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Chlorido(6,7-dimethyl-2,3-dipyridin-2-ylquinoxaline-κ^2N,N')(η^6-1,2,4,5-tetramethylbenzene)ruthenium(II) hexafluorophosphate acetonitrile solvate

Bruno Therrien and Georg Süss-Fink

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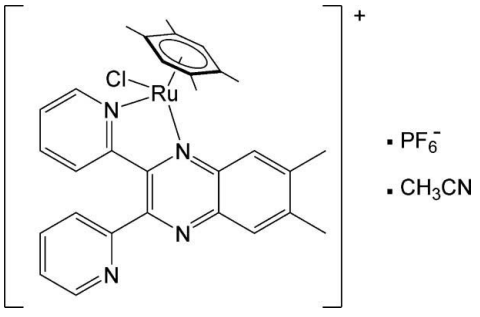
In the title salt, $[RuCl(C_{10}H_{14})(C_{20}H_{16}N_4)]PF_6\cdot CH_3CN$, the coordination of one pyridyl ring and of one N atom of the quinoxaline unit to ruthenium imposes considerable distortion on the 6,7-dimethyl-2,3-dipyridin-2-ylquinoxaline ligand. Indeed, the pyridyl ring and the plane of the quinoxaline unit become almost coplanar.

Related literature

The non-coordinated $dpqMe_2$ ligand crystallizes in the centrosymmetric space group $P2_1/a$ (Wozniak *et al.*, 1993). Other arene–ruthenium and osmium complexes with chelating quinoxaline pyridyl derivatives have been synthesized and characterized by X-ray structure analysis (Baumann *et al.*, 1998; Scott *et al.*, 1999; Lalrempuia & Kollipara, 2003; Therrien *et al.*, 2007).

For related literature, see: Berg *et al.* (2002); Canivet *et al.* (2005); Singh *et al.* (2002).



Experimental

Crystal data

$[RuCl(C_{10}H_{14})(C_{20}H_{16}N_4)]PF_6\cdot C_2H_3N$
M_r = 769.12
Monoclinic, *Cc*
a = 13.1725 (11) Å
b = 25.6651 (17) Å
c = 10.1222 (9) Å
β = 104.140 (10)°
V = 3318.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 173 (2) K
0.34 × 0.22 × 0.19 mm

Data collection

Stoe IPDS diffractometer
Absorption correction: none
13015 measured reflections
6142 independent reflections
5710 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.064
S = 1.01
6142 reflections
422 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 2912 Friedel pairs
Flack parameter: −0.01 (2)

Data collection: *EXPOSE* (Stoe, 2000); cell refinement: *CELL* (Stoe, 2000); data reduction: *INTEGRATE* (Stoe, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2001); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2001); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Swiss National Science Foundation.

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supplementary materials

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Comment

The title complex (I) shows a typical piano-stool geometry with the metal centre coordinated by the arene ligand, a terminal chloride and the chelating 6,7-dimethyl-2,3-di(pyridin-2-yl)quinoxaline ($dpqMe_2$) ligand. In the mononuclear complex (I), the metal centre is stereogenic. However, since none of the ligand contains a chiral information, (I) is obtained as a racemic mixture.

The Ru—N bond distances 2.060 (2) and 2.077 (2) Å in (I) are comparable to those in [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(2-pyridyl)pyrazine)][BF_4] (Singh *et al.*, 2002) and [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(α-pyridyl)quinoxaline)][PF_6] (Lalrempuia & Kollipara, 2003). Accordingly, there is no significant difference in the Ru—Cl bond length in (I) [2.3732 (9) Å] and reported values (Lalrempuia & Kollipara, 2003)(Scott *et al.*, 1999)(Canivet *et al.*, 2005). The N(1)—Ru(1)—N(2) bond angle in complex (I) [76.04 (9)°] is similar to those of complexes [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(2-pyridyl)pyrazine)]$^+$ [N(1)—Ru(1)—N(2) = 76.5 (2)°] (Singh *et al.*, 2002) and [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(α-pyridyl)quinoxaline)]$^+$ [N(1)—Ru(1)—N(2) = 76.2 (2)°] (Lalrempuia & Kollipara, 2003). An ORTEP drawing with the atom labelling scheme for (I) is shown in Figure 1.

Upon formation of the mononuclear complex (I), the bond length between the connecting carbon atoms [C(5)—C(6) = 1.472 (4) Å] of the coordinated pyridyl and quinoxaline moieties of the $dpqMe_2$ ligand is slightly reduced as compared to the corresponding connecting C—C atoms [C(7)—C(8) = 1.496 (4) Å] of the non-coordinated pyridyl quinoxaline moities. Similarly, the C—C distances [1.493 Å] in the free $dpqMe_2$ are longer (Wozniak *et al.*, 1993). These bond length changes are in agreement with a back-donation from the metallic fragments to the $dpqMe_2$ system, thus increasing the inter-ring bond order (Baumann *et al.*, 1998) (Berg *et al.*, 2002).

The major distortion imposed on the $dpqMe_2$ structure upon coordination is encountered by the pyridyl groups. In the free ligand the two equivalent pyridyl groups are twisted by 39.6° relative to the plane of the quinoxaline unit (Wozniak *et al.*, 1993). However, in (I) the twist of the coordinated pyridyl unit is 22.7 (1)°, while the non-coordinated pyridyl group is twisted by 54.2 (1)° relative to the plane of the quinoxaline unit.

Experimental

The dinuclear complex [(η^6-1,2,4,5-$C_6H_2Me_4$)Ru(μ-Cl)Cl]$_2$ (70 mg, 0.11 mmol) is dissolved in methanol (50 ml). The resulting solution is added dropwise to a two-necked flask equipped with a reflux condenser and containing a methanol solution (50 ml) of $dpqMe_2$ (71 mg, 0.23 mmol) and KPF_6 (42 mg, 0.11 mmol). The mixture is heated to 50°C and stirred for 24 h. After cooling to room temperature, the volume is reduced and the product is precipitated by addition of diethylether. The

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orange solid is filtered, washed with n-pentane and dried under vacuo to give [(η^6-1,2,4,5-$C_6H_2Me_4$)RuCl(dpqMe$_2$)][PF_6] (130 mg, 0.12 mmol, yield 78.1%).

Crystals of (I) were obtained by the slow diffusion of diethylether into an acetonitrile solution of (I).

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Figures

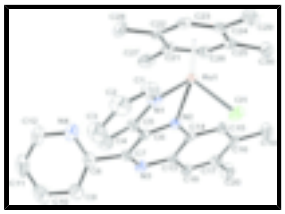

Fig. 1. Molecular structure of (I) at 50% probability level with hydrogen atoms, acetonitrile molecule and hexafluorophosphate anion being omitted for clarity.

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H-atom parameters constrained
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$\Delta\rho_{min} = -0.27$ e $Å^{-3}$
Extinction correction: none
Absolute structure: Flack (1983), 2912 Friedel pairs
Flack parameter: −0.01 (2)

Special details

Experimental. A crystal was mounted at 173 K on a Stoe Image Plate Diffraction System (Stoe & Cie, 2000) using MoKα graphite monochromated radiation. Image plate distance 70 mm, φ oscillation scans 0 - 200°, step Δφ = 1.0°, 3 minutes per frame.

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6737 (3)	0.39051 (13)	0.2351 (3)	0.0307 (7)
H1	0.7045	0.3767	0.1694	0.037*
C2	0.6820 (3)	0.44337 (14)	0.2618 (4)	0.0397 (8)
H2	0.7164	0.4650	0.2131	0.048*
C3	0.6390 (3)	0.46340 (14)	0.3613 (4)	0.0421 (9)
H3	0.6439	0.4989	0.3806	0.050*
C4	0.5882 (3)	0.43078 (12)	0.4326 (3)	0.0317 (7)
H4	0.5601	0.4439	0.5017	0.038*
C5	0.5796 (2)	0.37805 (11)	0.3999 (3)	0.0206 (6)
C6	0.5389 (2)	0.33765 (11)	0.4764 (3)	0.0177 (6)
C7	0.4808 (2)	0.34576 (11)	0.5771 (3)	0.0198 (6)

supplementary materials

C8	0.4218 (3)	0.39516 (13)	0.5831 (4)	0.0286 (7)
C9	0.4299 (3)	0.42035 (18)	0.7062 (4)	0.0475 (10)
H9	0.4766	0.4091	0.7854	0.057*
C10	0.3656 (4)	0.4631 (2)	0.7064 (5)	0.0673 (14)
H10	0.3693	0.4815	0.7866	0.081*
C11	0.2965 (4)	0.47820 (17)	0.5880 (5)	0.0583 (12)
H11	0.2524	0.5066	0.5863	0.070*
C12	0.2943 (3)	0.45008 (14)	0.4719 (4)	0.0416 (9)
H12	0.2473	0.4602	0.3916	0.050*
C13	0.5065 (2)	0.26073 (12)	0.6437 (3)	0.0197 (6)
C14	0.5539 (2)	0.24969 (11)	0.5364 (3)	0.0181 (6)
C15	0.5899 (2)	0.19883 (12)	0.5216 (3)	0.0206 (6)
H15	0.6230	0.1918	0.4523	0.025*
C16	0.5769 (2)	0.15968 (12)	0.6077 (3)	0.0231 (6)
C17	0.5245 (2)	0.17017 (12)	0.7126 (3)	0.0246 (6)
C18	0.4924 (2)	0.22013 (12)	0.7308 (3)	0.0223 (6)
H18	0.4608	0.2271	0.8016	0.027*
C19	0.6161 (3)	0.10587 (14)	0.5893 (4)	0.0386 (8)
H19A	0.6478	0.1057	0.5134	0.058*
H19B	0.5586	0.0818	0.5725	0.058*
H19C	0.6671	0.0957	0.6703	0.058*
C20	0.5024 (3)	0.12652 (14)	0.8013 (4)	0.0396 (9)
H20A	0.4624	0.1397	0.8616	0.059*
H20B	0.5673	0.1124	0.8537	0.059*
H20C	0.4634	0.0997	0.7449	0.059*
C21	0.4337 (3)	0.25966 (16)	0.1676 (4)	0.0231 (8)
C22	0.4661 (2)	0.29775 (13)	0.0880 (3)	0.0238 (6)
C23	0.5573 (3)	0.28838 (11)	0.0386 (3)	0.0227 (6)
H23	0.5774	0.3135	−0.0161	0.027*
C24	0.6182 (2)	0.24295 (13)	0.0690 (3)	0.0247 (6)
C25	0.5849 (3)	0.20366 (12)	0.1513 (3)	0.0248 (6)
C26	0.4947 (2)	0.21243 (12)	0.1960 (3)	0.0245 (7)
H26	0.4724	0.1866	0.2467	0.029*
C27	0.3433 (3)	0.26601 (16)	0.2315 (4)	0.0359 (8)
H27A	0.3435	0.3007	0.2670	0.054*
H27B	0.3495	0.2413	0.3043	0.054*
H27C	0.2791	0.2601	0.1643	0.054*
C28	0.4090 (3)	0.34842 (14)	0.0542 (4)	0.0374 (8)
H28A	0.3493	0.3434	−0.0210	0.056*
H28B	0.4549	0.3738	0.0301	0.056*
H28C	0.3861	0.3605	0.1320	0.056*
C29	0.7168 (3)	0.23601 (16)	0.0209 (4)	0.0375 (8)
H29A	0.7070	0.2085	−0.0454	0.056*
H29B	0.7736	0.2272	0.0969	0.056*
H29C	0.7328	0.2679	−0.0195	0.056*
C30	0.6503 (3)	0.15529 (13)	0.1905 (4)	0.0375 (8)
H30A	0.6237	0.1356	0.2554	0.056*
H30B	0.7216	0.1650	0.2303	0.056*
H30C	0.6471	0.1344	0.1109	0.056*

supplementary materials

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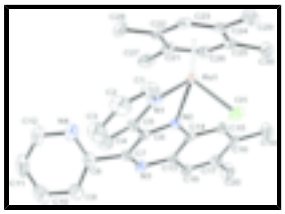

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Comment

The title complex (I) shows a typical piano-stool geometry with the metal centre coordinated by the arene ligand, a terminal chloride and the chelating 6,7-dimethyl-2,3-di(pyridin-2-yl)quinoxaline ($dpqMe_2$) ligand. In the mononuclear complex (I), the metal centre is stereogenic. However, since none of the ligand contains a chiral information, (I) is obtained as a racemic mixture.

The Ru—N bond distances 2.060 (2) and 2.077 (2) Å in (I) are comparable to those in [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(2-pyridyl)pyrazine)][BF_4] (Singh *et al.*, 2002) and [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(α-pyridyl)quinoxaline)][PF_6] (Lalrempuia & Kollipara, 2003). Accordingly, there is no significant difference in the Ru—Cl bond length in (I) [2.3732 (9) Å] and reported values (Lalrempuia & Kollipara, 2003)(Scott *et al.*, 1999)(Canivet *et al.*, 2005). The N(1)—Ru(1)—N(2) bond angle in complex (I) [76.04 (9)°] is similar to those of complexes [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(2-pyridyl)pyrazine)]$^+$ [N(1)—Ru(1)—N(2) = 76.5 (2)°] (Singh *et al.*, 2002) and [(η^6-*p*-Pr^{i}C$_6$H$_4$Me)RuCl(η^2-2,3-bis(α-pyridyl)quinoxaline)]$^+$ [N(1)—Ru(1)—N(2) = 76.2 (2)°] (Lalrempuia & Kollipara, 2003). An ORTEP drawing with the atom labelling scheme for (I) is shown in Figure 1.

Upon formation of the mononuclear complex (I), the bond length between the connecting carbon atoms [C(5)—C(6) = 1.472 (4) Å] of the coordinated pyridyl and quinoxaline moieties of the $dpqMe_2$ ligand is slightly reduced as compared to the corresponding connecting C—C atoms [C(7)—C(8) = 1.496 (4) Å] of the non-coordinated pyridyl quinoxaline moities. Similarly, the C—C distances [1.493 Å] in the free $dpqMe_2$ are longer (Wozniak *et al.*, 1993). These bond length changes are in agreement with a back-donation from the metallic fragments to the $dpqMe_2$ system, thus increasing the inter-ring bond order (Baumann *et al.*, 1998) (Berg *et al.*, 2002).

The major distortion imposed on the $dpqMe_2$ structure upon coordination is encountered by the pyridyl groups. In the free ligand the two equivalent pyridyl groups are twisted by 39.6° relative to the plane of the quinoxaline unit (Wozniak *et al.*, 1993). However, in (I) the twist of the coordinated pyridyl unit is 22.7 (1)°, while the non-coordinated pyridyl group is twisted by 54.2 (1)° relative to the plane of the quinoxaline unit.

Experimental

The dinuclear complex [(η^6-1,2,4,5-$C_6H_2Me_4$)Ru(μ-Cl)Cl]$_2$ (70 mg, 0.11 mmol) is dissolved in methanol (50 ml). The resulting solution is added dropwise to a two-necked flask equipped with a reflux condenser and containing a methanol solution (50 ml) of $dpqMe_2$ (71 mg, 0.23 mmol) and KPF_6 (42 mg, 0.11 mmol). The mixture is heated to 50°C and stirred for 24 h. After cooling to room temperature, the volume is reduced and the product is precipitated by addition of diethylether. The

supplementary materials

C10—C11—C12	118.0 (4)	C32—C31—H31A	109.5
C10—C11—H11	121.0	C32—C31—H31B	109.5
C12—C11—H11	121.0	H31A—C31—H31B	109.5
N4—C12—C11	123.7 (4)	C32—C31—H31C	109.5
N4—C12—H12	118.2	H31A—C31—H31C	109.5
C11—C12—H12	118.2	H31B—C31—H31C	109.5
N3—C13—C18	119.0 (3)	N5—C32—C31	178.3 (5)
N3—C13—C14	121.7 (3)	C1—N1—C5	119.4 (3)
C18—C13—C14	119.1 (3)	C1—N1—Ru1	124.3 (2)
N2—C14—C13	119.3 (3)	C5—N1—Ru1	116.24 (19)
N2—C14—C15	121.2 (3)	C6—N2—C14	118.3 (2)
C13—C14—C15	119.5 (3)	C6—N2—Ru1	114.41 (18)
C16—C15—C14	121.0 (3)	C14—N2—Ru1	126.31 (19)
C16—C15—H15	119.5	C7—N3—C13	118.0 (3)
C14—C15—H15	119.5	C12—N4—C8	117.7 (3)
C15—C16—C17	119.6 (3)	F2—P1—F5	93.3 (3)
C15—C16—C19	119.8 (3)	F2—P1—F3	92.0 (3)
C17—C16—C19	120.6 (3)	F5—P1—F3	174.4 (3)
C18—C17—C16	119.7 (3)	F2—P1—F4	95.3 (2)
C18—C17—C20	120.2 (3)	F5—P1—F4	90.7 (2)
C16—C17—C20	120.1 (3)	F3—P1—F4	90.6 (2)
C17—C18—C13	121.0 (3)	F2—P1—F6	88.1 (2)
C17—C18—H18	119.5	F5—P1—F6	89.2 (2)
C13—C18—H18	119.5	F3—P1—F6	89.1 (2)
C16—C19—H19A	109.5	F4—P1—F6	176.6 (2)
C16—C19—H19B	109.5	F2—P1—F1	177.0 (2)
H19A—C19—H19B	109.5	F5—P1—F1	88.7 (2)
C16—C19—H19C	109.5	F3—P1—F1	85.9 (2)
H19A—C19—H19C	109.5	F4—P1—F1	86.91 (17)
H19B—C19—H19C	109.5	F6—P1—F1	89.68 (19)
C17—C20—H20A	109.5	N1—Ru1—N2	76.04 (9)
C17—C20—H20B	109.5	N1—Ru1—C23	96.01 (10)
H20A—C20—H20B	109.5	N2—Ru1—C23	152.33 (11)
C17—C20—H20C	109.5	N1—Ru1—C22	90.94 (10)
H20A—C20—H20C	109.5	N2—Ru1—C22	114.50 (11)
H20B—C20—H20C	109.5	C23—Ru1—C22	38.26 (12)
C22—C21—C26	117.9 (3)	N1—Ru1—C21	114.07 (13)
C22—C21—C27	124.1 (4)	N2—Ru1—C21	91.16 (12)
C26—C21—C27	117.9 (3)	C23—Ru1—C21	67.59 (13)
C22—C21—Ru1	70.71 (18)	C22—Ru1—C21	37.21 (14)
C26—C21—Ru1	71.11 (18)	N1—Ru1—C26	151.92 (11)
C27—C21—Ru1	126.5 (2)	N2—Ru1—C26	96.24 (10)
C21—C22—C23	119.0 (3)	C23—Ru1—C26	78.23 (11)
C21—C22—C28	122.0 (3)	C22—Ru1—C26	67.40 (12)
C23—C22—C28	119.0 (3)	C21—Ru1—C26	38.24 (14)
C21—C22—Ru1	72.09 (17)	N1—Ru1—C24	123.48 (11)
C23—C22—Ru1	70.78 (16)	N2—Ru1—C24	160.48 (10)
C28—C22—Ru1	128.1 (2)	C23—Ru1—C24	37.16 (12)
C24—C23—C22	123.1 (3)	C22—Ru1—C24	68.81 (11)

supplementary materials

C24—C23—Ru1	73.31 (17)	C21—Ru1—C24	80.96 (13)
C22—C23—Ru1	70.96 (16)	C26—Ru1—C24	66.62 (12)
C24—C23—H23	118.4	N1—Ru1—C25	161.15 (11)
C22—C23—H23	118.4	N2—Ru1—C25	122.80 (11)
Ru1—C23—H23	130.1	C23—Ru1—C25	67.07 (11)
C23—C24—C25	118.0 (3)	C22—Ru1—C25	80.76 (11)
C23—C24—C29	121.2 (3)	C21—Ru1—C25	68.38 (13)
C25—C24—C29	120.8 (3)	C26—Ru1—C25	36.43 (12)
C23—C24—Ru1	69.53 (17)	C24—Ru1—C25	37.69 (13)
C25—C24—Ru1	71.49 (18)	N1—Ru1—Cl1	86.79 (7)
C29—C24—Ru1	128.8 (2)	N2—Ru1—Cl1	87.99 (7)
C26—C25—C24	118.6 (3)	C23—Ru1—Cl1	118.38 (9)
C26—C25—C30	121.8 (3)	C22—Ru1—Cl1	156.13 (9)
C24—C25—C30	119.6 (3)	C21—Ru1—Cl1	158.26 (10)
C26—C25—Ru1	70.55 (17)	C26—Ru1—Cl1	120.31 (9)
C24—C25—Ru1	70.82 (17)	C24—Ru1—Cl1	92.79 (8)
C30—C25—Ru1	128.6 (2)	C25—Ru1—Cl1	93.98 (8)
N1—C1—C2—C3	1.8 (6)	C14—N2—Ru1—C24	11.7 (4)
C1—C2—C3—C4	0.0 (6)	C6—N2—Ru1—C25	−155.77 (19)
C2—C3—C4—C5	−1.4 (5)	C14—N2—Ru1—C25	12.5 (3)
C3—C4—C5—N1	1.3 (5)	C6—N2—Ru1—Cl1	110.79 (19)
C3—C4—C5—C6	172.5 (3)	C14—N2—Ru1—Cl1	−80.9 (2)
N1—C5—C6—N2	10.1 (3)	C24—C23—Ru1—N1	141.19 (19)
C4—C5—C6—N2	−161.6 (3)	C22—C23—Ru1—N1	−83.85 (18)
N1—C5—C6—C7	−173.5 (2)	C24—C23—Ru1—N2	−147.6 (2)
C4—C5—C6—C7	14.7 (5)	C22—C23—Ru1—N2	−12.6 (3)
N2—C6—C7—N3	11.2 (4)	C24—C23—Ru1—C22	−135.0 (3)
C5—C6—C7—N3	−165.0 (3)	C24—C23—Ru1—C21	−105.2 (2)
N2—C6—C7—C8	−161.8 (3)	C22—C23—Ru1—C21	29.7 (2)
C5—C6—C7—C8	22.1 (4)	C24—C23—Ru1—C26	−66.65 (19)
N3—C7—C8—N4	−120.4 (3)	C22—C23—Ru1—C26	68.30 (19)
C6—C7—C8—N4	52.9 (4)	C22—C23—Ru1—C24	135.0 (3)
N3—C7—C8—C9	53.2 (4)	C24—C23—Ru1—C25	−30.19 (18)
C6—C7—C8—C9	−133.5 (4)	C22—C23—Ru1—C25	104.8 (2)
N4—C8—C9—C10	−1.0 (7)	C24—C23—Ru1—Cl1	51.60 (19)
C7—C8—C9—C10	−174.0 (4)	C22—C23—Ru1—Cl1	−173.45 (15)
C8—C9—C10—C11	1.0 (8)	C21—C22—Ru1—N1	−130.8 (2)
C9—C10—C11—C12	−0.4 (8)	C23—C22—Ru1—N1	98.54 (18)
C10—C11—C12—N4	−0.2 (7)	C28—C22—Ru1—N1	−13.7 (3)
N3—C13—C14—N2	3.7 (4)	C21—C22—Ru1—N2	−55.7 (2)
C18—C13—C14—N2	178.9 (2)	C23—C22—Ru1—N2	173.61 (16)
N3—C13—C14—C15	−177.8 (2)	C28—C22—Ru1—N2	61.4 (3)
C18—C13—C14—C15	−2.6 (4)	C21—C22—Ru1—C23	130.7 (3)
N2—C14—C15—C16	−179.7 (3)	C28—C22—Ru1—C23	−112.2 (4)
C13—C14—C15—C16	1.9 (4)	C23—C22—Ru1—C21	−130.7 (3)
C14—C15—C16—C17	1.1 (4)	C28—C22—Ru1—C21	117.1 (4)
C14—C15—C16—C19	180.0 (3)	C21—C22—Ru1—C26	30.8 (2)
C15—C16—C17—C18	−3.4 (4)	C23—C22—Ru1—C26	−99.85 (19)
C19—C16—C17—C18	177.8 (3)	C28—C22—Ru1—C26	147.9 (3)

supplementary materials

C15—C16—C17—C20	175.3 (3)	C21—C22—Ru1—C24	103.4 (2)
C19—C16—C17—C20	−3.5 (4)	C23—C22—Ru1—C24	−27.29 (17)
C16—C17—C18—C13	2.6 (4)	C28—C22—Ru1—C24	−139.5 (3)
C20—C17—C18—C13	−176.1 (3)	C21—C22—Ru1—C25	66.2 (2)
N3—C13—C18—C17	175.7 (3)	C23—C22—Ru1—C25	−64.46 (18)
C14—C13—C18—C17	0.3 (4)	C28—C22—Ru1—C25	−176.7 (3)
C26—C21—C22—C23	0.0 (4)	C21—C22—Ru1—Cl1	145.1 (2)
C27—C21—C22—C23	176.5 (3)	C23—C22—Ru1—Cl1	14.4 (3)
Ru1—C21—C22—C23	54.9 (2)	C28—C22—Ru1—Cl1	−97.9 (3)
C26—C21—C22—C28	−179.2 (3)	C22—C21—Ru1—N1	56.0 (2)
C27—C21—C22—C28	−2.7 (5)	C26—C21—Ru1—N1	−173.84 (17)
Ru1—C21—C22—C28	−124.2 (3)	C27—C21—Ru1—N1	−62.6 (4)
C26—C21—C22—Ru1	−55.0 (2)	C22—C21—Ru1—N2	131.2 (2)
C27—C21—C22—Ru1	121.5 (3)	C26—C21—Ru1—N2	−98.63 (19)
C21—C22—C23—C24	−1.6 (4)	C27—C21—Ru1—N2	12.6 (3)
C28—C22—C23—C24	177.7 (3)	C22—C21—Ru1—C23	−30.5 (2)
Ru1—C22—C23—C24	54.0 (3)	C26—C21—Ru1—C23	99.6 (2)
C21—C22—C23—Ru1	−55.6 (2)	C27—C21—Ru1—C23	−149.2 (4)
C28—C22—C23—Ru1	123.6 (3)	C26—C21—Ru1—C22	130.1 (3)
C22—C23—C24—C25	1.4 (4)	C27—C21—Ru1—C22	−118.6 (4)
Ru1—C23—C24—C25	54.4 (2)	C22—C21—Ru1—C26	−130.1 (3)
C22—C23—C24—C29	−176.7 (3)	C27—C21—Ru1—C26	111.2 (4)
Ru1—C23—C24—C29	−123.7 (3)	C22—C21—Ru1—C24	−66.7 (2)
C22—C23—C24—Ru1	−53.0 (2)	C26—C21—Ru1—C24	63.4 (2)
C23—C24—C25—C26	0.3 (4)	C27—C21—Ru1—C24	174.7 (4)
C29—C24—C25—C26	178.5 (3)	C22—C21—Ru1—C25	−103.7 (2)
Ru1—C24—C25—C26	53.7 (2)	C26—C21—Ru1—C25	26.45 (18)
C23—C24—C25—C30	−177.7 (3)	C27—C21—Ru1—C25	137.7 (4)
C29—C24—C25—C30	0.5 (4)	C22—C21—Ru1—Cl1	−141.3 (2)
Ru1—C24—C25—C30	−124.2 (3)	C26—C21—Ru1—Cl1	−11.1 (4)
C23—C24—C25—Ru1	−53.5 (2)	C27—C21—Ru1—Cl1	100.1 (4)
C29—C24—C25—Ru1	124.7 (3)	C25—C26—Ru1—N1	147.8 (2)
C24—C25—C26—C21	−1.9 (4)	C21—C26—Ru1—N1	12.0 (3)
C30—C25—C26—C21	176.0 (3)	C25—C26—Ru1—N2	−140.30 (17)
Ru1—C25—C26—C21	52.0 (3)	C21—C26—Ru1—N2	83.9 (2)
C24—C25—C26—Ru1	−53.9 (2)	C25—C26—Ru1—C23	67.18 (18)
C30—C25—C26—Ru1	124.1 (3)	C21—C26—Ru1—C23	−68.6 (2)
C22—C21—C26—C25	1.8 (5)	C25—C26—Ru1—C22	105.7 (2)
C27—C21—C26—C25	−175.0 (3)	C21—C26—Ru1—C22	−30.1 (2)
Ru1—C21—C26—C25	−53.0 (3)	C25—C26—Ru1—C21	135.8 (3)
C22—C21—C26—Ru1	54.8 (3)	C25—C26—Ru1—C24	30.00 (18)
C27—C21—C26—Ru1	−122.0 (3)	C21—C26—Ru1—C24	−105.8 (2)
C2—C1—N1—C5	−1.9 (5)	C21—C26—Ru1—C25	−135.8 (3)
C2—C1—N1—Ru1	176.0 (3)	C25—C26—Ru1—Cl1	−48.97 (19)
C4—C5—N1—C1	0.3 (4)	C21—C26—Ru1—Cl1	175.25 (19)
C6—C5—N1—C1	−171.9 (2)	C23—C24—Ru1—N1	−48.4 (2)
C4—C5—N1—Ru1	−177.7 (2)	C25—C24—Ru1—N1	−179.10 (16)
C6—C5—N1—Ru1	10.1 (3)	C29—C24—Ru1—N1	65.9 (3)
C7—C6—N2—C14	−11.3 (4)	C23—C24—Ru1—N2	131.8 (3)

supplementary materials

C5—C6—N2—C14	165.3 (2)	C25—C24—Ru1—N2	1.0 (4)
C7—C6—N2—Ru1	157.96 (19)	C29—C24—Ru1—N2	−114.0 (4)
C5—C6—N2—Ru1	−25.4 (3)	C25—C24—Ru1—C23	−130.7 (3)
C13—C14—N2—C6	4.3 (4)	C29—C24—Ru1—C23	114.2 (4)
C15—C14—N2—C6	−174.1 (2)	C23—C24—Ru1—C22	28.03 (19)
C13—C14—N2—Ru1	−163.54 (19)	C25—C24—Ru1—C22	−102.7 (2)
C15—C14—N2—Ru1	18.0 (4)	C29—C24—Ru1—C22	142.2 (3)
C6—C7—N3—C13	−3.0 (4)	C23—C24—Ru1—C21	64.6 (2)
C8—C7—N3—C13	170.3 (2)	C25—C24—Ru1—C21	−66.16 (19)
C18—C13—N3—C7	−179.5 (2)	C29—C24—Ru1—C21	178.8 (3)
C14—C13—N3—C7	−4.2 (4)	C23—C24—Ru1—C26	101.7 (2)
C11—C12—N4—C8	0.3 (6)	C25—C24—Ru1—C26	−29.06 (17)
C9—C8—N4—C12	0.3 (5)	C29—C24—Ru1—C26	−144.1 (3)
C7—C8—N4—C12	173.7 (3)	C23—C24—Ru1—C25	130.7 (3)
C1—N1—Ru1—N2	164.2 (3)	C29—C24—Ru1—C25	−115.0 (4)
C5—N1—Ru1—N2	−17.92 (19)	C23—C24—Ru1—Cl1	−136.35 (18)
C1—N1—Ru1—C23	−42.8 (3)	C25—C24—Ru1—Cl1	92.90 (17)
C5—N1—Ru1—C23	135.1 (2)	C29—C24—Ru1—Cl1	−22.1 (3)
C1—N1—Ru1—C22	−80.8 (2)	C26—C25—Ru1—N1	−129.0 (3)
C5—N1—Ru1—C22	97.1 (2)	C24—C25—Ru1—N1	2.3 (4)
C1—N1—Ru1—C21	−110.9 (3)	C30—C25—Ru1—N1	115.3 (4)
C5—N1—Ru1—C21	67.0 (2)	C26—C25—Ru1—N2	49.1 (2)
C1—N1—Ru1—C26	−119.0 (3)	C24—C25—Ru1—N2	−179.59 (16)
C5—N1—Ru1—C26	58.9 (3)	C30—C25—Ru1—N2	−66.6 (3)
C1—N1—Ru1—C24	−15.8 (3)	C26—C25—Ru1—C23	−101.55 (19)
C5—N1—Ru1—C24	162.13 (19)	C24—C25—Ru1—C23	29.79 (18)
C1—N1—Ru1—C25	−17.5 (5)	C30—C25—Ru1—C23	142.8 (4)
C5—N1—Ru1—C25	160.4 (3)	C26—C25—Ru1—C22	−64.20 (18)
C1—N1—Ru1—Cl1	75.4 (2)	C24—C25—Ru1—C22	67.14 (18)
C5—N1—Ru1—Cl1	−106.65 (19)	C30—C25—Ru1—C22	−179.8 (3)
C6—N2—Ru1—N1	23.60 (19)	C26—C25—Ru1—C21	−27.67 (19)
C14—N2—Ru1—N1	−168.1 (2)	C24—C25—Ru1—C21	103.7 (2)
C6—N2—Ru1—C23	−52.4 (3)	C30—C25—Ru1—C21	−143.3 (4)
C14—N2—Ru1—C23	115.8 (3)	C24—C25—Ru1—C26	131.3 (3)
C6—N2—Ru1—C22	−61.0 (2)	C30—C25—Ru1—C26	−115.6 (4)
C14—N2—Ru1—C22	107.3 (2)	C26—C25—Ru1—C24	−131.3 (3)
C6—N2—Ru1—C21	−90.9 (2)	C30—C25—Ru1—C24	113.0 (4)
C14—N2—Ru1—C21	77.3 (2)	C26—C25—Ru1—Cl1	139.24 (16)
C6—N2—Ru1—C26	−128.9 (2)	C24—C25—Ru1—Cl1	−89.41 (17)
C14—N2—Ru1—C26	39.3 (2)	C30—C25—Ru1—Cl1	23.6 (3)
C6—N2—Ru1—C24	−156.5 (3)		

supplementary materials

C15—C16—C17—C20	175.3 (3)	C21—C22—Ru1—C24	103.4 (2)
C19—C16—C17—C20	−3.5 (4)	C23—C22—Ru1—C24	−27.29 (17)
C16—C17—C18—C13	2.6 (4)	C28—C22—Ru1—C24	−139.5 (3)
C20—C17—C18—C13	−176.1 (3)	C21—C22—Ru1—C25	66.2 (2)
N3—C13—C18—C17	175.7 (3)	C23—C22—Ru1—C25	−64.46 (18)
C14—C13—C18—C17	0.3 (4)	C28—C22—Ru1—C25	−176.7 (3)
C26—C21—C22—C23	0.0 (4)	C21—C22—Ru1—Cl1	145.1 (2)
C27—C21—C22—C23	176.5 (3)	C23—C22—Ru1—Cl1	14.4 (3)
Ru1—C21—C22—C23	54.9 (2)	C28—C22—Ru1—Cl1	−97.9 (3)
C26—C21—C22—C28	−179.2 (3)	C22—C21—Ru1—N1	56.0 (2)
C27—C21—C22—C28	−2.7 (5)	C26—C21—Ru1—N1	−173.84 (17)
Ru1—C21—C22—C28	−124.2 (3)	C27—C21—Ru1—N1	−62.6 (4)
C26—C21—C22—Ru1	−55.0 (2)	C22—C21—Ru1—N2	131.2 (2)
C27—C21—C22—Ru1	121.5 (3)	C26—C21—Ru1—N2	−98.63 (19)
C21—C22—C23—C24	−1.6 (4)	C27—C21—Ru1—N2	12.6 (3)
C28—C22—C23—C24	177.7 (3)	C22—C21—Ru1—C23	−30.5 (2)
Ru1—C22—C23—C24	54.0 (3)	C26—C21—Ru1—C23	99.6 (2)
C21—C22—C23—Ru1	−55.6 (2)	C27—C21—Ru1—C23	−149.2 (4)
C28—C22—C23—Ru1	123.6 (3)	C26—C21—Ru1—C22	130.1 (3)
C22—C23—C24—C25	1.4 (4)	C27—C21—Ru1—C22	−118.6 (4)
Ru1—C23—C24—C25	54.4 (2)	C22—C21—Ru1—C26	−130.1 (3)
C22—C23—C24—C29	−176.7 (3)	C27—C21—Ru1—C26	111.2 (4)
Ru1—C23—C24—C29	−123.7 (3)	C22—C21—Ru1—C24	−66.7 (2)
C22—C23—C24—Ru1	−53.0 (2)	C26—C21—Ru1—C24	63.4 (2)
C23—C24—C25—C26	0.3 (4)	C27—C21—Ru1—C24	174.7 (4)
C29—C24—C25—C26	178.5 (3)	C22—C21—Ru1—C25	−103.7 (2)
Ru1—C24—C25—C26	53.7 (2)	C26—C21—Ru1—C25	26.45 (18)
C23—C24—C25—C30	−177.7 (3)	C27—C21—Ru1—C25	137.7 (4)
C29—C24—C25—C30	0.5 (4)	C22—C21—Ru1—Cl1	−141.3 (2)
Ru1—C24—C25—C30	−124.2 (3)	C26—C21—Ru1—Cl1	−11.1 (4)
C23—C24—C25—Ru1	−53.5 (2)	C27—C21—Ru1—Cl1	100.1 (4)
C29—C24—C25—Ru1	124.7 (3)	C25—C26—Ru1—N1	147.8 (2)
C24—C25—C26—C21	−1.9 (4)	C21—C26—Ru1—N1	12.0 (3)
C30—C25—C26—C21	176.0 (3)	C25—C26—Ru1—N2	−140.30 (17)
Ru1—C25—C26—C21	52.0 (3)	C21—C26—Ru1—N2	83.9 (2)
C24—C25—C26—Ru1	−53.9 (2)	C25—C26—Ru1—C23	67.18 (18)
C30—C25—C26—Ru1	124.1 (3)	C21—C26—Ru1—C23	−68.6 (2)
C22—C21—C26—C25	1.8 (5)	C25—C26—Ru1—C22	105.7 (2)
C27—C21—C26—C25	−175.0 (3)	C21—C26—Ru1—C22	−30.1 (2)
Ru1—C21—C26—C25	−53.0 (3)	C25—C26—Ru1—C21	135.8 (3)
C22—C21—C26—Ru1	54.8 (3)	C25—C26—Ru1—C24	30.00 (18)
C27—C21—C26—Ru1	−122.0 (3)	C21—C26—Ru1—C24	−105.8 (2)
C2—C1—N1—C5	−1.9 (5)	C21—C26—Ru1—C25	−135.8 (3)
C2—C1—N1—Ru1	176.0 (3)	C25—C26—Ru1—Cl1	−48.97 (19)
C4—C5—N1—C1	0.3 (4)	C21—C26—Ru1—Cl1	175.25 (19)
C6—C5—N1—C1	−171.9 (2)	C23—C24—Ru1—N1	−48.4 (2)
C4—C5—N1—Ru1	−177.7 (2)	C25—C24—Ru1—N1	−179.10 (16)
C6—C5—N1—Ru1	10.1 (3)	C29—C24—Ru1—N1	65.9 (3)
C7—C6—N2—C14	−11.3 (4)	C23—C24—Ru1—N2	131.8 (3)