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Dicarbonyldichlorido(*N,N,N',N'*-tetramethylethylenediamine)ruthenium(II)

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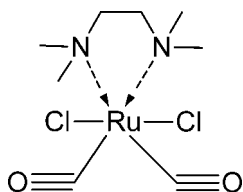
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 Key indicators: single-crystal X-ray study; $T = 160$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.023; wR factor = 0.061; data-to-parameter ratio = 15.6.

In the title compound, $[\text{RuCl}_2(\text{C}_6\text{H}_{16}\text{N}_2)(\text{CO})_2]$, the geometry around the Ru^{II} atom is a distorted $\text{RuC}_2\text{N}_2\text{Cl}_2$ octahedron, with pairs of C and Cl atoms *trans* to each other and the N atoms of the bidentate ligand in a *cis* conformation. The five-membered chelate ring is puckered on the C–C bond.

Related literature

For background to ruthenium carbonyl derivatives, see: Manchot & Konig (1924); Stephenson & Wilkinson (1966); Kingston *et al.* (1967); Baghlaf *et al.* (2007); Campbell (1975); Padhey & Kaufman (1985). For a related structure, see: Bakar *et al.* (1993).



Experimental

Crystal data

$[\text{RuCl}_2(\text{C}_6\text{H}_{16}\text{N}_2)(\text{CO})_2]$
 $M_r = 344.20$
 Monoclinic, $P2_1/c$
 $a = 7.463$ (6) Å
 $b = 14.579$ (6) Å
 $c = 12.718$ (12) Å
 $\beta = 106.37$ (8)°

$V = 1327.7$ (17) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.57$ mm⁻¹
 $T = 160$ K
 $0.38 \times 0.38 \times 0.25$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\text{min}} = 0.591$, $T_{\text{max}} = 0.69$
 3153 measured reflections

2877 independent reflections
 2644 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 2 standard reflections every 100 reflections
 intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.061$
 $S = 1.07$
 2877 reflections
 184 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.06$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ru1–C1	1.872 (3)	Ru1–N1	2.220 (2)
Ru1–C2	1.872 (2)	Ru1–Cl1	2.413 (2)
Ru1–N2	2.211 (2)	Ru1–Cl2	2.408 (2)
<hr/>			
N2–Ru1–N1	82.75 (9)		

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5901).

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Dicarbonyldichlorido(*N,N,N',N'*-tetramethylethylenediamine)ruthenium(II)

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S1. Comment

The salt $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ was first reported by (Manchot & Konig, 1924) but its importance and chemistry was shown in late 1960's by (Stephenson & Wilkinson, 1966), (Kingston *et al.*, 1967) who have reported several compounds of the type $[\text{Ru}(\text{CO})_2\text{Cl}_2\text{L}_2]$ where L =monodentate ligand. This was due to the fact that the salt $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ has proved a useful precursor for the synthesis of a variety of organometallic compounds. We have also reported from our laboratories compounds with ligands containing N, O and S atom as electron donor (e.g. Baghlaf *et al.*, 2007). However (Campbell (1975) and Padhey & Kaufman (1985) have reported about remarkable biological activities of such compounds against microbes, viruses and tumours. This has been the main reason for our research activity in the field of bio-inorganic chemistry of transition metal complexes. The metal atom Ru in the salt $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ being electron deficient acts as electron acceptor. This enhances its ability to coordinate with electron donor ligand (TMEDA) to give a stable octahedral electron rich compound of low ionization energy $[\text{Ru}(\text{CO})_2\text{Cl}_2\text{TMEDA}]$. The bidentate nature of the ligand (TMEDA) has also been reported in the X-ray structure of the complex $[\text{Mo}(\text{CO})_4\text{TMEDA}]$ by (Bakar *et al.*, 1993).

In the crystal structure of title compound, ruthenium atom is almost octahedrally coordinated to the two nitrogen-donors atoms of tetramethyl ethylene-1,2-diamine (TMEDA), two chloro and two carbonyl groups. A five membered non-planer ring formed through Ru1/N1/C5/C6/N2 as both of the carbon atoms are sp^3 hybridized. The root mean square deviation for the ring measure 0.2149 Å with the maximum deviation from C5 and C6 measures -0.2999 (19) Å and 0.3144 (19) Å respectively.

S2. Experimental

In a 100-ml round bottom flask fitted with nitrogen gas inlet, water condenser and magnetic stirrer was added 0.2 g of $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ and 0.5 ml of tetramethylethylenediamine (TMEDA) in 15 ml MeOH. The reaction mixture was heated at about 70 °C for 1 h. The yellow green solution was reduced in volume and passed through a small alumina column (15 g. Al_2O_3). The yellow band was eluted with MeOH. The solvent was reduced in volume and on cooling it gave yellow blocks of (I). Yield 70%.

S3. Refinement

All the C—H H-atoms were positioned *via* fourier map with C—H = 0.91 (3)—1.11 (3) Å with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$ for aromatic C atoms.

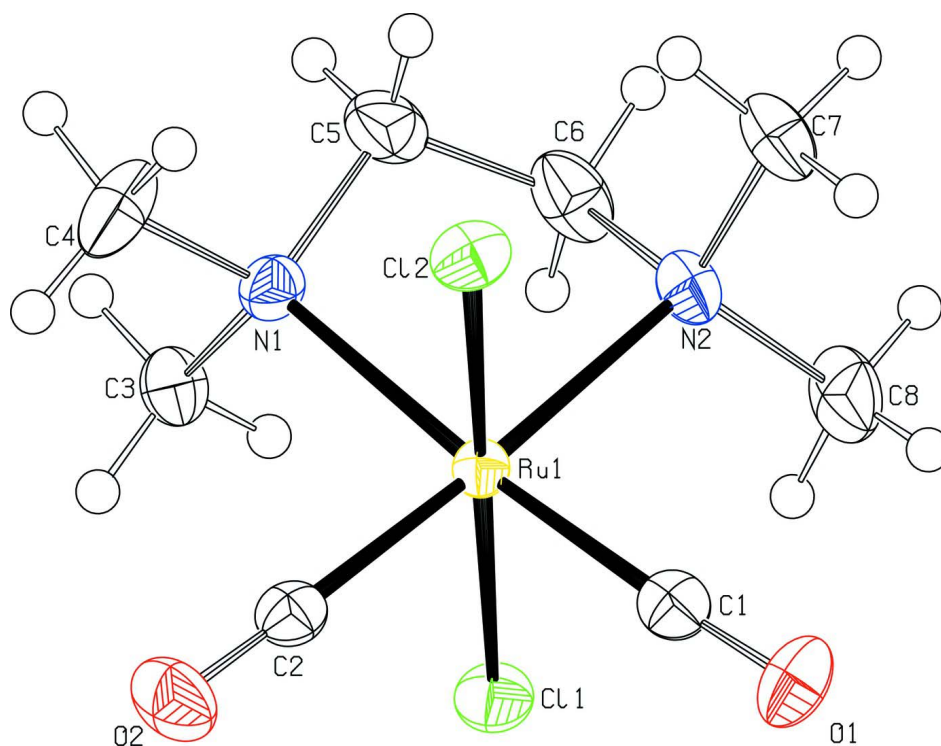
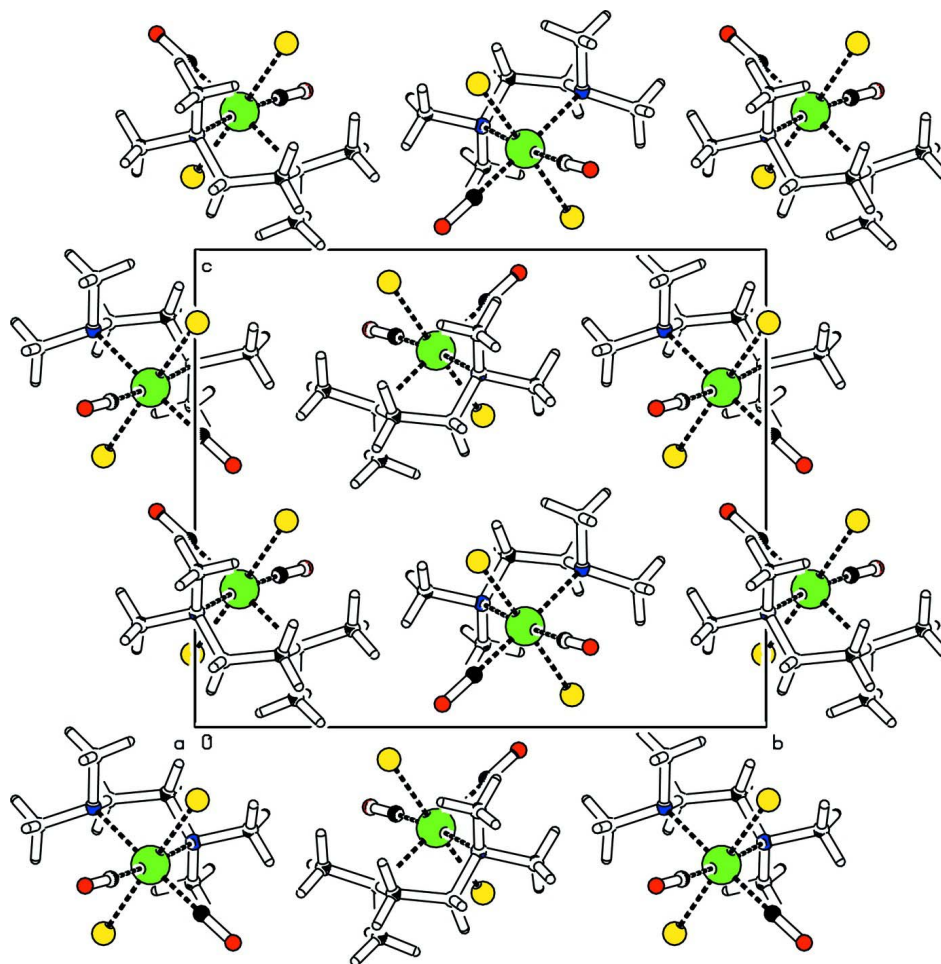


Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

Unit cell packing for (I).

Dicarbonyldichlorido(*N,N,N',N'*-tetramethylethylenediamine)ruthenium(II)*Crystal data*[RuCl₂(C₆H₁₆N₂)(CO)₂] $M_r = 344.20$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 7.463$ (6) Å $b = 14.579$ (6) Å $c = 12.718$ (12) Å $\beta = 106.37$ (8)° $V = 1327.7$ (17) Å³ $Z = 4$ $F(000) = 688$ $D_x = 1.722$ Mg m⁻³

Melting point: 493 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

 $\theta = 10.6$ – 14.0 ° $\mu = 1.57$ mm⁻¹ $T = 160$ K

Block, yellow

 $0.38 \times 0.38 \times 0.25$ mm*Data collection*Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Nonprofiled $\omega/2\theta$ scansAbsorption correction: ψ scan
(North *et al.*, 1968) $T_{\min} = 0.591$, $T_{\max} = 0.69$

3153 measured reflections

2877 independent reflections

2644 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.2^\circ$
 $h = -9 \rightarrow 9$

$k = 0 \rightarrow 18$
 $l = 0 \rightarrow 16$
 2 standard reflections every 100 reflections
 intensity decay: 5%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.061$
 $S = 1.07$
 2877 reflections
 184 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0385P)^2 + 0.6002P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.06 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.48 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.0497 (3)	0.14822 (15)	0.31835 (17)	0.0244 (4)
C2	0.1219 (3)	-0.01145 (15)	0.39077 (17)	0.0237 (4)
C3	0.5479 (3)	0.00447 (17)	0.3248 (2)	0.0269 (4)
H3A	0.634 (4)	-0.025 (2)	0.292 (2)	0.032*
H3B	0.542 (4)	-0.025 (2)	0.386 (2)	0.032*
H3C	0.587 (4)	0.065 (2)	0.342 (2)	0.032*
C4	0.3204 (4)	-0.09661 (19)	0.2146 (3)	0.0397 (6)
H4A	0.197 (5)	-0.105 (2)	0.163 (3)	0.048*
H4B	0.331 (4)	-0.131 (2)	0.294 (3)	0.048*
H4C	0.417 (5)	-0.121 (2)	0.187 (3)	0.048*
C5	0.3756 (4)	0.0491 (2)	0.1397 (2)	0.0376 (6)
H5A	0.269 (5)	0.028 (2)	0.078 (3)	0.045*
H5B	0.496 (5)	0.032 (2)	0.127 (3)	0.045*
C6	0.3660 (4)	0.1513 (2)	0.1516 (2)	0.0362 (6)
H6A	0.459 (5)	0.170 (2)	0.218 (3)	0.043*
H6B	0.373 (4)	0.180 (2)	0.087 (3)	0.043*
C7	0.0379 (4)	0.17649 (19)	0.0602 (2)	0.0352 (5)
H7A	-0.077 (5)	0.197 (2)	0.078 (2)	0.042*
H7B	0.026 (4)	0.115 (2)	0.023 (3)	0.042*
H7C	0.077 (4)	0.219 (2)	0.010 (3)	0.042*

C8	0.1967 (4)	0.27503 (17)	0.2056 (2)	0.0385 (6)
H8A	0.229 (5)	0.311 (2)	0.152 (3)	0.046*
H8B	0.285 (5)	0.285 (2)	0.277 (3)	0.046*
H8C	0.075 (5)	0.294 (2)	0.214 (2)	0.046*
Cl1	0.36315 (7)	0.16019 (4)	0.43227 (4)	0.02799 (12)
Cl2	-0.09471 (7)	-0.00421 (4)	0.15266 (4)	0.02621 (12)
O1	-0.1658 (2)	0.19233 (12)	0.33235 (15)	0.0343 (4)
O2	0.1117 (3)	-0.06675 (12)	0.45157 (15)	0.0377 (4)
Ru1	0.14044 (2)	0.078019 (10)	0.288724 (12)	0.01683 (7)
N1	0.3635 (2)	0.00254 (13)	0.24068 (15)	0.0233 (4)
N2	0.1815 (3)	0.17845 (13)	0.16732 (15)	0.0246 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0238 (10)	0.0231 (10)	0.0239 (10)	-0.0067 (9)	0.0027 (8)	-0.0024 (8)
C2	0.0224 (10)	0.0243 (10)	0.0224 (9)	-0.0033 (8)	0.0034 (8)	-0.0050 (8)
C3	0.0185 (10)	0.0303 (12)	0.0289 (11)	0.0033 (9)	0.0018 (8)	0.0029 (9)
C4	0.0287 (12)	0.0310 (13)	0.0561 (18)	0.0060 (10)	0.0064 (12)	-0.0169 (12)
C5	0.0329 (13)	0.0558 (16)	0.0263 (12)	0.0170 (12)	0.0120 (10)	0.0091 (11)
C6	0.0270 (12)	0.0467 (15)	0.0368 (13)	0.0019 (11)	0.0119 (10)	0.0174 (12)
C7	0.0338 (13)	0.0380 (14)	0.0281 (12)	0.0010 (11)	-0.0007 (10)	0.0110 (10)
C8	0.0492 (16)	0.0200 (11)	0.0423 (14)	-0.0074 (11)	0.0065 (12)	0.0060 (10)
Cl1	0.0275 (3)	0.0288 (3)	0.0241 (2)	-0.0044 (2)	0.0016 (2)	-0.0063 (2)
Cl2	0.0198 (2)	0.0304 (3)	0.0266 (2)	-0.00546 (19)	0.00355 (19)	-0.0077 (2)
O1	0.0267 (8)	0.0290 (9)	0.0485 (10)	0.0042 (7)	0.0129 (7)	-0.0069 (7)
O2	0.0473 (11)	0.0342 (9)	0.0326 (9)	-0.0072 (8)	0.0129 (8)	0.0088 (8)
Ru1	0.01583 (10)	0.01612 (10)	0.01779 (10)	-0.00122 (5)	0.00351 (7)	-0.00079 (5)
N1	0.0207 (8)	0.0256 (9)	0.0229 (8)	0.0035 (7)	0.0049 (7)	-0.0021 (7)
N2	0.0242 (9)	0.0230 (9)	0.0244 (9)	-0.0019 (7)	0.0035 (7)	0.0046 (7)

Geometric parameters (Å, °)

Ru1—C1	1.872 (3)	C5—N1	1.477 (3)
Ru1—C2	1.872 (2)	C5—C6	1.502 (4)
Ru1—N2	2.211 (2)	C5—H5A	1.00 (3)
Ru1—N1	2.220 (2)	C5—H5B	0.99 (3)
Ru1—Cl1	2.413 (2)	C6—N2	1.500 (3)
Ru1—Cl2	2.408 (2)	C6—H6A	0.97 (3)
C1—O1	1.133 (3)	C6—H6B	0.94 (3)
C2—O2	1.134 (3)	C7—N2	1.477 (3)
C3—N1	1.486 (3)	C7—H7A	1.00 (3)
C3—H3A	0.96 (3)	C7—H7B	1.00 (3)
C3—H3B	0.91 (3)	C7—H7C	0.99 (3)
C3—H3C	0.93 (3)	C8—N2	1.484 (3)
C4—N1	1.498 (3)	C8—H8A	0.94 (3)
C4—H4A	0.98 (4)	C8—H8B	0.97 (3)
C4—H4B	1.11 (3)	C8—H8C	0.98 (3)

C4—H4C	0.95 (4)		
O1—C1—Ru1	177.3 (2)	N2—C8—H8B	114.7 (19)
O2—C2—Ru1	178.8 (2)	H8A—C8—H8B	110 (3)
N1—C3—H3A	106.0 (16)	N2—C8—H8C	108.2 (19)
N1—C3—H3B	110.6 (18)	H8A—C8—H8C	111 (3)
H3A—C3—H3B	111 (2)	H8B—C8—H8C	105 (3)
N1—C3—H3C	110.5 (19)	C1—Ru1—C2	91.90 (11)
H3A—C3—H3C	109 (3)	C1—Ru1—N2	92.33 (10)
H3B—C3—H3C	110 (3)	C2—Ru1—N2	175.71 (8)
N1—C4—H4A	111.8 (19)	C1—Ru1—N1	174.94 (8)
N1—C4—H4B	106.3 (17)	C2—Ru1—N1	93.04 (10)
H4A—C4—H4B	111 (3)	N2—Ru1—N1	82.75 (9)
N1—C4—H4C	108 (2)	C1—Ru1—Cl2	88.66 (9)
H4A—C4—H4C	112 (3)	C2—Ru1—Cl2	88.29 (9)
H4B—C4—H4C	107 (3)	N2—Ru1—Cl2	92.53 (8)
N1—C5—C6	110.6 (2)	N1—Ru1—Cl2	90.38 (8)
N1—C5—H5A	108.2 (18)	C1—Ru1—Cl1	88.62 (9)
C6—C5—H5A	109.8 (18)	C2—Ru1—Cl1	89.49 (9)
N1—C5—H5B	108.0 (18)	N2—Ru1—Cl1	89.89 (8)
C6—C5—H5B	110.0 (19)	N1—Ru1—Cl1	92.53 (8)
H5A—C5—H5B	110 (3)	Cl2—Ru1—Cl1	176.424 (19)
N2—C6—C5	110.3 (2)	C5—N1—C3	110.3 (2)
N2—C6—H6A	105.4 (18)	C5—N1—C4	108.2 (2)
C5—C6—H6A	108.6 (18)	C3—N1—C4	105.97 (18)
N2—C6—H6B	106.0 (19)	C5—N1—Ru1	104.24 (15)
C5—C6—H6B	110.2 (19)	C3—N1—Ru1	113.96 (15)
H6A—C6—H6B	116 (3)	C4—N1—Ru1	114.10 (15)
N2—C7—H7A	103.4 (18)	C7—N2—C8	106.8 (2)
N2—C7—H7B	113.4 (18)	C7—N2—C6	109.1 (2)
H7A—C7—H7B	114 (3)	C8—N2—C6	107.8 (2)
N2—C7—H7C	108.3 (19)	C7—N2—Ru1	115.10 (15)
H7A—C7—H7C	113 (2)	C8—N2—Ru1	114.35 (16)
H7B—C7—H7C	105 (2)	C6—N2—Ru1	103.38 (14)
N2—C8—H8A	107.7 (19)		
N1—C5—C6—N2	-62.9 (3)	C5—C6—N2—C7	-78.1 (2)
C6—C5—N1—C3	-80.0 (2)	C5—C6—N2—C8	166.3 (2)
C6—C5—N1—C4	164.5 (2)	C5—C6—N2—Ru1	44.8 (2)
C6—C5—N1—Ru1	42.7 (2)	C1—Ru1—N2—C7	-76.26 (19)
C1—Ru1—N1—C5	-0.1 (9)	C2—Ru1—N2—C7	113.4 (10)
C2—Ru1—N1—C5	167.17 (16)	N1—Ru1—N2—C7	102.56 (18)
N2—Ru1—N1—C5	-13.63 (16)	Cl2—Ru1—N2—C7	12.49 (17)
Cl2—Ru1—N1—C5	78.87 (17)	Cl1—Ru1—N2—C7	-164.88 (17)
Cl1—Ru1—N1—C5	-103.20 (17)	C1—Ru1—N2—C8	48.00 (19)
C1—Ru1—N1—C3	120.1 (9)	C2—Ru1—N2—C8	-122.3 (10)
C2—Ru1—N1—C3	-72.57 (17)	N1—Ru1—N2—C8	-133.18 (18)
N2—Ru1—N1—C3	106.62 (16)	Cl2—Ru1—N2—C8	136.76 (17)

Cl2—Ru1—N1—C3	-160.87 (15)	Cl1—Ru1—N2—C8	-40.61 (17)
Cl1—Ru1—N1—C3	17.05 (15)	C1—Ru1—N2—C6	164.90 (16)
C1—Ru1—N1—C4	-117.9 (9)	C2—Ru1—N2—C6	-5.4 (11)
C2—Ru1—N1—C4	49.35 (19)	N1—Ru1—N2—C6	-16.28 (15)
N2—Ru1—N1—C4	-131.46 (18)	Cl2—Ru1—N2—C6	-106.34 (16)
Cl2—Ru1—N1—C4	-38.96 (17)	Cl1—Ru1—N2—C6	76.29 (16)
Cl1—Ru1—N1—C4	138.97 (17)		
