A chloro(2-pyridinecarboxaldehyde)(2,2':6',2''-terpyridine)-ruthenium(II) complex

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The aldehyde moiety in the title complex, chloro(2-pyridinecarboxaldehyde-*N,O*)(2,2':6',2''-terpyridine- κ^3N)ruthenium(II)–chloro(2-pyridinecarboxylic acid-*N,O*)(2,2':6',2''-terpyridine- κ^3N)ruthenium(II)–perchlorate–chloroform–water (1.8/0.2/2/1/1), $[\text{RuCl}(\text{C}_6\text{H}_5\text{NO})(\text{C}_{15}\text{H}_{11}\text{N}_3)]_{1.8}[\text{RuCl}(\text{C}_6\text{H}_5\text{NO}_2)(\text{C}_{15}\text{H}_{11}\text{N}_3)]_{0.2}(\text{ClO}_4)_2\cdot\text{CHCl}_3\cdot\text{H}_2\text{O}$, is a structural model of substrate coordination to a transfer hydrogenation catalyst. The title complex features two independent Ru^{II} complex cations that display very similar distorted octahedral coordination provided by the three N atoms of the 2,2':6',2''-terpyridine ligand, the N and O atoms of the 2-pyridinecarboxaldehyde (pyCHO) ligand and a chloride ligand. One of the cation sites is disordered such that the aldehyde group is replaced by a 20 (1)% contribution from a carboxylic acid group (aldehyde H replaced by carboxyl O–H). Notable dimensions in the non-disordered complex cation are Ru–N 2.034 (2) Å and Ru–O 2.079 (2) Å to the pyCHO ligand and O–C 1.239 (4) Å for the pyCHO carbonyl group.

Comment

Metal complexes of aldehydes and ketones have attracted recent attention as possible models for catalytic intermediates in the transfer hydrogenation of ketones by alcohols. Though many ruthenium(II)-based transfer hydrogenation catalysts are known, the specifics of the catalytic process remain a matter of debate. Several mechanistic proposals suggest that the key step involves activation of the coordinated substrate, making ruthenium(II) ketone and aldehyde complexes of structural interest (Yamakawa *et al.*, 2000). Though a few ruthenium(II) ketone and aldehyde complexes have been reported, the coordination environments do not resemble those of known transfer hydrogenation catalysts. As a continuation of our research into the activity of a new class of Ru^{II} (terpy)-based (terpy is 2,2':6',2''-terpyridine) transfer hydrogenation catalysts, the present work is concerned with

the crystal structure of the newly synthesized complex obtained from the reaction of $\text{Ru}^{\text{III}}(\text{terpy})\text{Cl}_3$ with 2-pyridinecarboxaldehyde (pyCHO). This compound, (I), is the first crystallographically characterized ruthenium(II) complex with pyCHO and features a coordinated aldehyde within the ligand set of a transfer hydrogenation catalyst.

Our analysis shows that complex (I) consists of two crystallographically independent $[\text{Ru}^{\text{II}}\text{Cl}(\text{pyCHO})(\text{terpy})]^+$ cations and two ClO_4^- anions, together with lattice molecules of chloroform and water. During the refinement process, it became apparent that one of the cation sites (cation A) was disordered, with the aldehyde group being replaced by a 20 (1)% contribution from a carboxylic acid group (aldehyde H replaced by carboxyl O–H); details are provided in the *Experimental* section. The two cations are nonetheless very similar and exhibit the same structural features. In each cation, the Ru atom is surrounded by a distorted octahedral arrangement of one terpy molecule coordinated in a tridentate manner, one pyCHO ligand coordinated through the N and O atoms, and a Cl^- ion placed *trans* with respect to the pyCHO N atom (Fig. 1). Replacement of 20 (1)% of the aldehyde function in cation A with a carboxylic acid function appears to cause little difference to the principal geometry features. The mean Ru–N distances to the terpy ligands are typical of Ru^{II} (terpy) complexes, with values of 2.064 (6) Å to the outer pyridine rings and 1.952 (4) Å to the inner pyridine rings. The mean $\text{Ru}^{\text{II}}-\text{Cl}$ distance of 2.391 (5) Å is also as expected. The Ru–N distances to the pyCHO ligand, 2.042 (2) and 2.034 (2) Å for cations A and B, respectively, are short of the typical range from 2.114 to 2.100 Å for monopyridyl ligands in Ru^{II} (terpy) complexes (Hecker *et al.*, 1991; Rasmussen *et al.*, 1995). Similarly, the Ru–O distances to the pyCHO ligand, 2.071 (2) and 2.079 (2) Å for cations A and B, respectively, are somewhat shorter than the value of 2.11 Å reported for monodentate Ru^{II} –aldehyde complexes (Carmona *et al.*, 1997).

The mean bite angle for the terpy ligands is 79.5 (3)° which is expected for Ru^{II} (terpy) complexes. The mean bite angle for the pyCHO ligands is comparable [78.4 (1)°]. The equatorial planes formed by the Ru atoms, the coordinating terpy N atoms, and the pyCHO O atoms are slightly distorted from planarity, with maximum deviations of 0.011 (1) Å for Ru1A and 0.013 (1) Å for Ru1B toward the apical Cl ligands. The terpy molecules themselves are planar, with maximum deviations of 0.081 (2) Å for N3A and 0.075 (2) Å for N4B, and the dihedral angles between the terpy molecules and the equatorial planes are 2.6 (2) and 3.9 (1)° for cations A and B, respectively, with both terpy planes tipped slightly toward the

Cl ligands likely in response to the steric influence of the axial pyridine ring of pyCHO. The pyCHO ligand is also planar, with a maximum deviation of 0.028 (2) Å for O1A and 0.027 (3) Å for C6B. The dihedral angles between the mean planes of the terpy and pyCHO ligands are 87.39 (4) and 82.16 (6)° for cations A and B, respectively, suggesting modest flexibility in the tipping angle of the pyCHO ligand.

Bond distances and angles within the terpy molecule have expected values. The pyCHO carbonyl C6B—O1B distance of 1.239 (4) Å in the non-disordered cation B is significantly longer than 1.22 Å of monodentate Ru^{II}–aldehyde complexes (Carmona *et al.*, 1997). Further, the corresponding pyCHO

Figure 1
Perspective view of cation B of compound (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

ring N1B—C5B distance of 1.370 (4) Å is longer than the N1B—C1B distance of 1.345 (4) Å (a typical value for pyridine; Hecker *et al.*, 1991; Rasmussen *et al.*, 1995). The other bond lengths and angles within the pyCHO ligand have expected values. The relatively long carbonyl C6B—O1B and N1B—C5B distances taken with the shorter than expected Ru1—N1 and Ru1—O1 distances suggest a modest shift of bond order around the π -system of the Ru–pyCHO metallocycle that weakens the carbonyl bond. An analogous bond order shift is reported for the metallocycle of [(C₆Me₄H₂)Ru{CH=C(C₆H₄-*p*-NMe₂)CHO}(PPh₃)]PF₆, which results in an unusually long carbonyl C—O distance [1.260 (5) Å; Pilette *et al.*, 1994]. Compound (I) provides an example of this effect within a hard-donor coordination environment. This mechanism for weakening coordinated carbonyls may have relevance to substrate activation by Ru^{II}–ligand moieties in transfer hydrogenation catalysis.

Hydrogen bonds (see Table 1) from the lattice water molecule to one perchlorate O atom [O6···O5B 3.034 (5) Å] and the Cl atom of cation B [O6···Cl1B 3.300 (3) Å] are evident. There is also a hydrogen bond from the chloroform C—H group to another perchlorate O atom [C22···O3A 3.174 (6) Å]. These interactions do not appear to exert a dominant influence on the structure.

Experimental

The title compound was prepared by refluxing Ru^{III}(terpy)Cl₃ (0.12 g) with 2-pyridinecarbinol (0.5 ml) in ethanol (7.5 ml) under an argon atmosphere followed by crystallization with HClO₄ (70% w/w) (0.5 ml). Elemental analysis: C 43.53, H 2.97, N 9.58%; expected for [Ru^{II}Cl(pyCHO)(terpy)](ClO₄): C 43.76, H 2.80, N 9.72%. Vapor diffusion of chloroform into an acetone solution of the product at room temperature over 3 d afforded dark-red prisms of the title compound.

Crystal data

[RuCl(C₆H₅NO)(C₁₅H₁₁N₃)]_{1.8}
[RuCl(C₆H₅NO₂)(C₁₅H₁₁N₃)]_{0.2}
(ClO₄)₂·CHCl₃·H₂O
M_r = 1293.11
Triclinic, *P*1
a = 10.8871 (2) Å
b = 13.4187 (3) Å
c = 17.9641 (4) Å
 α = 68.7915 (12)°
 β = 83.8241 (10)°
 γ = 87.0651 (11)°

V = 2432.23 (9) Å³
Z = 2
D_x = 1.766 Mg m^{−3}
Mo *K*α radiation
Cell parameters from 21 341 reflections
 θ = 2.14–32.60°
 μ = 1.074 mm^{−1}
T = 200 (2) K
Prism, dark red
0.28 × 0.20 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
Absorption correction: multi-scan (DENZO-SMN; Otwinowski & Minor, 1997)
*T*_{min} = 0.753, *T*_{max} = 0.919
21 341 measured reflections
16 021 independent reflections

11 021 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.030
 θ _{max} = 32.60°
h = −16 → 16
k = −19 → 18
l = −19 → 27
Intensity decay: <2.0%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.049
wR (*F*²) = 0.116
S = 1.017
16 021 reflections
652 parameters
H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0395*P*)² + 2.2430*P*]
where *P* = (*F_o*² + 2*F_c*²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.89 e Å^{−3}
Δρ_{min} = −0.89 e Å^{−3}
Extinction correction: SHELXL97 (Sheldrick, 1997)
Extinction coefficient: 0.0019 (3)

Table 1
Hydrogen-bonding 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>
O6—H61···O5B	0.93 (3)	2.13 (4)	3.034 (5)	161 (5)
O6—H62···Cl1B	0.93 (3)	2.37 (4)	3.300 (3)	175 (5)
C22—H22···O3A	1.00	2.25	3.174 (6)	153

The H atoms bound to carbon were positioned with idealized geometry (*Csp*²—H = 0.95 Å and chloroform *Csp*³—H = 1.00 Å), assigned isotropic displacement parameters equal to 1.2*U*_{eq} of the parent atom and refined with a riding model. The H atoms of the lattice water molecule were found in a difference Fourier synthesis and were refined with a restrained O—H distance. During the refinement calculations, a large electron-density peak (approximately 2 e Å^{−3}) appeared about 1.32 Å from the aldehyde carbon of cation A adjacent to the aldehyde H-atom site. In the final refinement, the additional site was allowed for as a partially occupied O atom (O1A2)

with an isotropic displacement parameter; the occupancy parameter refined to 0.201 (10) [aldehyde C—H = 0.799 (10)] and physically corresponds with the aldehyde H atom being replaced in 20% of the sites by a carboxylic acid O—H group. The additional partial O atom so introduced is approximately 2.88 (2) Å from an adjacent perchlorate O atom [O2B(1 + x, 1 + y, z)], a distance consistent with O—H...O hydrogen bonding.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and IUCr *SHELXL97* template.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1610). Services for accessing these data are described at the back of the journal.

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Computing details

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and IUCr *SHELXL97* template.

Chloro(2-pyridinecarboxaldehyde)(2,2':6',2''-terpyridine)ruthenium(II) Perchlorate, Chloro(2-pyridinecarboxylic acid)(2,2':6',2''-terpyridine)ruthenium(II) Perchlorate, Chloroform, Hydrate (1.8/0.2/1/1)

Crystal data

$[\text{RuCl}(\text{C}_6\text{H}_5\text{NO})(\text{C}_{15}\text{H}_{11}\text{N}_3)]_{1.8}[\text{RuCl}(\text{C}_6\text{H}_5\text{NO}_2)(\text{C}_{15}\text{H}_{11}\text{N}_3)]_{0.2}(\text{ClO}_4)_2\cdot\text{CHCl}_3\cdot\text{H}_2\text{O}$
 $M_r = 1293.11$
 Triclinic, $P\bar{1}$
 $a = 10.8871$ (2) Å
 $b = 13.4187$ (3) Å
 $c = 17.9641$ (4) Å
 $\alpha = 68.7915$ (12)°
 $\beta = 83.8241$ (10)°
 $\gamma = 87.0651$ (11)°
 $V = 2432.23$ (9) Å³

$Z = 2$
 $F(000) = 1291$
 $D_x = 1.766$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 21341 reflections
 $\theta = 2.1\text{--}32.6^\circ$
 $\mu = 1.07$ mm⁻¹
 $T = 200$ K
 Prism, dark red
 $0.28 \times 0.20 \times 0.08$ mm

Data collection

Nonius KappaCCD Diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Phi plus ω Scan scans
 Absorption correction: multi-scan
 (*DENZO-SMN*; Otwinowski & Minor, 1997)
 $T_{\min} = 0.753$, $T_{\max} = 0.919$
 21341 measured reflections

16021 independent reflections
 11021 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\max} = 32.6^\circ$, $\theta_{\min} = 2.1^\circ$
 $h = -16 \rightarrow 16$
 $k = -19 \rightarrow 18$
 $l = -19 \rightarrow 27$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.116$
 $S = 1.02$
 16021 reflections

652 parameters
 1 restraint
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 2.243P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.89 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.89 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick,
1997), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0019 (3)

Special details

Experimental. The program *DENZO-SMN* (Otwinowski & Minor, 1997) uses a scaling algorithm (Fox & Holmes, 1966) which effectively corrects for absorption effects. High redundancy data were used in the scaling program hence the 'multi-scan' code word was used. No transmission coefficients are available from the program (only scale factors for each frame). The scale factors in the experimental table are calculated from the 'size' command in the *SHELXL97* input file. Data was collected to a maximum theta of 32.6 degrees with 90% completion prior to spontaneous fracture of the reported crystal. This represents the best result from several crystals which all fractured.

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.

Mean-plane data from final *SHELXL* refinement run:-

Least-squares planes (*x,y,z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

- 6.087 (0.007) *x* - 1.426 (0.011) *y* + 12.182 (0.011) *z* = 2.489 (0.008)

* 0.011 (0.001) Ru1A * -0.003 (0.001) N2A * -0.001 (0.001) N3A * -0.003 (0.001) N4A * -0.003 (0.001) O1A -1.990 (0.003) N1A 2.394 (0.001) C11A -3.000 (0.003) C1A -4.310 (0.003) C2A -4.632 (0.004) C3A -3.616 (0.004) C4A -2.307 (0.004) C5A -1.146 (0.004) C6A -0.013 (0.004) C7A 0.051 (0.005) C8A 0.087 (0.005) C9A 0.062 (0.005) C10A 0.033 (0.004) C11A 0.064 (0.004) C12A 0.199 (0.005) C13A 0.279 (0.005) C14A 0.219 (0.005) C15A 0.076 (0.004) C16A 0.029 (0.004) C17A 0.046 (0.005) C18A 0.041 (0.006) C19A 0.002 (0.006) C20A -0.013 (0.004) C21A

Rms deviation of fitted atoms = 0.005

- 6.379 (0.004) *x* - 0.989 (0.006) *y* + 12.077 (0.004) *z* = 2.630 (0.003)

Angle to previous plane (with approximate e.s.d.) = 2.65 (0.10)

* -0.016 (0.002) N2A * -0.081 (0.002) N3A * -0.004 (0.002) N4A * 0.013 (0.003) C7A * 0.056 (0.003) C8A * 0.030 (0.003) C9A * -0.036 (0.003) C10A * -0.042 (0.003) C11A * -0.050 (0.003) C12A * 0.021 (0.003) C13A * 0.074 (0.003) C14A * 0.049 (0.003) C15A * -0.030 (0.003) C16A * -0.032 (0.003) C17A * -0.030 (0.003) C18A * 0.012 (0.004) C19A * 0.034 (0.003) C20A * 0.031 (0.003) C21A

Rms deviation of fitted atoms = 0.041

6.107 (0.007) *x* + 9.246 (0.012) *y* + 13.626 (0.011) *z* = 8.282 (0.006)

Angle to previous plane (with approximate e.s.d.) = 87.39 (0.04)

* 0.028 (0.002) O1A * -0.009 (0.002) N1A * -0.008 (0.002) C1A * 0.010 (0.003) C2A * 0.012 (0.003) C3A * -0.001 (0.003) C4A * -0.013 (0.003) C5A * -0.019 (0.003) C6A

Rms deviation of fitted atoms = 0.015

- 4.626 (0.008) *x* - 1.192 (0.012) *y* + 13.797 (0.010) *z* = 6.448 (0.002)

Angle to previous plane (with approximate e.s.d.) = 80.39 (0.06)

* 0.013 (0.001) Ru1B * -0.010 (0.001) N2B * 0.006 (0.001) N3B * -0.010 (0.001) N4B * 0.002 (0.001) O1B -1.978 (0.003) N1B 2.409 (0.001) C11B -4.306 (0.004) C2B -4.606 (0.004) C3B -3.565 (0.004) C4B -2.280 (0.004) C5B -1.114 (0.004) C6B -0.033 (0.005) C7B -0.014 (0.006) C8B 0.012 (0.006) C9B 0.028 (0.005) C10B 0.018 (0.004) C11B 0.067 (0.004) C12B 0.219 (0.005) C13B 0.296 (0.005) C14B 0.213 (0.005) C15B 0.079 (0.004) C16B 0.027 (0.004) C17B -0.020 (0.005) C18B -0.134 (0.006) C19B -0.190 (0.005) C20B -0.112 (0.004) C21B

Rms deviation of fitted atoms = 0.009

- 5.274 (0.005) *x* - 1.004 (0.005) *y* + 13.302 (0.004) *z* = 6.454 (0.001)

Angle to previous plane (with approximate e.s.d.) = 3.93 (0.10)

* 0.026 (0.003) N2B * -0.040 (0.002) N3B * 0.075 (0.002) N4B * 0.050 (0.003) C7B * 0.027 (0.003) C8B * -0.041 (0.004) C9B * -0.075 (0.003) C10B * -0.040 (0.003) C11B * -0.037 (0.003) C12B * 0.021 (0.003) C13B * 0.067 (0.003) C14B * 0.044 (0.003) C15B * 0.004 (0.003) C16B * 0.026 (0.003) C17B * -0.035 (0.003) C18B * -0.074 (0.003) C19B * -0.044 (0.003) C20B * 0.046 (0.003) C21B

Rms deviation of fitted atoms = 0.047

- 3.468 (0.011) *x* + 12.882 (0.005) *y* + 7.917 (0.018) *z* = 0.327 (0.004)

Angle to previous plane (with approximate e.s.d.) = 82.16 (0.06)

* 0.010 (0.002) O1B * 0.014 (0.002) N1B * -0.007 (0.002) C1B * -0.011 (0.003) C2B * 0.005 (0.003) C3B * 0.009 (0.003) C4B * 0.007 (0.003) C5B * -0.027 (0.003) C6B

Rms deviation of fitted atoms = 0.013

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)
Ru1A	0.05068 (2)	0.447944 (18)	0.282976 (13)	0.02472 (7)	
Ru1B	-0.41894 (2)	-0.12194 (2)	0.317285 (13)	0.02683 (7)	

Cl1A	-0.08773 (7)	0.36858 (6)	0.40013 (4)	0.03124 (15)	
Cl1B	-0.52710 (7)	-0.19643 (7)	0.44823 (5)	0.03993 (19)	
Cl2B	-0.88429 (8)	-0.10861 (7)	0.18935 (5)	0.04210 (19)	
Cl2A	0.48865 (8)	0.29293 (7)	0.16219 (5)	0.0467 (2)	
Cl3	0.32203 (16)	0.34890 (19)	-0.07917 (10)	0.1254 (7)	
Cl4	0.18199 (15)	0.18206 (11)	0.04475 (9)	0.0878 (4)	
Cl5	0.11380 (13)	0.40157 (10)	0.01295 (8)	0.0757 (3)	
O1A	-0.0609 (2)	0.58256 (18)	0.24188 (14)	0.0366 (5)	
O1B	-0.5761 (2)	-0.0377 (2)	0.27106 (14)	0.0400 (5)	
O2A	0.5759 (3)	0.2096 (2)	0.16217 (18)	0.0565 (7)	
O2B	-0.9729 (3)	-0.1863 (3)	0.2384 (2)	0.0812 (10)	
O3A	0.3651 (3)	0.2537 (3)	0.1761 (2)	0.0764 (10)	
O3B	-0.9313 (3)	-0.0527 (3)	0.11434 (18)	0.0798 (11)	
O4A	0.5019 (4)	0.3764 (3)	0.0870 (2)	0.1066 (15)	
O4B	-0.7721 (3)	-0.1607 (3)	0.1765 (2)	0.0840 (11)	
O5A	0.5061 (4)	0.3289 (3)	0.2251 (2)	0.0883 (11)	
O5B	-0.8619 (4)	-0.0380 (3)	0.2292 (2)	0.0828 (10)	
O6	-0.8141 (3)	-0.1315 (3)	0.40466 (19)	0.0632 (8)	
H61	-0.826 (5)	-0.119 (4)	0.351 (2)	0.095*	
H62	-0.734 (4)	-0.150 (4)	0.420 (3)	0.095*	
N1A	0.1482 (2)	0.5351 (2)	0.17764 (14)	0.0283 (5)	
N1B	-0.3592 (2)	-0.0522 (2)	0.19903 (14)	0.0288 (5)	
N2A	0.1719 (2)	0.49094 (19)	0.34739 (13)	0.0254 (5)	
N2B	-0.3372 (3)	-0.0121 (2)	0.35253 (15)	0.0343 (6)	
N3A	0.1536 (2)	0.3187 (2)	0.31828 (14)	0.0276 (5)	
N3B	-0.2714 (2)	-0.2016 (2)	0.35935 (14)	0.0257 (5)	
N4A	-0.0285 (2)	0.3565 (2)	0.23151 (14)	0.0302 (5)	
N4B	-0.4417 (2)	-0.2608 (2)	0.29600 (14)	0.0304 (5)	
C1A	0.2572 (3)	0.5094 (3)	0.14624 (18)	0.0340 (7)	
H1A	0.2950	0.4425	0.1737	0.041*	
C1B	-0.2460 (3)	-0.0586 (3)	0.16262 (19)	0.0371 (7)	
H1B	-0.1815	-0.0944	0.1943	0.045*	
C2A	0.3159 (3)	0.5764 (3)	0.0758 (2)	0.0412 (8)	
H2A	0.3935	0.5560	0.0557	0.049*	
C2B	-0.2210 (3)	-0.0141 (3)	0.0799 (2)	0.0449 (8)	
H2B	-0.1401	-0.0201	0.0560	0.054*	
C3A	0.2629 (4)	0.6730 (3)	0.0342 (2)	0.0456 (9)	
H3A	0.3029	0.7197	-0.0147	0.055*	
C3B	-0.3130 (4)	0.0388 (3)	0.0319 (2)	0.0456 (8)	
H3B	-0.2969	0.0686	-0.0248	0.055*	
C4A	0.1506 (4)	0.7006 (3)	0.0648 (2)	0.0443 (8)	
H4A	0.1117	0.7668	0.0373	0.053*	
C4B	-0.4291 (3)	0.0469 (3)	0.06914 (19)	0.0416 (8)	
H4B	-0.4942	0.0832	0.0382	0.050*	
C5A	0.0953 (3)	0.6305 (3)	0.13631 (18)	0.0336 (7)	
C5B	-0.4495 (3)	0.0018 (3)	0.15159 (18)	0.0329 (6)	
C6A	-0.0228 (3)	0.6508 (3)	0.1751 (2)	0.0401 (8)	
H6A	-0.0697	0.7137	0.1507	0.048*	0.799 (10)

O1A2	−0.1101 (12)	0.7233 (11)	0.1503 (8)	0.054 (5)*	0.201 (10)
C6B	−0.5669 (3)	0.0081 (3)	0.1972 (2)	0.0419 (8)	
H6B	−0.6351	0.0465	0.1708	0.050*	
C7A	0.1743 (3)	0.5845 (3)	0.35878 (19)	0.0324 (6)	
H7A	0.1187	0.6403	0.3332	0.039*	
C7B	−0.3768 (4)	0.0870 (3)	0.3461 (2)	0.0465 (9)	
H7B	−0.4506	0.1139	0.3214	0.056*	
C8A	0.2562 (3)	0.6017 (3)	0.4069 (2)	0.0400 (8)	
H8A	0.2549	0.6676	0.4155	0.048*	
C8B	−0.3138 (5)	0.1513 (3)	0.3742 (3)	0.0629 (12)	
H8B	−0.3449	0.2206	0.3694	0.075*	
C9A	0.3389 (3)	0.5225 (3)	0.4420 (2)	0.0419 (8)	
H9A	0.3955	0.5332	0.4751	0.050*	
C9B	−0.2059 (5)	0.1142 (3)	0.4090 (2)	0.0632 (13)	
H9B	−0.1615	0.1576	0.4282	0.076*	
C10A	0.3398 (3)	0.4267 (3)	0.42917 (19)	0.0366 (7)	
H10A	0.3982	0.3719	0.4521	0.044*	
C10B	−0.1629 (4)	0.0126 (3)	0.4159 (2)	0.0491 (9)	
H10B	−0.0886	−0.0144	0.4398	0.059*	
C11A	0.2543 (3)	0.4120 (2)	0.38232 (16)	0.0283 (6)	
C11B	−0.2294 (3)	−0.0495 (3)	0.38745 (18)	0.0352 (7)	
C12A	0.2425 (3)	0.3124 (3)	0.36726 (17)	0.0295 (6)	
C12B	−0.1940 (3)	−0.1586 (3)	0.39347 (17)	0.0302 (6)	
C13A	0.3075 (3)	0.2167 (3)	0.39965 (19)	0.0358 (7)	
H13A	0.3706	0.2112	0.4336	0.043*	
C13B	−0.0967 (3)	−0.2199 (3)	0.43181 (18)	0.0381 (8)	
H13B	−0.0422	−0.1907	0.4562	0.046*	
C14A	0.2775 (3)	0.1294 (3)	0.3811 (2)	0.0433 (8)	
H14A	0.3206	0.0634	0.4028	0.052*	
C14B	−0.0800 (3)	−0.3230 (3)	0.43413 (19)	0.0404 (8)	
H14B	−0.0140	−0.3654	0.4607	0.048*	
C15A	0.1860 (3)	0.1372 (3)	0.3313 (2)	0.0410 (8)	
H15A	0.1660	0.0772	0.3189	0.049*	
C15B	−0.1580 (3)	−0.3652 (3)	0.39826 (18)	0.0353 (7)	
H15B	−0.1451	−0.4361	0.3989	0.042*	
C16A	0.1240 (3)	0.2338 (3)	0.29992 (18)	0.0326 (6)	
C16B	−0.2556 (3)	−0.3035 (2)	0.36118 (17)	0.0283 (6)	
C17A	0.0226 (3)	0.2573 (3)	0.24811 (18)	0.0340 (7)	
C17B	−0.3506 (3)	−0.3359 (3)	0.32270 (17)	0.0308 (6)	
C18A	−0.0227 (4)	0.1854 (3)	0.2184 (2)	0.0454 (8)	
H18A	0.0137	0.1163	0.2297	0.055*	
C18B	−0.3491 (3)	−0.4330 (3)	0.3114 (2)	0.0413 (8)	
H18B	−0.2863	−0.4848	0.3314	0.050*	
C19A	−0.1216 (4)	0.2162 (4)	0.1722 (2)	0.0548 (10)	
H19A	−0.1542	0.1676	0.1521	0.066*	
C19B	−0.4398 (4)	−0.4534 (3)	0.2710 (2)	0.0509 (10)	
H19B	−0.4401	−0.5192	0.2623	0.061*	
C20A	−0.1724 (4)	0.3162 (3)	0.1554 (2)	0.0497 (9)	

H20A	−0.2395	0.3383	0.1231	0.060*
C20B	−0.5299 (4)	−0.3770 (4)	0.2433 (2)	0.0521 (10)
H20B	−0.5922	−0.3894	0.2146	0.063*
C21A	−0.1239 (3)	0.3845 (3)	0.18635 (19)	0.0385 (7)
H21A	−0.1594	0.4539	0.1752	0.046*
C21B	−0.5298 (3)	−0.2826 (3)	0.25718 (19)	0.0399 (8)
H21B	−0.5938	−0.2314	0.2388	0.048*
C22	0.2381 (4)	0.3119 (4)	0.0144 (3)	0.0669 (12)
H22	0.2939	0.3145	0.0544	0.080*

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

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1A	0.02573 (11)	0.02453 (13)	0.02353 (11)	−0.00004 (9)	−0.00183 (8)	−0.00836 (9)
Ru1B	0.02474 (11)	0.03083 (14)	0.02533 (11)	0.00157 (9)	−0.00238 (9)	−0.01084 (9)
Cl1A	0.0330 (4)	0.0310 (4)	0.0284 (3)	−0.0038 (3)	0.0032 (3)	−0.0102 (3)
Cl1B	0.0324 (4)	0.0552 (5)	0.0283 (4)	0.0008 (4)	0.0002 (3)	−0.0114 (3)
Cl2B	0.0425 (4)	0.0415 (5)	0.0407 (4)	0.0035 (4)	−0.0067 (3)	−0.0126 (4)
Cl2A	0.0467 (5)	0.0424 (5)	0.0440 (5)	0.0031 (4)	0.0011 (4)	−0.0089 (4)
Cl3	0.0871 (10)	0.185 (2)	0.0696 (9)	−0.0056 (11)	0.0226 (8)	−0.0127 (11)
Cl4	0.1119 (11)	0.0629 (8)	0.0812 (9)	0.0103 (8)	0.0014 (8)	−0.0215 (7)
Cl5	0.0819 (8)	0.0635 (8)	0.0797 (8)	0.0045 (6)	−0.0153 (7)	−0.0220 (6)
O1A	0.0336 (11)	0.0374 (13)	0.0392 (12)	0.0052 (10)	−0.0052 (9)	−0.0144 (10)
O1B	0.0316 (11)	0.0473 (14)	0.0369 (12)	0.0089 (10)	−0.0043 (9)	−0.0109 (11)
O2A	0.0599 (17)	0.0418 (16)	0.0719 (19)	0.0078 (13)	−0.0058 (14)	−0.0264 (14)
O2B	0.083 (2)	0.073 (2)	0.069 (2)	−0.0236 (19)	0.0030 (18)	−0.0032 (18)
O3A	0.0461 (16)	0.098 (3)	0.075 (2)	−0.0138 (17)	0.0050 (15)	−0.0199 (19)
O3B	0.0577 (18)	0.103 (3)	0.0536 (18)	0.0056 (18)	−0.0188 (15)	0.0044 (18)
O4A	0.101 (3)	0.079 (3)	0.078 (2)	0.031 (2)	0.039 (2)	0.0279 (19)
O4B	0.066 (2)	0.117 (3)	0.084 (2)	0.043 (2)	−0.0243 (18)	−0.054 (2)
O5A	0.102 (3)	0.084 (3)	0.108 (3)	0.012 (2)	−0.015 (2)	−0.069 (2)
O5B	0.106 (3)	0.067 (2)	0.095 (3)	0.001 (2)	−0.017 (2)	−0.050 (2)
O6	0.0502 (16)	0.066 (2)	0.0647 (19)	−0.0063 (15)	−0.0068 (15)	−0.0125 (16)
N1A	0.0320 (12)	0.0270 (13)	0.0261 (12)	−0.0028 (10)	−0.0039 (10)	−0.0093 (10)
N1B	0.0331 (13)	0.0286 (13)	0.0254 (11)	−0.0015 (10)	−0.0031 (10)	−0.0104 (10)
N2A	0.0236 (11)	0.0276 (13)	0.0233 (11)	−0.0023 (9)	0.0011 (9)	−0.0078 (9)
N2B	0.0445 (15)	0.0320 (15)	0.0291 (13)	−0.0002 (12)	0.0015 (11)	−0.0153 (11)
N3A	0.0290 (12)	0.0267 (13)	0.0267 (12)	0.0020 (10)	−0.0007 (9)	−0.0100 (10)
N3B	0.0227 (11)	0.0307 (13)	0.0253 (11)	−0.0025 (10)	−0.0016 (9)	−0.0119 (10)
N4A	0.0330 (13)	0.0344 (14)	0.0257 (12)	−0.0032 (11)	−0.0012 (10)	−0.0140 (10)
N4B	0.0273 (12)	0.0370 (15)	0.0272 (12)	−0.0095 (11)	0.0000 (10)	−0.0113 (11)
C1A	0.0332 (15)	0.0379 (18)	0.0306 (15)	−0.0040 (13)	0.0008 (12)	−0.0125 (13)
C1B	0.0315 (15)	0.047 (2)	0.0341 (16)	−0.0034 (14)	−0.0005 (13)	−0.0166 (15)
C2A	0.0413 (18)	0.049 (2)	0.0345 (17)	−0.0131 (16)	0.0068 (14)	−0.0180 (15)
C2B	0.0401 (18)	0.057 (2)	0.0387 (18)	−0.0079 (17)	0.0061 (15)	−0.0197 (17)
C3A	0.062 (2)	0.047 (2)	0.0249 (15)	−0.0194 (18)	0.0038 (15)	−0.0089 (14)
C3B	0.060 (2)	0.046 (2)	0.0289 (16)	−0.0129 (18)	0.0007 (15)	−0.0119 (15)
C4A	0.064 (2)	0.0327 (18)	0.0306 (16)	−0.0078 (16)	−0.0052 (16)	−0.0029 (14)

C4B	0.055 (2)	0.0385 (19)	0.0308 (16)	−0.0026 (16)	−0.0123 (15)	−0.0097 (14)
C5A	0.0429 (17)	0.0293 (16)	0.0276 (14)	−0.0027 (13)	−0.0068 (13)	−0.0076 (12)
C5B	0.0369 (16)	0.0302 (16)	0.0309 (15)	−0.0014 (13)	−0.0069 (12)	−0.0089 (12)
C6A	0.0488 (19)	0.0313 (18)	0.0379 (17)	0.0083 (15)	−0.0129 (15)	−0.0082 (14)
C6B	0.0397 (18)	0.041 (2)	0.0429 (19)	0.0102 (15)	−0.0116 (15)	−0.0118 (15)
C7A	0.0331 (15)	0.0286 (16)	0.0358 (16)	−0.0053 (12)	0.0019 (12)	−0.0128 (13)
C7B	0.065 (2)	0.036 (2)	0.0373 (18)	0.0019 (17)	0.0058 (17)	−0.0151 (15)
C8A	0.0399 (17)	0.043 (2)	0.0416 (18)	−0.0105 (15)	−0.0007 (14)	−0.0201 (16)
C8B	0.107 (4)	0.039 (2)	0.047 (2)	−0.011 (2)	0.016 (2)	−0.0246 (18)
C9A	0.0375 (17)	0.053 (2)	0.0366 (17)	−0.0115 (16)	−0.0045 (14)	−0.0161 (16)
C9B	0.103 (4)	0.049 (2)	0.045 (2)	−0.036 (2)	0.007 (2)	−0.0252 (19)
C10A	0.0294 (15)	0.0424 (19)	0.0336 (16)	−0.0019 (13)	−0.0034 (12)	−0.0080 (14)
C10B	0.063 (2)	0.053 (2)	0.0375 (18)	−0.0264 (19)	0.0007 (17)	−0.0212 (17)
C11A	0.0257 (13)	0.0323 (16)	0.0232 (13)	−0.0027 (12)	0.0017 (10)	−0.0062 (11)
C11B	0.0402 (17)	0.0400 (18)	0.0265 (14)	−0.0118 (14)	0.0022 (12)	−0.0131 (13)
C12A	0.0285 (14)	0.0344 (17)	0.0220 (13)	0.0012 (12)	0.0020 (11)	−0.0074 (12)
C12B	0.0271 (14)	0.0393 (17)	0.0255 (13)	−0.0080 (12)	0.0011 (11)	−0.0133 (12)
C13A	0.0348 (16)	0.0358 (18)	0.0308 (15)	0.0056 (14)	−0.0005 (12)	−0.0062 (13)
C13B	0.0253 (14)	0.063 (2)	0.0296 (15)	−0.0036 (15)	−0.0025 (12)	−0.0210 (15)
C14A	0.051 (2)	0.0294 (18)	0.0391 (18)	0.0093 (15)	0.0024 (15)	−0.0027 (14)
C14B	0.0295 (15)	0.061 (2)	0.0301 (15)	0.0128 (15)	−0.0053 (12)	−0.0167 (15)
C15A	0.053 (2)	0.0274 (17)	0.0399 (18)	0.0036 (15)	0.0012 (15)	−0.0109 (14)
C15B	0.0370 (16)	0.0392 (18)	0.0288 (15)	0.0085 (14)	0.0017 (12)	−0.0136 (13)
C16A	0.0378 (16)	0.0293 (16)	0.0290 (14)	−0.0021 (13)	0.0046 (12)	−0.0105 (12)
C16B	0.0282 (14)	0.0324 (16)	0.0251 (13)	−0.0012 (12)	0.0027 (11)	−0.0127 (12)
C17A	0.0403 (17)	0.0344 (17)	0.0306 (15)	−0.0041 (14)	0.0018 (13)	−0.0165 (13)
C17B	0.0310 (15)	0.0352 (17)	0.0269 (14)	−0.0039 (13)	0.0042 (11)	−0.0135 (12)
C18A	0.062 (2)	0.040 (2)	0.0387 (18)	−0.0048 (17)	−0.0001 (17)	−0.0207 (16)
C18B	0.0455 (19)	0.0362 (19)	0.0458 (19)	−0.0068 (15)	0.0079 (15)	−0.0216 (16)
C19A	0.066 (3)	0.062 (3)	0.047 (2)	−0.022 (2)	−0.0027 (19)	−0.030 (2)
C19B	0.059 (2)	0.051 (2)	0.051 (2)	−0.025 (2)	0.0170 (18)	−0.0314 (19)
C20A	0.050 (2)	0.060 (3)	0.047 (2)	−0.0087 (19)	−0.0115 (17)	−0.0257 (19)
C20B	0.049 (2)	0.076 (3)	0.042 (2)	−0.029 (2)	0.0046 (16)	−0.032 (2)
C21A	0.0367 (17)	0.047 (2)	0.0340 (16)	−0.0031 (15)	−0.0049 (13)	−0.0166 (15)
C21B	0.0339 (16)	0.057 (2)	0.0307 (16)	−0.0146 (15)	−0.0034 (13)	−0.0155 (15)
C22	0.063 (3)	0.076 (3)	0.052 (2)	−0.004 (2)	−0.010 (2)	−0.010 (2)

Geometric parameters (Å, °)

Ru1A—Cl1A	2.3863 (7)	C4B—H4B	0.9500
Ru1A—O1A	2.071 (2)	C5A—C6A	1.456 (5)
Ru1A—N1A	2.042 (2)	C5B—C6B	1.458 (5)
Ru1A—N2A	2.074 (2)	C6A—O1A2	1.316 (14)
Ru1A—N3A	1.956 (2)	C6A—H6A	0.9500
Ru1A—N4A	2.051 (2)	C6B—H6B	0.9500
Ru1B—Cl1B	2.3963 (8)	C7A—C8A	1.388 (4)
Ru1B—O1B	2.079 (2)	C7A—H7A	0.9500
Ru1B—N1B	2.034 (2)	C7B—C8B	1.386 (6)

Ru1B—N2B	2.072 (3)	C7B—H7B	0.9500
Ru1B—N3B	1.948 (2)	C8A—C9A	1.370 (5)
Ru1B—N4B	2.065 (3)	C8A—H8A	0.9500
Cl2B—O4B	1.415 (3)	C8B—C9B	1.375 (7)
Cl2B—O3B	1.421 (3)	C8B—H8B	0.9500
Cl2B—O5B	1.422 (3)	C9A—C10A	1.385 (5)
Cl2B—O2B	1.432 (3)	C9A—H9A	0.9500
Cl2A—O4A	1.408 (3)	C9B—C10B	1.385 (6)
Cl2A—O5A	1.414 (3)	C9B—H9B	0.9500
Cl2A—O2A	1.430 (3)	C10A—C11A	1.387 (4)
Cl2A—O3A	1.433 (3)	C10A—H10A	0.9500
Cl3—C22	1.737 (5)	C10B—C11B	1.388 (5)
Cl4—C22	1.748 (5)	C10B—H10B	0.9500
Cl5—C22	1.760 (5)	C11A—C12A	1.471 (4)
O1A—C6A	1.260 (4)	C11B—C12B	1.463 (5)
O1B—C6B	1.239 (4)	C12A—C13A	1.394 (4)
O6—H61	0.93 (3)	C12B—C13B	1.390 (4)
O6—H62	0.93 (3)	C13A—C14A	1.391 (5)
N1A—C1A	1.342 (4)	C13A—H13A	0.9500
N1A—C5A	1.360 (4)	C13B—C14B	1.371 (5)
N1B—C1B	1.345 (4)	C13B—H13B	0.9500
N1B—C5B	1.370 (4)	C14A—C15A	1.385 (5)
N2A—C7A	1.346 (4)	C14A—H14A	0.9500
N2A—C11A	1.365 (4)	C14B—C15B	1.375 (5)
N2B—C7B	1.345 (4)	C14B—H14B	0.9500
N2B—C11B	1.376 (4)	C15A—C16A	1.385 (4)
N3A—C12A	1.355 (4)	C15A—H15A	0.9500
N3A—C16A	1.357 (4)	C15B—C16B	1.387 (4)
N3B—C12B	1.355 (4)	C15B—H15B	0.9500
N3B—C16B	1.358 (4)	C16A—C17A	1.469 (4)
N4A—C21A	1.341 (4)	C16B—C17B	1.468 (4)
N4A—C17A	1.357 (4)	C17A—C18A	1.392 (5)
N4B—C21B	1.346 (4)	C17B—C18B	1.389 (4)
N4B—C17B	1.370 (4)	C18A—C19A	1.385 (6)
C1A—C2A	1.371 (4)	C18A—H18A	0.9500
C1A—H1A	0.9500	C18B—C19B	1.380 (5)
C1B—C2B	1.388 (5)	C18B—H18B	0.9500
C1B—H1B	0.9500	C19A—C20A	1.365 (6)
C2A—C3A	1.373 (5)	C19A—H19A	0.9500
C2A—H2A	0.9500	C19B—C20B	1.376 (6)
C2B—C3B	1.386 (5)	C19B—H19B	0.9500
C2B—H2B	0.9500	C20A—C21A	1.383 (5)
C3A—C4A	1.375 (5)	C20A—H20A	0.9500
C3A—H3A	0.9500	C20B—C21B	1.377 (5)
C3B—C4B	1.382 (5)	C20B—H20B	0.9500
C3B—H3B	0.9500	C21A—H21A	0.9500
C4A—C5A	1.384 (4)	C21B—H21B	0.9500
C4A—H4A	0.9500	C22—H22	1.0000

C4B—C5B	1.380 (4)		
Cl1A—Ru1A—O1A	92.50 (7)	O1A—C6A—H6A	120.9
Cl1A—Ru1A—N1A	170.75 (7)	O1A2—C6A—H6A	13.6
Cl1A—Ru1A—N2A	92.08 (7)	C5A—C6A—H6A	120.9
Cl1A—Ru1A—N3A	88.14 (7)	O1B—C6B—C5B	118.8 (3)
Cl1A—Ru1A—N4A	87.90 (7)	O1B—C6B—H6B	120.6
O1A—Ru1A—N1A	78.27 (9)	C5B—C6B—H6B	120.6
O1A—Ru1A—N2A	103.23 (9)	N2A—C7A—C8A	121.6 (3)
O1A—Ru1A—N3A	177.51 (9)	N2A—C7A—H7A	119.2
O1A—Ru1A—N4A	97.80 (10)	C8A—C7A—H7A	119.2
N1A—Ru1A—N2A	90.62 (9)	N2B—C7B—C8B	122.3 (4)
N1A—Ru1A—N3A	101.06 (10)	N2B—C7B—H7B	118.9
N1A—Ru1A—N4A	92.75 (10)	C8B—C7B—H7B	118.9
N2A—Ru1A—N3A	79.14 (10)	C9A—C8A—C7A	119.3 (3)
N2A—Ru1A—N4A	158.94 (10)	C9A—C8A—H8A	120.4
N3A—Ru1A—N4A	79.81 (10)	C7A—C8A—H8A	120.4
Cl1B—Ru1B—O1B	91.32 (7)	C9B—C8B—C7B	119.6 (4)
Cl1B—Ru1B—N1B	169.29 (7)	C9B—C8B—H8B	120.2
Cl1B—Ru1B—N2B	89.95 (8)	C7B—C8B—H8B	120.2
Cl1B—Ru1B—N3B	89.21 (7)	C8A—C9A—C10A	119.8 (3)
Cl1B—Ru1B—N4B	90.95 (7)	C8A—C9A—H9A	120.1
O1B—Ru1B—N1B	78.44 (9)	C10A—C9A—H9A	120.1
O1B—Ru1B—N2B	101.24 (10)	C8B—C9B—C10B	119.2 (4)
O1B—Ru1B—N3B	179.18 (9)	C8B—C9B—H9B	120.4
O1B—Ru1B—N4B	99.67 (10)	C10B—C9B—H9B	120.4
N1B—Ru1B—N2B	95.05 (10)	C9A—C10A—C11A	119.0 (3)
N1B—Ru1B—N3B	101.00 (10)	C9A—C10A—H10A	120.5
N1B—Ru1B—N4B	87.75 (10)	C11A—C10A—H10A	120.5
N2B—Ru1B—N3B	79.39 (10)	C9B—C10B—C11B	119.4 (4)
N2B—Ru1B—N4B	159.05 (10)	C9B—C10B—H10B	120.3
N3B—Ru1B—N4B	79.69 (10)	C11B—C10B—H10B	120.3
O4B—Cl2B—O3B	109.2 (2)	N2A—C11A—C10A	121.2 (3)
O4B—Cl2B—O5B	108.9 (2)	N2A—C11A—C12A	114.8 (2)
O3B—Cl2B—O5B	111.4 (2)	C10A—C11A—C12A	124.0 (3)
O4B—Cl2B—O2B	109.6 (2)	N2B—C11B—C10B	121.4 (3)
O3B—Cl2B—O2B	108.5 (2)	N2B—C11B—C12B	114.8 (3)
O5B—Cl2B—O2B	109.3 (2)	C10B—C11B—C12B	123.9 (3)
O4A—Cl2A—O5A	111.6 (3)	N3A—C12A—C13A	119.8 (3)
O4A—Cl2A—O2A	109.15 (19)	N3A—C12A—C11A	112.8 (2)
O5A—Cl2A—O2A	109.9 (2)	C13A—C12A—C11A	127.3 (3)
O4A—Cl2A—O3A	108.6 (2)	N3B—C12B—C13B	119.6 (3)
O5A—Cl2A—O3A	107.1 (2)	N3B—C12B—C11B	113.0 (3)
O2A—Cl2A—O3A	110.4 (2)	C13B—C12B—C11B	127.4 (3)
C6A—O1A—Ru1A	115.2 (2)	C14A—C13A—C12A	118.3 (3)
C6B—O1B—Ru1B	114.9 (2)	C14A—C13A—H13A	120.8
H61—O6—H62	118 (5)	C12A—C13A—H13A	120.8
C1A—N1A—C5A	117.7 (3)	C14B—C13B—C12B	119.5 (3)

C1A—N1A—Ru1A	127.5 (2)	C14B—C13B—H13B	120.3
C5A—N1A—Ru1A	114.9 (2)	C12B—C13B—H13B	120.3
C1B—N1B—C5B	117.6 (3)	C15A—C14A—C13A	121.0 (3)
C1B—N1B—Ru1B	128.0 (2)	C15A—C14A—H14A	119.5
C5B—N1B—Ru1B	114.3 (2)	C13A—C14A—H14A	119.5
C7A—N2A—C11A	119.1 (3)	C13B—C14B—C15B	120.4 (3)
C7A—N2A—Ru1A	127.1 (2)	C13B—C14B—H14B	119.8
C11A—N2A—Ru1A	113.79 (19)	C15B—C14B—H14B	119.8
C7B—N2B—C11B	118.2 (3)	C14A—C15A—C16A	118.9 (3)
C7B—N2B—Ru1B	128.4 (3)	C14A—C15A—H15A	120.6
C11B—N2B—Ru1B	113.3 (2)	C16A—C15A—H15A	120.6
C12A—N3A—C16A	122.1 (3)	C14B—C15B—C16B	119.4 (3)
C12A—N3A—Ru1A	119.4 (2)	C14B—C15B—H15B	120.3
C16A—N3A—Ru1A	118.1 (2)	C16B—C15B—H15B	120.3
C12B—N3B—C16B	121.5 (2)	N3A—C16A—C15A	119.8 (3)
C12B—N3B—Ru1B	119.4 (2)	N3A—C16A—C17A	113.1 (3)
C16B—N3B—Ru1B	118.79 (19)	C15A—C16A—C17A	127.1 (3)
C21A—N4A—C17A	119.3 (3)	N3B—C16B—C15B	119.7 (3)
C21A—N4A—Ru1A	126.8 (2)	N3B—C16B—C17B	113.1 (2)
C17A—N4A—Ru1A	114.0 (2)	C15B—C16B—C17B	127.2 (3)
C21B—N4B—C17B	118.4 (3)	N4A—C17A—C18A	120.6 (3)
C21B—N4B—Ru1B	128.0 (2)	N4A—C17A—C16A	114.8 (3)
C17B—N4B—Ru1B	113.53 (19)	C18A—C17A—C16A	124.6 (3)
N1A—C1A—C2A	122.1 (3)	N4B—C17B—C18B	121.4 (3)
N1A—C1A—H1A	118.9	N4B—C17B—C16B	114.7 (3)
C2A—C1A—H1A	118.9	C18B—C17B—C16B	123.9 (3)
N1B—C1B—C2B	121.7 (3)	C19A—C18A—C17A	119.0 (4)
N1B—C1B—H1B	119.1	C19A—C18A—H18A	120.5
C2B—C1B—H1B	119.1	C17A—C18A—H18A	120.5
C1A—C2A—C3A	120.3 (3)	C19B—C18B—C17B	119.2 (3)
C1A—C2A—H2A	119.8	C19B—C18B—H18B	120.4
C3A—C2A—H2A	119.8	C17B—C18B—H18B	120.4
C3B—C2B—C1B	120.6 (3)	C20A—C19A—C18A	120.2 (4)
C3B—C2B—H2B	119.7	C20A—C19A—H19A	119.9
C1B—C2B—H2B	119.7	C18A—C19A—H19A	119.9
C2A—C3A—C4A	118.6 (3)	C20B—C19B—C18B	119.0 (3)
C2A—C3A—H3A	120.7	C20B—C19B—H19B	120.5
C4A—C3A—H3A	120.7	C18B—C19B—H19B	120.5
C4B—C3B—C2B	117.9 (3)	C19A—C20A—C21A	118.5 (4)
C4B—C3B—H3B	121.0	C19A—C20A—H20A	120.8
C2B—C3B—H3B	121.0	C21A—C20A—H20A	120.8
C3A—C4A—C5A	119.0 (3)	C19B—C20B—C21B	120.1 (3)
C3A—C4A—H4A	120.5	C19B—C20B—H20B	120.0
C5A—C4A—H4A	120.5	C21B—C20B—H20B	120.0
C5B—C4B—C3B	119.5 (3)	N4A—C21A—C20A	122.5 (4)
C5B—C4B—H4B	120.3	N4A—C21A—H21A	118.8
C3B—C4B—H4B	120.3	C20A—C21A—H21A	118.8
N1A—C5A—C4A	122.3 (3)	N4B—C21B—C20B	121.9 (3)

N1A—C5A—C6A	113.5 (3)	N4B—C21B—H21B	119.1
C4A—C5A—C6A	124.2 (3)	C20B—C21B—H21B	119.1
N1B—C5B—C4B	122.7 (3)	Cl3—C22—Cl4	111.6 (3)
N1B—C5B—C6B	113.2 (3)	Cl3—C22—Cl5	110.4 (3)
C4B—C5B—C6B	124.1 (3)	Cl4—C22—Cl5	109.4 (3)
O1A—C6A—O1A2	109.0 (7)	Cl3—C22—H22	108.4
O1A—C6A—C5A	118.1 (3)	Cl4—C22—H22	108.4
O1A2—C6A—C5A	132.4 (7)	Cl5—C22—H22	108.4
N1A—Ru1A—O1A—C6A	1.4 (2)	Ru1B—N1B—C5B—C6B	6.5 (3)
N4A—Ru1A—O1A—C6A	−89.8 (2)	C3B—C4B—C5B—N1B	−0.5 (5)
N2A—Ru1A—O1A—C6A	89.2 (2)	C3B—C4B—C5B—C6B	178.2 (3)
Cl1A—Ru1A—O1A—C6A	−178.0 (2)	Ru1A—O1A—C6A—O1A2	170.3 (7)
N1B—Ru1B—O1B—C6B	4.3 (3)	Ru1A—O1A—C6A—C5A	−2.5 (4)
N4B—Ru1B—O1B—C6B	−81.4 (3)	N1A—C5A—C6A—O1A	2.4 (4)
N2B—Ru1B—O1B—C6B	97.2 (3)	C4A—C5A—C6A—O1A	−177.0 (3)
Cl1B—Ru1B—O1B—C6B	−172.6 (3)	N1A—C5A—C6A—O1A2	−168.4 (9)
N3A—Ru1A—N1A—C1A	−3.5 (3)	C4A—C5A—C6A—O1A2	12.2 (10)
N4A—Ru1A—N1A—C1A	−83.6 (3)	Ru1B—O1B—C6B—C5B	−2.0 (4)
O1A—Ru1A—N1A—C1A	179.0 (3)	N1B—C5B—C6B—O1B	−3.0 (5)
N2A—Ru1A—N1A—C1A	75.6 (3)	C4B—C5B—C6B—O1B	178.2 (3)
N3A—Ru1A—N1A—C5A	177.5 (2)	C11A—N2A—C7A—C8A	−1.8 (4)
N4A—Ru1A—N1A—C5A	97.3 (2)	Ru1A—N2A—C7A—C8A	176.5 (2)
O1A—Ru1A—N1A—C5A	−0.1 (2)	C11B—N2B—C7B—C8B	−0.8 (5)
N2A—Ru1A—N1A—C5A	−103.4 (2)	Ru1B—N2B—C7B—C8B	178.4 (3)
N3B—Ru1B—N1B—C1B	−2.0 (3)	N2A—C7A—C8A—C9A	1.8 (5)
N4B—Ru1B—N1B—C1B	−81.0 (3)	N2B—C7B—C8B—C9B	0.9 (6)
N2B—Ru1B—N1B—C1B	78.2 (3)	C7A—C8A—C9A—C10A	−0.1 (5)
O1B—Ru1B—N1B—C1B	178.6 (3)	C7B—C8B—C9B—C10B	−0.5 (6)
Cl1B—Ru1B—N1B—C1B	−164.2 (3)	C8A—C9A—C10A—C11A	−1.6 (5)
N3B—Ru1B—N1B—C5B	173.6 (2)	C8B—C9B—C10B—C11B	0.0 (6)
N4B—Ru1B—N1B—C5B	94.5 (2)	C7A—N2A—C11A—C10A	0.1 (4)
N2B—Ru1B—N1B—C5B	−106.3 (2)	Ru1A—N2A—C11A—C10A	−178.4 (2)
O1B—Ru1B—N1B—C5B	−5.8 (2)	C7A—N2A—C11A—C12A	179.0 (3)
Cl1B—Ru1B—N1B—C5B	11.3 (6)	Ru1A—N2A—C11A—C12A	0.5 (3)
N3A—Ru1A—N2A—C7A	179.5 (3)	C9A—C10A—C11A—N2A	1.6 (4)
N1A—Ru1A—N2A—C7A	78.4 (2)	C9A—C10A—C11A—C12A	−177.2 (3)
N4A—Ru1A—N2A—C7A	177.6 (3)	C7B—N2B—C11B—C10B	0.3 (5)
O1A—Ru1A—N2A—C7A	0.3 (3)	Ru1B—N2B—C11B—C10B	−179.0 (3)
Cl1A—Ru1A—N2A—C7A	−92.8 (2)	C7B—N2B—C11B—C12B	178.9 (3)
N3A—Ru1A—N2A—C11A	−2.15 (19)	Ru1B—N2B—C11B—C12B	−0.4 (3)
N1A—Ru1A—N2A—C11A	−103.29 (19)	C9B—C10B—C11B—N2B	0.0 (5)
N4A—Ru1A—N2A—C11A	−4.0 (4)	C9B—C10B—C11B—C12B	−178.4 (3)
O1A—Ru1A—N2A—C11A	178.61 (18)	C16A—N3A—C12A—C13A	0.8 (4)
Cl1A—Ru1A—N2A—C11A	85.55 (18)	Ru1A—N3A—C12A—C13A	173.2 (2)
N3B—Ru1B—N2B—C7B	179.1 (3)	C16A—N3A—C12A—C11A	−176.8 (3)
N1B—Ru1B—N2B—C7B	78.8 (3)	Ru1A—N3A—C12A—C11A	−4.4 (3)
N4B—Ru1B—N2B—C7B	175.8 (3)	N2A—C11A—C12A—N3A	2.3 (4)

O1B—Ru1B—N2B—C7B	−0.3 (3)	C10A—C11A—C12A—N3A	−178.8 (3)
Cl1B—Ru1B—N2B—C7B	−91.7 (3)	N2A—C11A—C12A—C13A	−175.0 (3)
N3B—Ru1B—N2B—C11B	−1.6 (2)	C10A—C11A—C12A—C13A	3.9 (5)
N1B—Ru1B—N2B—C11B	−101.9 (2)	C16B—N3B—C12B—C13B	−0.5 (4)
N4B—Ru1B—N2B—C11B	−5.0 (4)	Ru1B—N3B—C12B—C13B	172.6 (2)
O1B—Ru1B—N2B—C11B	178.9 (2)	C16B—N3B—C12B—C11B	−178.0 (3)
Cl1B—Ru1B—N2B—C11B	87.6 (2)	Ru1B—N3B—C12B—C11B	−4.9 (3)
N1A—Ru1A—N3A—C12A	92.2 (2)	N2B—C11B—C12B—N3B	3.3 (4)
N4A—Ru1A—N3A—C12A	−177.0 (2)	C10B—C11B—C12B—N3B	−178.2 (3)
N2A—Ru1A—N3A—C12A	3.7 (2)	N2B—C11B—C12B—C13B	−174.0 (3)
Cl1A—Ru1A—N3A—C12A	−88.8 (2)	C10B—C11B—C12B—C13B	4.6 (5)
N1A—Ru1A—N3A—C16A	−95.1 (2)	N3A—C12A—C13A—C14A	−0.7 (4)
N4A—Ru1A—N3A—C16A	−4.3 (2)	C11A—C12A—C13A—C14A	176.5 (3)
N2A—Ru1A—N3A—C16A	176.4 (2)	N3B—C12B—C13B—C14B	0.4 (4)
Cl1A—Ru1A—N3A—C16A	83.9 (2)	C11B—C12B—C13B—C14B	177.5 (3)
N1B—Ru1B—N3B—C12B	96.9 (2)	C12A—C13A—C14A—C15A	0.4 (5)
N4B—Ru1B—N3B—C12B	−177.5 (2)	C12B—C13B—C14B—C15B	0.6 (5)
N2B—Ru1B—N3B—C12B	3.7 (2)	C13A—C14A—C15A—C16A	0.0 (5)
Cl1B—Ru1B—N3B—C12B	−86.4 (2)	C13B—C14B—C15B—C16B	−1.4 (5)
N1B—Ru1B—N3B—C16B	−89.9 (2)	C12A—N3A—C16A—C15A	−0.4 (4)
N4B—Ru1B—N3B—C16B	−4.3 (2)	Ru1A—N3A—C16A—C15A	−172.9 (2)
N2B—Ru1B—N3B—C16B	176.9 (2)	C12A—N3A—C16A—C17A	178.1 (3)
Cl1B—Ru1B—N3B—C16B	86.8 (2)	Ru1A—N3A—C16A—C17A	5.6 (3)
N3A—Ru1A—N4A—C21A	−179.5 (3)	C14A—C15A—C16A—N3A	0.0 (5)
N1A—Ru1A—N4A—C21A	−78.7 (3)	C14A—C15A—C16A—C17A	−178.2 (3)
O1A—Ru1A—N4A—C21A	−0.2 (3)	C12B—N3B—C16B—C15B	−0.4 (4)
N2A—Ru1A—N4A—C21A	−177.6 (2)	Ru1B—N3B—C16B—C15B	−173.5 (2)
Cl1A—Ru1A—N4A—C21A	92.0 (3)	C12B—N3B—C16B—C17B	178.6 (2)
N3A—Ru1A—N4A—C17A	2.0 (2)	Ru1B—N3B—C16B—C17B	5.5 (3)
N1A—Ru1A—N4A—C17A	102.7 (2)	C14B—C15B—C16B—N3B	1.3 (4)
O1A—Ru1A—N4A—C17A	−178.8 (2)	C14B—C15B—C16B—C17B	−177.5 (3)
N2A—Ru1A—N4A—C17A	3.8 (4)	C21A—N4A—C17A—C18A	0.3 (5)
Cl1A—Ru1A—N4A—C17A	−86.5 (2)	Ru1A—N4A—C17A—C18A	179.0 (2)
N3B—Ru1B—N4B—C21B	−174.9 (3)	C21A—N4A—C17A—C16A	−178.3 (3)
N1B—Ru1B—N4B—C21B	−73.3 (3)	Ru1A—N4A—C17A—C16A	0.4 (3)
N2B—Ru1B—N4B—C21B	−171.6 (3)	N3A—C16A—C17A—N4A	−3.7 (4)
O1B—Ru1B—N4B—C21B	4.6 (3)	C15A—C16A—C17A—N4A	174.6 (3)
Cl1B—Ru1B—N4B—C21B	96.1 (2)	N3A—C16A—C17A—C18A	177.7 (3)
N3B—Ru1B—N4B—C17B	2.1 (2)	C15A—C16A—C17A—C18A	−3.9 (5)
N1B—Ru1B—N4B—C17B	103.7 (2)	C21B—N4B—C17B—C18B	−0.9 (4)
N2B—Ru1B—N4B—C17B	5.4 (4)	Ru1B—N4B—C17B—C18B	−178.2 (2)
O1B—Ru1B—N4B—C17B	−178.46 (19)	C21B—N4B—C17B—C16B	177.5 (3)
Cl1B—Ru1B—N4B—C17B	−86.96 (19)	Ru1B—N4B—C17B—C16B	0.2 (3)
C5A—N1A—C1A—C2A	1.1 (4)	N3B—C16B—C17B—N4B	−3.5 (4)
Ru1A—N1A—C1A—C2A	−177.9 (2)	C15B—C16B—C17B—N4B	175.4 (3)
C5B—N1B—C1B—C2B	−1.2 (5)	N3B—C16B—C17B—C18B	174.8 (3)
Ru1B—N1B—C1B—C2B	174.3 (3)	C15B—C16B—C17B—C18B	−6.3 (5)
N1A—C1A—C2A—C3A	−0.8 (5)	N4A—C17A—C18A—C19A	−0.6 (5)

N1B—C1B—C2B—C3B	0.1 (6)	C16A—C17A—C18A—C19A	177.9 (3)
C1A—C2A—C3A—C4A	0.2 (5)	N4B—C17B—C18B—C19B	1.4 (5)
C1B—C2B—C3B—C4B	0.8 (6)	C16B—C17B—C18B—C19B	−176.8 (3)
C2A—C3A—C4A—C5A	0.1 (5)	C17A—C18A—C19A—C20A	0.9 (6)
C2B—C3B—C4B—C5B	−0.6 (5)	C17B—C18B—C19B—C20B	−0.4 (5)
C1A—N1A—C5A—C4A	−0.8 (4)	C18A—C19A—C20A—C21A	−0.8 (6)
Ru1A—N1A—C5A—C4A	178.3 (3)	C18B—C19B—C20B—C21B	−1.0 (5)
C1A—N1A—C5A—C6A	179.8 (3)	C17A—N4A—C21A—C20A	−0.3 (5)
Ru1A—N1A—C5A—C6A	−1.1 (3)	Ru1A—N4A—C21A—C20A	−178.8 (3)
C3A—C4A—C5A—N1A	0.2 (5)	C19A—C20A—C21A—N4A	0.6 (6)
C3A—C4A—C5A—C6A	179.6 (3)	C17B—N4B—C21B—C20B	−0.6 (5)
C1B—N1B—C5B—C4B	1.4 (5)	Ru1B—N4B—C21B—C20B	176.3 (2)
Ru1B—N1B—C5B—C4B	−174.6 (3)	C19B—C20B—C21B—N4B	1.5 (5)
C1B—N1B—C5B—C6B	−177.5 (3)		

Hydrogen-bond geometry (Å, °)

<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>
O6—H61 $\cdots$ O5 <i>B</i>	0.93 (3)	2.13 (4)	3.034 (5)	161 (5)
O6—H62 $\cdots$ C11 <i>B</i>	0.93 (3)	2.37 (4)	3.300 (3)	175 (5)
C22—H22 $\cdots$ O3 <i>A</i>	1.00	2.25	3.174 (6)	153