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ISSN 2414-3146

(η^4-Bicyclo[2.2.1]hepta-2,5-diene)bis(η^2-3,5-di-*tert*-butyl-1,2,4-diazaphospholido)ruthenium

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Received 14 January 2016
Accepted 18 January 2016

Edited by A. J. Lough, University of Toronto, Canada

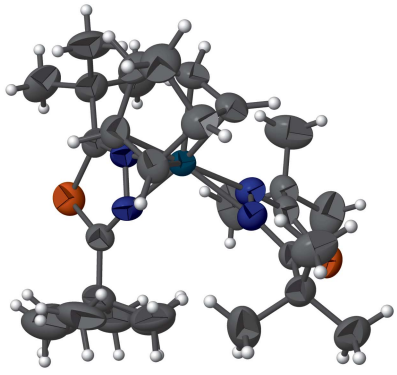
Keywords: crystal structure; ruthenium; 1,2,4-diazaphospholide.

CCDC reference: 660752

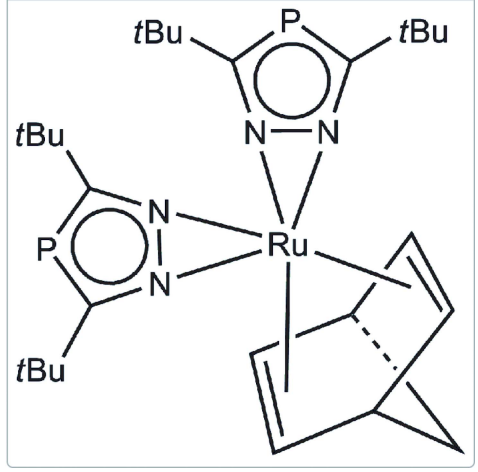
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[Ru(C_{10}H_{18}N_2P)_2(C_7H_8)]$, the two 1,2,4-diazaphospholide (dp) ligands are coordinated to the Ru^{II} ion in an almost perfect η^2-geometry, with Ru—N bond lengths in the range 2.071 (3)–2.135 (3) Å. The N—Ru—N angles within each η^2-1,2,4-diazaphospholide ligand are similar, with values of 37.10 (11) and 37.44 (11)°, and hence the coodination geometry around the Ru^{II} ion is highly distorted from the expected octahedral geometry. One of the *tert*-butyl groups was refined as disordered over two sets of sites, with a ratio of occupancies of 0.719 (6):0.281 (6).

3D view



Chemical scheme



Structure description

The molecular structure of the title compound which incorporates a bicyclo[2.2.1]hepta-2,5-diene (NBD) ligand, $[(\eta^4\text{-NBD})Ru(\eta^2\text{-3,5-}t\text{Bu}_2\text{dp})_2]$, is illustrated in Fig. 1. The two 1,2,4-diazaphospholide (dp) ligands are coordinated to the ruthenium(II) atom with nearly perfect η^2 geometry, with Ru—N bond lengths ranging from 2.071 (3) to 2.135 (3) Å. The N—Ru—N angles within each η^2-1,2,4-diazaphospholide ligand are 37.10 (11) and 37.44 (11)°. The N—N bonds of the dp ligands are oriented with respect to the Ru^{II} so as to suggest σ-donation of the in-plane nitrogen atom lone pairs to the metal atom while the coordinating bicyclo[2.2.1]hepta-2,5-diene ligand is in a π-bonding mode with η^4-coordination. This may be evidenced by the C═C bond lengths [C21═C22 1.395 (5); C26═C27 1.400 (5) Å] and Ru—C bonds [Ru1—C21 2.124 (4); Ru1—C22 2.133 (3) Å]. The bond angles (N1/N2-centroid)—Ru—(C26/C27-centroid) and (N3/N4-centroid)—Ru—(C21/C22-centroid) are 76.6 and 80.0°, respectively, suggesting a psuedo-tetrahedral arrangement of the ligands around Ru^{II}, although the coordination is highly distorted from the expected octahedral arrangement.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Ru(C_{10}H_{18}N_2P)_2(C_7H_8)]$
M_r	587.67
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	10.922 (5), 11.661 (5), 13.641 (6)
α, β, γ (°)	86.619 (5), 79.750 (5), 62.441 (5)
V (Å^3)	1515.2 (11)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.64
Crystal size (mm)	0.15 × 0.12 × 0.10
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.910, 0.938
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6963, 5438, 4323
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.102, 1.00
No. of reflections	5438
No. of parameters	335
No. of restraints	36
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.73, −0.29

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008), *publCIF* (Westrip, 2010).

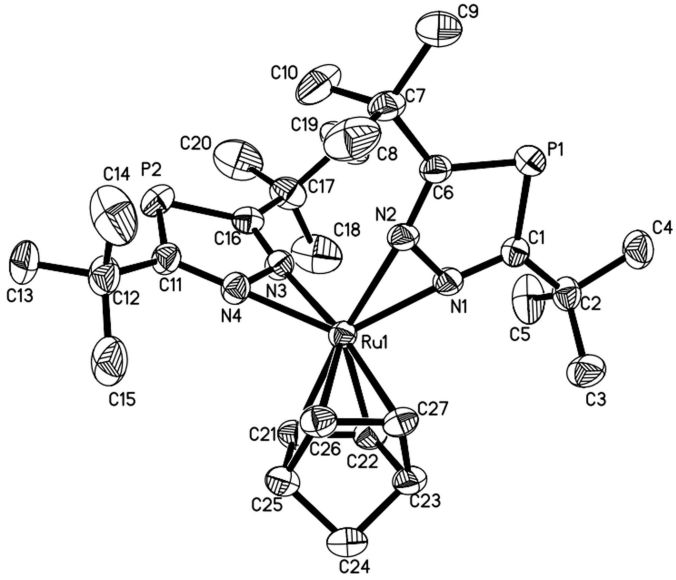


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms and the minor disorder component have been omitted for clarity.

Synthesis and crystallization

All manipulations were carried out using standard Schlenk techniques in an N_2 filled glovebox. Solvents were dried over and distilled from Na/K alloy prior to use.

To a mixture of [(η^4-NBD)$RuCl_2$] (Albers *et al.*, 1989) (0.53 g, 2.0 mmol) and K[3,5-*t*Bu_2dp] (Zheng *et al.*, 2006) (1.04 g, 4.4 mmol) was added 30 ml tetrahydrofuran *via* a syringe. After the solution had been stirred for 48 h, the volatile components were removed in high vacuum. The resulting residue was extracted with ether (3 × 10 ml). The solvent was reduced to about 10 ml and then the solution was kept at 253 K to give the title compound as deep-red crystals (0.88 g, 75%). M.p. 453 K (dep.). ^{1}H NMR (C_6D_6, 296 K): δ = 1.15 (*d*, 18 H, CH_3), 1.65 (*d*, 18 H, CH_3), 1.19 (*d*, 2 H, CH_2), 3.39 (*d*, 2 H, C*H*), 3.62 (*t*, 2 H, = C*H*), 4.83 (*t*, 2 H, = C*H*) p.p.m.; ^{13}C{^{1}H} NMR (C_6D_6, 296 K): δ = 31.80 (*d*, $^3J_{CP}$ = 6.25 Hz, *C*H_3), 33.94 (*d*, $^3J_{CP}$ = 301.25 Hz, *C*H_3), 50.92 (*s*, *C*H), 58.63 (*s*, *C*H_2), 59.49, 60.26 (*s*, = *C*H), 181.81 (*d*, $^2J_{CP}$ = 62.13 *C*CH_3), 185.24 (*d*, $^1J_{CP}$ = 63.50 Hz, P*C*N) p.p.m.; ^{31}P{^{1}H} NMR (C_6D_6, 296 K): δ = 67.79 (s), 114.75 (*s*) p.p.m.; MS(EI) *m*/*z* (%): 588 ([M^+], 100); IR (KBr, Nujol mull, cm^{-1}): 1463(*s*), 1377(*s*), 1291(*m*), 1086(*w*), 779(*w*), 718(*m*); Analysis calculated for $C_{27}H_{44}RuP_2N_4$: C 55.18; H 7.55; N 9.53. Found: C 55.31; H, 7.61; N, 9.67.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. One of the *tert*-butyl groups (C18/C19/C20) was refined as disordered over two sets of sites with an occupancy ratio of 0.719 (6):0.281 (6).

Acknowledgements

This work was supported by the National Natural Science Foundation of China (NSFC; grant No. 21272143).

References

Albers, M. O., Singleton, E. & Yates, J. E. (1989). *Inorg. Synth.* **26**, 249–258.
Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zheng, W., Zhang, G. & Fan, K. (2006). *Organometallics*, **25**, 1548–1550.

full crystallographic data

IUCrData (2016). **1**, x160110 [doi:10.1107/S2414314616001103]

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(η^4-Bicyclo[2.2.1]hepta-2,5-diene)bis(η^2-3,5-di-*tert*-butyl-1,2,4-diazaphospholido)ruthenium

Crystal data

$[Ru(C_{10}H_{18}N_2P)_2(C_7H_8)]$
M_r = 587.67
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 10.922 (5) Å
b = 11.661 (5) Å
c = 13.641 (6) Å
α = 86.619 (5)°
β = 79.750 (5)°
γ = 62.441 (5)°
V = 1515.2 (11) Å^3
Z = 2
$F(000)$ = 616
D_x = 1.288 Mg m^{-3}
Melting point: 453 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 894 reflections
θ = 2.8–25.9°
μ = 0.64 mm^{-1}
T = 293 K
Block, colourless
0.15 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.910, T_{max} = 0.938
6963 measured reflections
5438 independent reflections
4323 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.4°, θ_{min} = 1.5°
h = −12→13
k = −14→14
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.102
S = 1.00
5438 reflections
335 parameters
36 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ru1	0.97741 (3)	0.75701 (3)	0.69344 (2)	0.04137 (11)	
P1	0.64266 (12)	1.08096 (10)	0.55908 (8)	0.0570 (3)	
P2	0.74186 (13)	0.68084 (12)	0.96868 (8)	0.0676 (3)	
N1	0.8569 (3)	0.8730 (3)	0.5912 (2)	0.0471 (7)	
N2	0.8111 (3)	0.9430 (3)	0.6770 (2)	0.0483 (7)	
N3	0.8358 (3)	0.7021 (3)	0.7873 (2)	0.0499 (7)	
N4	0.9151 (3)	0.7239 (3)	0.8407 (2)	0.0507 (8)	
C1	0.7819 (4)	0.9295 (3)	0.5202 (3)	0.0462 (8)	
C2	0.8208 (5)	0.8601 (4)	0.4197 (3)	0.0555 (10)	
C3	0.9706 (5)	0.8301 (5)	0.3733 (3)	0.0777 (14)	
H3A	0.9949	0.7866	0.3099	0.117*	
H3B	1.0337	0.7755	0.4165	0.117*	
H3C	0.9775	0.9093	0.3643	0.117*	
C4	0.7207 (5)	0.9482 (4)	0.3512 (3)	0.0724 (13)	
H4A	0.7447	0.9053	0.2875	0.109*	
H4B	0.7281	1.0272	0.3427	0.109*	
H4C	0.6263	0.9674	0.3803	0.109*	
C5	0.8029 (7)	0.7370 (4)	0.4348 (4)	0.0942 (18)	
H5A	0.8273	0.6926	0.3717	0.141*	
H5B	0.7072	0.7598	0.4626	0.141*	
H5C	0.8630	0.6815	0.4793	0.141*	
C6	0.6988 (4)	1.0549 (3)	0.6753 (3)	0.0478 (9)	
C7	0.6301 (4)	1.1376 (4)	0.7697 (3)	0.0573 (10)	
C8	0.7348 (6)	1.1659 (5)	0.8091 (4)	0.1039 (19)	
H8A	0.6899	1.2184	0.8690	0.156*	
H8B	0.7692	1.2112	0.7600	0.156*	
H8C	0.8116	1.0860	0.8232	0.156*	
C9	0.5068 (6)	1.2648 (5)	0.7496 (4)	0.1033 (19)	
H9A	0.4641	1.3165	0.8100	0.155*	
H9B	0.4392	1.2474	0.7260	0.155*	
H9C	0.5396	1.3108	0.7000	0.155*	
C10	0.5777 (6)	1.0641 (5)	0.8472 (4)	0.0978 (18)	
H10A	0.5337	1.1155	0.9077	0.147*	
H10B	0.6553	0.9842	0.8602	0.147*	
H10C	0.5110	1.0463	0.8226	0.147*	

C11	0.8812 (4)	0.7183 (4)	0.9387 (3)	0.0535 (9)	
C12	0.9547 (5)	0.7475 (4)	1.0109 (3)	0.0688 (12)	
C13	0.9391 (6)	0.6801 (5)	1.1093 (3)	0.0919 (17)	
H13A	0.8415	0.7110	1.1359	0.138*	
H13B	0.9837	0.6991	1.1561	0.138*	
H13C	0.9825	0.5883	1.0972	0.138*	
C14	0.8812 (7)	0.8946 (5)	1.0312 (5)	0.126 (3)	
H14A	0.7837	0.9231	1.0572	0.188*	
H14B	0.8901	0.9377	0.9702	0.188*	
H14C	0.9238	0.9149	1.0787	0.188*	
C15	1.1085 (6)	0.6987 (6)	0.9697 (4)	0.0981 (18)	
H15A	1.1526	0.7183	1.0166	0.147*	
H15B	1.1188	0.7401	0.9080	0.147*	
H15C	1.1519	0.6068	0.9581	0.147*	
C16	0.7396 (4)	0.6784 (4)	0.8408 (3)	0.0530 (9)	
C17	0.6415 (5)	0.6519 (4)	0.7904 (4)	0.0709 (12)	
C21	1.1543 (4)	0.5732 (3)	0.6960 (3)	0.0544 (10)	
H21	1.1489	0.5004	0.7327	0.065*	
C22	1.1300 (4)	0.5984 (3)	0.5981 (3)	0.0539 (10)	
H22	1.1057	0.5452	0.5603	0.065*	
C23	1.2215 (4)	0.6608 (4)	0.5502 (3)	0.0586 (10)	
H23	1.2396	0.6620	0.4773	0.070*	
C24	1.3504 (4)	0.5920 (5)	0.6037 (3)	0.0735 (12)	
H24A	1.4040	0.5003	0.5871	0.088*	
H24B	1.4111	0.6330	0.5931	0.088*	
C25	1.2607 (4)	0.6204 (4)	0.7078 (3)	0.0613 (11)	
H25	1.3101	0.5890	0.7648	0.074*	
C26	1.1641 (4)	0.7663 (4)	0.7051 (3)	0.0551 (10)	
H26	1.1656	0.8296	0.7484	0.066*	
C27	1.1412 (4)	0.7915 (4)	0.6065 (3)	0.0518 (9)	
H27	1.1243	0.8739	0.5749	0.062*	
C18	0.7298 (8)	0.5446 (8)	0.7118 (7)	0.112 (3)	0.719 (6)
H18A	0.7735	0.4635	0.7439	0.168*	0.719 (6)
H18B	0.8007	0.5628	0.6726	0.168*	0.719 (6)
H18C	0.6710	0.5398	0.6693	0.168*	0.719 (6)
C19	0.5422 (10)	0.7740 (8)	0.7461 (9)	0.122 (3)	0.719 (6)
H19A	0.4621	0.8224	0.7958	0.184*	0.719 (6)
H19B	0.5123	0.7521	0.6911	0.184*	0.719 (6)
H19C	0.5890	0.8254	0.7231	0.184*	0.719 (6)
C20	0.5612 (9)	0.5975 (10)	0.8662 (7)	0.121 (3)	0.719 (6)
H20A	0.6193	0.5474	0.9137	0.181*	0.719 (6)
H20B	0.5367	0.5433	0.8320	0.181*	0.719 (6)
H20C	0.4775	0.6676	0.9002	0.181*	0.719 (6)
C18′	0.4912 (16)	0.740 (2)	0.8434 (19)	0.122 (3)	0.281 (6)
H18D	0.4768	0.7088	0.9091	0.182*	0.281 (6)
H18E	0.4252	0.7383	0.8061	0.182*	0.281 (6)
H18F	0.4779	0.8266	0.8481	0.182*	0.281 (6)
C20′	0.629 (3)	0.723 (2)	0.6899 (15)	0.120 (4)	0.281 (6)

H20D	0.7165	0.6810	0.6450	0.181*	0.281 (6)
H20E	0.6069	0.8113	0.7013	0.181*	0.281 (6)
H20F	0.5563	0.7198	0.6615	0.181*	0.281 (6)
C19′	0.667 (3)	0.5115 (16)	0.790 (2)	0.119 (3)	0.281 (6)
H19D	0.6845	0.4763	0.8547	0.179*	0.281 (6)
H19E	0.7476	0.4619	0.7409	0.179*	0.281 (6)
H19F	0.5867	0.5081	0.7751	0.179*	0.281 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.04163 (19)	0.04155 (17)	0.03939 (17)	−0.01768 (13)	−0.00559 (12)	−0.00448 (11)
P1	0.0529 (7)	0.0499 (6)	0.0562 (6)	−0.0147 (5)	−0.0079 (5)	0.0067 (5)
P2	0.0661 (8)	0.0777 (8)	0.0537 (7)	−0.0345 (7)	0.0081 (5)	−0.0034 (6)
N1	0.0462 (18)	0.0443 (16)	0.0436 (17)	−0.0146 (14)	−0.0053 (14)	−0.0077 (13)
N2	0.0420 (18)	0.0479 (17)	0.0447 (17)	−0.0121 (15)	−0.0038 (13)	−0.0096 (13)
N3	0.0477 (19)	0.064 (2)	0.0451 (17)	−0.0300 (17)	−0.0102 (14)	−0.0027 (14)
N4	0.053 (2)	0.0606 (19)	0.0447 (18)	−0.0307 (17)	−0.0105 (14)	−0.0001 (14)
C1	0.050 (2)	0.0460 (19)	0.044 (2)	−0.0233 (18)	−0.0073 (16)	0.0017 (16)
C2	0.068 (3)	0.055 (2)	0.046 (2)	−0.028 (2)	−0.0159 (19)	0.0010 (17)
C3	0.064 (3)	0.098 (4)	0.055 (3)	−0.023 (3)	−0.004 (2)	−0.024 (2)
C4	0.083 (3)	0.087 (3)	0.052 (2)	−0.039 (3)	−0.024 (2)	0.008 (2)
C5	0.163 (6)	0.070 (3)	0.071 (3)	−0.066 (4)	−0.040 (3)	0.008 (2)
C6	0.044 (2)	0.0405 (19)	0.051 (2)	−0.0156 (17)	0.0005 (16)	−0.0010 (16)
C7	0.054 (3)	0.045 (2)	0.060 (2)	−0.0163 (19)	0.0055 (19)	−0.0100 (18)
C8	0.095 (4)	0.117 (5)	0.100 (4)	−0.049 (4)	0.005 (3)	−0.056 (3)
C9	0.100 (4)	0.064 (3)	0.090 (4)	0.005 (3)	0.002 (3)	−0.015 (3)
C10	0.121 (5)	0.073 (3)	0.074 (3)	−0.040 (3)	0.037 (3)	−0.016 (3)
C11	0.061 (3)	0.052 (2)	0.042 (2)	−0.022 (2)	−0.0072 (18)	−0.0043 (17)
C12	0.084 (3)	0.075 (3)	0.051 (2)	−0.036 (3)	−0.020 (2)	−0.005 (2)
C13	0.108 (4)	0.111 (4)	0.045 (3)	−0.036 (4)	−0.021 (3)	−0.004 (3)
C14	0.180 (7)	0.079 (4)	0.129 (5)	−0.048 (4)	−0.082 (5)	−0.010 (3)
C15	0.107 (5)	0.154 (5)	0.066 (3)	−0.081 (4)	−0.033 (3)	0.002 (3)
C16	0.043 (2)	0.051 (2)	0.062 (2)	−0.0209 (19)	−0.0056 (18)	−0.0002 (18)
C17	0.056 (3)	0.076 (3)	0.093 (3)	−0.039 (3)	−0.019 (2)	0.006 (3)
C21	0.051 (2)	0.0396 (19)	0.061 (2)	−0.0114 (18)	−0.0076 (19)	−0.0028 (17)
C22	0.057 (3)	0.0381 (19)	0.057 (2)	−0.0144 (18)	−0.0014 (19)	−0.0144 (17)
C23	0.047 (2)	0.062 (2)	0.053 (2)	−0.015 (2)	0.0001 (18)	−0.0096 (19)
C24	0.045 (3)	0.077 (3)	0.079 (3)	−0.016 (2)	0.001 (2)	−0.005 (2)
C25	0.045 (2)	0.068 (3)	0.061 (3)	−0.016 (2)	−0.0107 (19)	−0.001 (2)
C26	0.047 (2)	0.061 (2)	0.062 (2)	−0.029 (2)	−0.0069 (18)	−0.0069 (19)
C27	0.044 (2)	0.057 (2)	0.056 (2)	−0.0276 (19)	0.0017 (17)	−0.0047 (18)
C18	0.091 (5)	0.106 (5)	0.163 (7)	−0.055 (4)	−0.037 (5)	−0.040 (5)
C19	0.088 (6)	0.110 (5)	0.182 (8)	−0.039 (5)	−0.076 (5)	0.008 (5)
C20	0.089 (5)	0.123 (5)	0.175 (7)	−0.063 (4)	−0.038 (4)	−0.003 (5)
C18′	0.085 (5)	0.119 (5)	0.179 (7)	−0.051 (5)	−0.055 (5)	0.001 (5)
C20′	0.087 (7)	0.114 (7)	0.180 (9)	−0.043 (6)	−0.079 (7)	0.010 (7)
C19′	0.092 (5)	0.113 (5)	0.172 (7)	−0.056 (5)	−0.038 (5)	−0.020 (5)

Geometric parameters (Å, º)

Ru1—N1	2.071 (3)	C13—H13B	0.9600
Ru1—N4	2.077 (3)	C13—H13C	0.9600
Ru1—N2	2.121 (3)	C14—H14A	0.9600
Ru1—C21	2.124 (4)	C14—H14B	0.9600
Ru1—C26	2.125 (4)	C14—H14C	0.9600
Ru1—C22	2.133 (3)	C15—H15A	0.9600
Ru1—N3	2.135 (3)	C15—H15B	0.9600
Ru1—C27	2.150 (4)	C15—H15C	0.9600
P1—C1	1.747 (4)	C16—C17	1.520 (6)
P1—C6	1.760 (4)	C17—C19	1.507 (7)
P2—C11	1.745 (4)	C17—C18	1.523 (7)
P2—C16	1.751 (4)	C17—C19′	1.527 (15)
N1—C1	1.324 (4)	C17—C20	1.535 (7)
N1—N2	1.346 (4)	C17—C18′	1.541 (15)
N2—C6	1.316 (4)	C17—C20′	1.551 (15)
N3—C16	1.298 (5)	C21—C22	1.395 (5)
N3—N4	1.341 (4)	C21—C25	1.534 (5)
N4—C11	1.328 (5)	C21—H21	0.9800
C1—C2	1.523 (5)	C22—C23	1.529 (5)
C2—C3	1.526 (6)	C22—H22	0.9800
C2—C4	1.529 (5)	C23—C27	1.530 (5)
C2—C5	1.534 (6)	C23—C24	1.551 (6)
C3—H3A	0.9600	C23—H23	0.9800
C3—H3B	0.9600	C24—C25	1.532 (6)
C3—H3C	0.9600	C24—H24A	0.9700
C4—H4A	0.9600	C24—H24B	0.9700
C4—H4B	0.9600	C25—C26	1.534 (5)
C4—H4C	0.9600	C25—H25	0.9800
C5—H5A	0.9600	C26—C27	1.400 (5)
C5—H5B	0.9600	C26—H26	0.9800
C5—H5C	0.9600	C27—H27	0.9800
C6—C7	1.512 (5)	C18—H18A	0.9600
C7—C8	1.515 (6)	C18—H18B	0.9600
C7—C10	1.519 (6)	C18—H18C	0.9600
C7—C9	1.525 (6)	C19—H19A	0.9600
C8—H8A	0.9600	C19—H19B	0.9600
C8—H8B	0.9600	C19—H19C	0.9600
C8—H8C	0.9600	C20—H20A	0.9600
C9—H9A	0.9600	C20—H20B	0.9600
C9—H9B	0.9600	C20—H20C	0.9600
C9—H9C	0.9600	C18′—H18D	0.9600
C10—H10A	0.9600	C18′—H18E	0.9600
C10—H10B	0.9600	C18′—H18F	0.9600
C10—H10C	0.9600	C20′—H20D	0.9600
C11—C12	1.518 (5)	C20′—H20E	0.9600
C12—C15	1.509 (7)	C20′—H20F	0.9600

C12—C13	1.537 (6)	C19′—H19D	0.9600
C12—C14	1.539 (6)	C19′—H19E	0.9600
C13—H13A	0.9600	C19′—H19F	0.9600
N1—Ru1—N4	129.37 (12)	H14A—C14—H14B	109.5
N1—Ru1—N2	37.44 (11)	C12—C14—H14C	109.5
N4—Ru1—N2	100.08 (12)	H14A—C14—H14C	109.5
N1—Ru1—C21	138.29 (14)	H14B—C14—H14C	109.5
N4—Ru1—C21	85.52 (14)	C12—C15—H15A	109.5
N2—Ru1—C21	174.32 (13)	C12—C15—H15B	109.5
N1—Ru1—C26	118.31 (14)	H15A—C15—H15B	109.5
N4—Ru1—C26	100.32 (14)	C12—C15—H15C	109.5
N2—Ru1—C26	111.66 (14)	H15A—C15—H15C	109.5
C21—Ru1—C26	66.04 (15)	H15B—C15—H15C	109.5
N1—Ru1—C22	100.09 (13)	N3—C16—C17	119.9 (4)
N4—Ru1—C22	119.54 (14)	N3—C16—P2	112.6 (3)
N2—Ru1—C22	137.02 (13)	C17—C16—P2	127.5 (3)
C21—Ru1—C22	38.25 (14)	C19—C17—C16	110.1 (5)
C26—Ru1—C22	78.78 (15)	C19—C17—C18	112.0 (7)
N1—Ru1—N3	102.47 (12)	C16—C17—C18	108.4 (4)
N4—Ru1—N3	37.10 (11)	C19—C17—C19′	134.1 (10)
N2—Ru1—N3	90.76 (12)	C16—C17—C19′	115.7 (9)
C21—Ru1—N3	94.32 (14)	C18—C17—C19′	50.5 (11)
C26—Ru1—N3	136.30 (13)	C19—C17—C20	111.1 (6)
C22—Ru1—N3	110.33 (14)	C16—C17—C20	109.6 (4)
N1—Ru1—C27	84.55 (14)	C18—C17—C20	105.6 (5)
N4—Ru1—C27	138.47 (14)	C19′—C17—C20	55.6 (11)
N2—Ru1—C27	96.30 (14)	C19—C17—C18′	57.0 (10)
C21—Ru1—C27	78.73 (16)	C16—C17—C18′	106.9 (9)
C26—Ru1—C27	38.23 (15)	C18—C17—C18′	144.6 (9)
C22—Ru1—C27	65.62 (15)	C19′—C17—C18′	109.8 (14)
N3—Ru1—C27	172.61 (13)	C20—C17—C18′	58.8 (10)
C1—P1—C6	87.57 (17)	C19—C17—C20′	40.4 (9)
C11—P2—C16	87.69 (18)	C16—C17—C20′	107.5 (9)
C1—N1—N2	114.3 (3)	C18—C17—C20′	75.5 (11)
C1—N1—Ru1	170.7 (3)	C19′—C17—C20′	118.1 (15)
N2—N1—Ru1	73.30 (18)	C20—C17—C20′	140.1 (9)
C6—N2—N1	114.1 (3)	C18′—C17—C20′	96.7 (14)
C6—N2—Ru1	173.2 (3)	C22—C21—C25	106.1 (4)
N1—N2—Ru1	69.26 (17)	C22—C21—Ru1	71.2 (2)
C16—N3—N4	114.0 (3)	C25—C21—Ru1	98.0 (2)
C16—N3—Ru1	174.2 (3)	C22—C21—H21	122.9
N4—N3—Ru1	69.12 (19)	C25—C21—H21	122.9
C11—N4—N3	114.6 (3)	Ru1—C21—H21	122.9
C11—N4—Ru1	170.1 (3)	C21—C22—C23	106.2 (3)
N3—N4—Ru1	73.79 (19)	C21—C22—Ru1	70.5 (2)
N1—C1—C2	119.9 (3)	C23—C22—Ru1	98.2 (2)
N1—C1—P1	112.0 (3)	C21—C22—H22	122.9

C2—C1—P1	128.1 (3)	C23—C22—H22	122.9
C1—C2—C3	109.5 (3)	Ru1—C22—H22	122.9
C1—C2—C4	108.9 (3)	C22—C23—C27	98.7 (3)
C3—C2—C4	109.3 (4)	C22—C23—C24	101.6 (3)
C1—C2—C5	108.9 (3)	C27—C23—C24	101.5 (3)
C3—C2—C5	111.9 (4)	C22—C23—H23	117.3
C4—C2—C5	108.3 (4)	C27—C23—H23	117.3
C2—C3—H3A	109.5	C24—C23—H23	117.3
C2—C3—H3B	109.5	C25—C24—C23	93.6 (3)
H3A—C3—H3B	109.5	C25—C24—H24A	113.0
C2—C3—H3C	109.5	C23—C24—H24A	113.0
H3A—C3—H3C	109.5	C25—C24—H24B	113.0
H3B—C3—H3C	109.5	C23—C24—H24B	113.0
C2—C4—H4A	109.5	H24A—C24—H24B	110.4
C2—C4—H4B	109.5	C24—C25—C21	101.8 (3)
H4A—C4—H4B	109.5	C24—C25—C26	102.1 (3)
C2—C4—H4C	109.5	C21—C25—C26	98.0 (3)
H4A—C4—H4C	109.5	C24—C25—H25	117.3
H4B—C4—H4C	109.5	C21—C25—H25	117.3
C2—C5—H5A	109.5	C26—C25—H25	117.3
C2—C5—H5B	109.5	C27—C26—C25	106.0 (3)
H5A—C5—H5B	109.5	C27—C26—Ru1	71.8 (2)
C2—C5—H5C	109.5	C25—C26—Ru1	97.9 (2)
H5A—C5—H5C	109.5	C27—C26—H26	122.8
H5B—C5—H5C	109.5	C25—C26—H26	122.8
N2—C6—C7	119.1 (3)	Ru1—C26—H26	122.8
N2—C6—P1	112.0 (3)	C26—C27—C23	106.1 (3)
C7—C6—P1	128.7 (3)	C26—C27—Ru1	69.9 (2)
C6—C7—C8	110.3 (4)	C23—C27—Ru1	97.5 (2)
C6—C7—C10	108.5 (3)	C26—C27—H27	123.2
C8—C7—C10	109.2 (4)	C23—C27—H27	123.2
C6—C7—C9	110.2 (4)	Ru1—C27—H27	123.2
C8—C7—C9	109.2 (4)	C17—C18—H18A	109.5
C10—C7—C9	109.4 (4)	C17—C18—H18B	109.5
C7—C8—H8A	109.5	H18A—C18—H18B	109.5
C7—C8—H8B	109.5	C17—C18—H18C	109.5
H8A—C8—H8B	109.5	H18A—C18—H18C	109.5
C7—C8—H8C	109.5	H18B—C18—H18C	109.5
H8A—C8—H8C	109.5	C17—C19—H19A	109.5
H8B—C8—H8C	109.5	C17—C19—H19B	109.5
C7—C9—H9A	109.5	H19A—C19—H19B	109.5
C7—C9—H9B	109.5	C17—C19—H19C	109.5
H9A—C9—H9B	109.5	H19A—C19—H19C	109.5
C7—C9—H9C	109.5	H19B—C19—H19C	109.5
H9A—C9—H9C	109.5	C17—C20—H20A	109.5
H9B—C9—H9C	109.5	C17—C20—H20B	109.5
C7—C10—H10A	109.5	H20A—C20—H20B	109.5
C7—C10—H10B	109.5	C17—C20—H20C	109.5

H10A—C10—H10B	109.5	H20A—C20—H20C	109.5
C7—C10—H10C	109.5	H20B—C20—H20C	109.5
H10A—C10—H10C	109.5	C17—C18′—H18D	109.5
H10B—C10—H10C	109.5	C17—C18′—H18E	109.5
N4—C11—C12	121.8 (4)	H18D—C18′—H18E	109.5
N4—C11—P2	111.1 (3)	C17—C18′—H18F	109.5
C12—C11—P2	127.0 (3)	H18D—C18′—H18F	109.5
C15—C12—C11	111.1 (4)	H18E—C18′—H18F	109.5
C15—C12—C13	109.2 (4)	C17—C20′—H20D	109.5
C11—C12—C13	108.5 (4)	C17—C20′—H20E	109.5
C15—C12—C14	111.2 (5)	H20D—C20′—H20E	109.5
C11—C12—C14	108.1 (4)	C17—C20′—H20F	109.5
C13—C12—C14	108.8 (4)	H20D—C20′—H20F	109.5
C12—C13—H13A	109.5	H20E—C20′—H20F	109.5
C12—C13—H13B	109.5	C17—C19′—H19D	109.5
H13A—C13—H13B	109.5	C17—C19′—H19E	109.5
C12—C13—H13C	109.5	H19D—C19′—H19E	109.5
H13A—C13—H13C	109.5	C17—C19′—H19F	109.5
H13B—C13—H13C	109.5	H19D—C19′—H19F	109.5
C12—C14—H14A	109.5	H19E—C19′—H19F	109.5
C12—C14—H14B	109.5		

N4—Ru1—N1—C1	−100.0 (17)	Ru1—N3—C16—C17	59 (3)
N2—Ru1—N1—C1	−145.6 (18)	N4—N3—C16—P2	−0.4 (4)
C21—Ru1—N1—C1	40.3 (18)	Ru1—N3—C16—P2	−122 (3)
C26—Ru1—N1—C1	125.2 (17)	C11—P2—C16—N3	0.7 (3)
C22—Ru1—N1—C1	42.6 (17)	C11—P2—C16—C17	−179.6 (4)
N3—Ru1—N1—C1	−71.1 (17)	N3—C16—C17—C19	−70.2 (7)
C27—Ru1—N1—C1	106.6 (17)	P2—C16—C17—C19	110.2 (6)
N4—Ru1—N1—N2	45.6 (2)	N3—C16—C17—C18	52.6 (6)
C21—Ru1—N1—N2	−174.1 (2)	P2—C16—C17—C18	−127.0 (5)
C26—Ru1—N1—N2	−89.2 (2)	N3—C16—C17—C19′	106.9 (14)
C22—Ru1—N1—N2	−171.8 (2)	P2—C16—C17—C19′	−72.7 (14)
N3—Ru1—N1—N2	74.5 (2)	N3—C16—C17—C20	167.3 (5)
C27—Ru1—N1—N2	−107.8 (2)	P2—C16—C17—C20	−12.3 (6)
C1—N1—N2—C6	0.6 (5)	N3—C16—C17—C18′	−130.4 (12)
Ru1—N1—N2—C6	−173.6 (3)	P2—C16—C17—C18′	50.0 (12)
C1—N1—N2—Ru1	174.3 (3)	N3—C16—C17—C20′	−27.5 (12)
N1—Ru1—N2—C6	121 (2)	P2—C16—C17—C20′	152.9 (11)
N4—Ru1—N2—C6	−25 (2)	N1—Ru1—C21—C22	3.6 (3)
C21—Ru1—N2—C6	164 (2)	N4—Ru1—C21—C22	153.9 (2)
C26—Ru1—N2—C6	−131 (2)	N2—Ru1—C21—C22	−35.5 (14)
C22—Ru1—N2—C6	133 (2)	C26—Ru1—C21—C22	−102.7 (3)
N3—Ru1—N2—C6	11 (2)	N3—Ru1—C21—C22	117.8 (2)
C27—Ru1—N2—C6	−167 (2)	C27—Ru1—C21—C22	−64.7 (2)
N4—Ru1—N2—N1	−145.86 (19)	N1—Ru1—C21—C25	108.0 (3)
C21—Ru1—N2—N1	43.7 (14)	N4—Ru1—C21—C25	−101.7 (2)
C26—Ru1—N2—N1	108.7 (2)	N2—Ru1—C21—C25	68.9 (14)

C22—Ru1—N2—N1	11.8 (3)	C26—Ru1—C21—C25	1.7 (2)
N3—Ru1—N2—N1	−109.7 (2)	C22—Ru1—C21—C25	104.4 (3)
C27—Ru1—N2—N1	72.5 (2)	N3—Ru1—C21—C25	−137.8 (2)
N1—Ru1—N3—C16	−18 (3)	C27—Ru1—C21—C25	39.7 (2)
N4—Ru1—N3—C16	123 (3)	C25—C21—C22—C23	0.1 (4)
N2—Ru1—N3—C16	17 (3)	Ru1—C21—C22—C23	93.2 (2)
C21—Ru1—N3—C16	−160 (3)	C25—C21—C22—Ru1	−93.1 (3)
C26—Ru1—N3—C16	141 (3)	N1—Ru1—C22—C21	−177.6 (2)
C22—Ru1—N3—C16	−124 (3)	N4—Ru1—C22—C21	−30.3 (3)
C27—Ru1—N3—C16	−180 (100)	N2—Ru1—C22—C21	175.2 (2)
N1—Ru1—N3—N4	−141.70 (19)	C26—Ru1—C22—C21	65.3 (2)
N2—Ru1—N3—N4	−105.8 (2)	N3—Ru1—C22—C21	−70.1 (3)
C21—Ru1—N3—N4	76.7 (2)	C27—Ru1—C22—C21	103.2 (3)
C26—Ru1—N3—N4	17.4 (3)	N1—Ru1—C22—C23	78.1 (2)
C22—Ru1—N3—N4	112.4 (2)	N4—Ru1—C22—C23	−134.6 (2)
C27—Ru1—N3—N4	56.9 (10)	N2—Ru1—C22—C23	70.8 (3)
C16—N3—N4—C11	−0.4 (5)	C21—Ru1—C22—C23	−104.3 (3)
Ru1—N3—N4—C11	174.4 (3)	C26—Ru1—C22—C23	−39.0 (2)
C16—N3—N4—Ru1	−174.7 (3)	N3—Ru1—C22—C23	−174.4 (2)
N1—Ru1—N4—C11	−97.4 (16)	C27—Ru1—C22—C23	−1.1 (2)
N2—Ru1—N4—C11	−71.2 (16)	C21—C22—C23—C27	−70.6 (3)
C21—Ru1—N4—C11	107.9 (16)	Ru1—C22—C23—C27	1.5 (3)
C26—Ru1—N4—C11	43.2 (16)	C21—C22—C23—C24	33.1 (4)
C22—Ru1—N4—C11	126.1 (16)	Ru1—C22—C23—C24	105.1 (3)
N3—Ru1—N4—C11	−148.9 (17)	C22—C23—C24—C25	−50.5 (4)
C27—Ru1—N4—C11	40.5 (17)	C27—C23—C24—C25	50.9 (4)
N1—Ru1—N4—N3	51.5 (2)	C23—C24—C25—C21	50.5 (4)
N2—Ru1—N4—N3	77.7 (2)	C23—C24—C25—C26	−50.4 (4)
C21—Ru1—N4—N3	−103.2 (2)	C22—C21—C25—C24	−33.7 (4)
C26—Ru1—N4—N3	−167.9 (2)	Ru1—C21—C25—C24	−106.4 (3)
C22—Ru1—N4—N3	−85.0 (2)	C22—C21—C25—C26	70.5 (3)
C27—Ru1—N4—N3	−170.6 (2)	Ru1—C21—C25—C26	−2.2 (3)
N2—N1—C1—C2	−179.1 (3)	C24—C25—C26—C27	32.9 (4)
Ru1—N1—C1—C2	−35.5 (19)	C21—C25—C26—C27	−71.1 (4)
N2—N1—C1—P1	−0.1 (4)	C24—C25—C26—Ru1	106.1 (3)
Ru1—N1—C1—P1	143.5 (16)	C21—C25—C26—Ru1	2.2 (3)
C6—P1—C1—N1	−0.3 (3)	N1—Ru1—C26—C27	−30.9 (3)
C6—P1—C1—C2	178.6 (4)	N4—Ru1—C26—C27	−177.0 (2)
N1—C1—C2—C3	−58.8 (5)	N2—Ru1—C26—C27	−71.7 (2)
P1—C1—C2—C3	122.4 (4)	C21—Ru1—C26—C27	102.6 (3)
N1—C1—C2—C4	−178.2 (3)	C22—Ru1—C26—C27	64.6 (2)
P1—C1—C2—C4	3.0 (5)	N3—Ru1—C26—C27	172.4 (2)
N1—C1—C2—C5	63.9 (5)	N1—Ru1—C26—C25	−135.2 (2)
P1—C1—C2—C5	−114.9 (4)	N4—Ru1—C26—C25	78.6 (2)
N1—N2—C6—C7	175.0 (3)	N2—Ru1—C26—C25	−176.1 (2)
Ru1—N2—C6—C7	57 (2)	C21—Ru1—C26—C25	−1.7 (2)
N1—N2—C6—P1	−0.8 (4)	C22—Ru1—C26—C25	−39.7 (2)
Ru1—N2—C6—P1	−119 (2)	N3—Ru1—C26—C25	68.1 (3)

C1—P1—C6—N2	0.6 (3)	C27—Ru1—C26—C25	−104.3 (3)
C1—P1—C6—C7	−174.7 (4)	C25—C26—C27—C23	1.0 (4)
N2—C6—C7—C8	56.7 (5)	Ru1—C26—C27—C23	−92.2 (3)
P1—C6—C7—C8	−128.3 (4)	C25—C26—C27—Ru1	93.3 (3)
N2—C6—C7—C10	−62.9 (5)	C22—C23—C27—C26	69.8 (4)
P1—C6—C7—C10	112.1 (4)	C24—C23—C27—C26	−34.0 (4)
N2—C6—C7—C9	177.3 (4)	C22—C23—C27—Ru1	−1.4 (3)
P1—C6—C7—C9	−7.7 (5)	C24—C23—C27—Ru1	−105.2 (3)
N3—N4—C11—C12	−176.7 (4)	N1—Ru1—C27—C26	153.0 (2)
Ru1—N4—C11—C12	−29.8 (18)	N4—Ru1—C27—C26	4.4 (3)
N3—N4—C11—P2	0.9 (4)	N2—Ru1—C27—C26	117.4 (2)
Ru1—N4—C11—P2	147.8 (15)	C21—Ru1—C27—C26	−65.4 (2)
C16—P2—C11—N4	−0.9 (3)	C22—Ru1—C27—C26	−103.3 (3)
C16—P2—C11—C12	176.5 (4)	N3—Ru1—C27—C26	−45.2 (10)
N4—C11—C12—C15	−36.6 (6)	N1—Ru1—C27—C23	−102.6 (2)
P2—C11—C12—C15	146.2 (4)	N4—Ru1—C27—C23	108.9 (3)
N4—C11—C12—C13	−156.6 (4)	N2—Ru1—C27—C23	−138.2 (2)
P2—C11—C12—C13	26.2 (5)	C21—Ru1—C27—C23	39.0 (2)
N4—C11—C12—C14	85.6 (5)	C26—Ru1—C27—C23	104.4 (3)
P2—C11—C12—C14	−91.6 (5)	C22—Ru1—C27—C23	1.1 (2)
N4—N3—C16—C17	180.0 (3)	N3—Ru1—C27—C23	59.2 (10)