, −z.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···Cl1^{ii}	0.93	2.79	3.472 (7)	132

C6—H6···Cl1[iii]	0.93	2.83	3.673 (7)	151
C3—H3···Br1[iv]	0.93	3.13	3.797 (8)	131
C4—H4···Cl1[v]	0.93	2.94	3.529 (7)	122

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, *y*+1/2, −*z*+1/2; (iv) *x*+1, −*y*+3/2, *z*−1/2; (v) *x*, −*y*+3/2, *z*−1/2.