Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Acetonitrile-κN){1,2-bis[bis(pentafluorophenyl)-phosphino]ethane-$\kappa^2 P,P$}(η^5-pentamethylcyclopentadienyl)ruthenium(II) hexafluorophosphate

Ronan M. Bellabarba, Mark Nieuwenhuyzen and Graham C. Saunders*

School of Chemistry, Queen's University Belfast, David Keir Building, Belfast BT9 5AG, Northern Ireland

Correspondence e-mail: g.saunders@qub.ac.uk

Received 26 April 2005
Accepted 24 June 2005
Online 16 July 2005

Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.006 Å
R factor = 0.047
wR factor = 0.109
Data-to-parameter ratio = 15.1

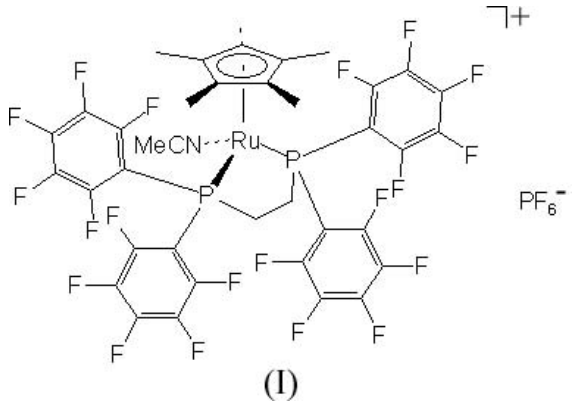
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The cation of the title salt, $[Ru(\eta^5\text{-}C_5Me_5)(NCMe)\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]PF_6$ or $[Ru(C_{10}H_{15})(C_{26}H_4F_{20}P_2)(C_2H_3N)]PF_6$, has contacts with three anions. One lies close to the pentamethylcyclopentadienyl ring, such that three F atoms of the anion are *ca* 3.5 Å from two of the ring methyl C atoms of the cation and there is one H···F distance shorter than the sum of the van der Waals radii.

Comment

Salts of the cation $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]^+$ have been found to undergo intramolecular dehydrofluorinative C–C reactions on thermolysis or in the presence of a proton sponge or fluoride, to yield $[\{\eta^5,\kappa P,\kappa P\text{-}C_5Me_4CH_2C_6F_4\text{-}2\text{-}P(C_6F_5)CH_2CH_2P(C_6F_5)_2\}RhCl]^+$ and then $[\{\eta^5,\kappa P,\kappa P\text{-}C_5Me_3[CH_2C_6F_4\text{-}2\text{-}P(C_6F_5)CH_2]\text{-}1,3\}RhCl]^+$ (Atherton *et al.*, 1996; Bellabarba *et al.*, 2001). The thermolysis is dependent on the solvent and the anion. The reaction for the tetrafluoroborate salt occurs only in polar protic solvents, such as ethanol, whereas for chloride, hexafluorophosphate and tetraphenylborate salts, the reaction also occurs readily in non-polar aprotic solvents, such as benzene (Atherton *et al.*, 1999).

The structure of $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]BF_4$ revealed that a tetrafluoroborate anion is positioned close to the pentamethylcyclopentadienyl ligand, such that three F atoms of the anion form a plane almost parallel (5.1° deviation) to the C_5 plane, with a separation between the two planes of *ca* 3.19 Å. The anion is displaced slightly from the (η^5-C_5Me_5)–Rh axis, giving rise to short F···H and F···C distances between the anion and the pentamethylcyclopentadienyl ligand of 2.4–2.7 and 3.1–3.3 Å, respectively (Atherton *et al.*, 1996). A similar positioning of the anion and cation is found in the related salts $[(\eta^5\text{-}C_5Me_5)IrCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]BF_4$ (Atherton *et al.*, 1996) and $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6H_3F_2\text{-}2,6)_2PCH_2CH_2P(C_6H_3F_2\text{-}2,6)_2\}]BF_4$ (Fawcett *et al.*, 1998). If $BF_4^-\cdots C_5Me_5$ interactions are present in aprotic solvents, then the absence of similar anion···C_5Me_5 interactions in the salts of the other anions may provide the basis for an explanation for the difference in reactivity. Of particular relevance is the salt of the hexafluorophosphate anion, which is the most similar to the tetrafluoroborate anion. These two anions comprise a periphery of F atoms, with equilateral triangular faces with edges of *ca* 2.1–2.3 Å (Allen *et al.*, 1987; Atherton *et al.*, 1996; Fawcett *et al.*, 1998). Unfortunately, crystals suitable for single-crystal X-ray diffraction studies of the non-tetrafluoroborate salts of $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]^+$ have been elusive. However, the structure of the title isoelectronic ruthenium salt, $[(\eta^5\text{-}C_5Me_5)Ru(NCMe)\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]PF_6$, (I), has now been determined and is presented here.



(I)

The structure of (I) (Fig. 1) reveals that the hexafluorophosphate anion does not adopt a similar position to that of the tetrafluoroborate anion in $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2\}]BF_4$ and $[(\eta^5\text{-}C_5Me_5)RhCl\{(C_6H_3F_2\text{-}2,6)_2PCH_2CH_2P(C_6H_3F_2\text{-}2,6)_2\}]BF_4$. The cation shows contacts to three anions which are shorter than the sum of the van der Waals radii of the respective atoms. One anion position is close to the pentamethylcyclopentadienyl ligand, such that there is one F···H distance shorter than the sum of the van der Waals radii (F36···H10*C* = 2.626 Å). The shortest inter-ion F···C(C_5Me_5) distances are between atoms F34 and C9 [3.493 (6) Å], F36 and C10 [3.564 (7) Å], and F32 and C10 [3.597 (7) Å]. However, for this anion, the shortest inter-ion F···C distance of 3.028 (6) Å is between atoms F36 and C2*S* of the acetonitrile. The distance between atoms C3*S* and F36 is 3.123 (5) Å, with F36···H3*S*2 = 2.609 Å, and that between atoms C3*S* and F34 is 3.396 (7) Å, with F34···H3*S*2 = 2.481 Å.

Another anion position gives three short contacts with a C_6F_5 ring (F32···F26*B* = 2.888 (6) Å, F32···C26*B* = 2.964 (6) Å and F32···C25*B* = 3.110 (6) Å) and a contact with a CH_2 H atom (F35···H2*A*2 = 2.633 Å). The third anion position gives a contact with a C_6F_5 ring (F31···C14*B* = 3.113 (7) Å), a CH_2 H atom (F33···H1*A*1 = 2.599 Å) and an acetonitrile H atom (F35···H3*S*1 = 2.428 Å).

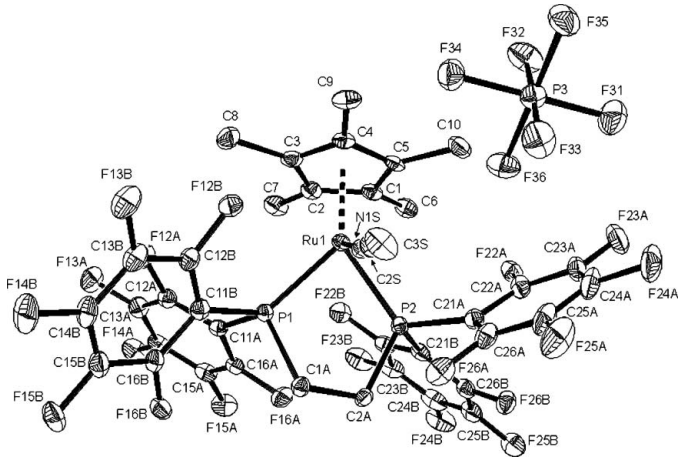


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Experimental

The title complex was obtained from the reaction of $[(\eta^5\text{-}C_5Me_5)Ru(NCMe)_3]PF_6$ with $(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$ in dichloromethane (10 ml) gave a yellow–green solution from which a small number of yellow crystals of (I) were obtained by cooling the reaction mixture to 273 K.

Crystal data

$[Ru(C_{10}H_{15})(C_{26}H_4F_{20}P_2)(C_2H_3N)]PF_6$	D_x = 1.874 Mg m^{-3}
M_r = 1180.55	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 5351 reflections
a = 12.5380 (9) Å	θ = 4–50°
b = 10.6157 (8) Å	μ = 0.64 mm^{-1}
c = 31.760 (2) Å	T = 153 (2) K
β = 98.062 (2)°	Needle, red
V = 4185.4 (5) Å^3	0.42 × 0.10 × 0.08 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	9479 independent reflections
	5575 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.098
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 27.5°
	$h = -16 \rightarrow 16$
T_{min} = 0.775, T_{max} = 0.951	$k = -13 \rightarrow 13$
37499 measured reflections	$l = -41 \rightarrow 40$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.109	$(\Delta/\sigma)_{max} < 0.001$
S = 0.94	$\Delta\rho_{max}$ = 0.67 e Å^{-3}
9479 reflections	$\Delta\rho_{min}$ = −0.83 e Å^{-3}
627 parameters	

H atoms were added in idealized positions and a riding model with fixed displacement parameters [U_{iso}(H) = 1.2U_{eq} of the parent atom (1.5U_{eq} for methyl H atoms].

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SHELXTL* (Bruker, 2001) and *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank the EPSRC for support.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Atherton, M. J., Fawcett, J., Holloway, J. H., Hope, E. G., Karaçar, A., Russell, D. R. & Saunders, G. C. (1996). *J. Chem. Soc. Dalton Trans.* pp. 3215–3220.

Atherton, M. J. Fawcett, J., Holloway, J. H., Hope, E. G., Russell, D. R. & Saunders G. C. (1999). *J. Organomet. Chem.* **582**, 163–172.

Bellabarba, R. M., Nieuwenhuyzen, M. & Saunders, G. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 512–514.

Bruker (2001). *SMART* (Version 5.622) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2002). *SAINT*. Version 6.36a. Bruker AXS Inc., Madison, Wisconsin, USA.

Fawcett, J., Friedrichs, S., Holloway, J. H., Hope, E. G., McKee, V., Nieuwenhuyzen, M., Russell, D. R. & Saunders, G. C. (1998). *J. Chem. Soc. Dalton Trans.* pp. 1477–1484.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supporting information

Acta Cryst. (2005). E**61**, m1588–m1589 [https://doi.org/10.1107/S1600536805020052]

(Acetonitrile-κN){1,2-bis[bis(pentafluorophenyl)phosphino]ethane-$\kappa^2 P,P$}(η^5-pentamethylcyclopentadienyl)ruthenium(II) hexafluorophosphate

Ronan M. Bellabarba, Mark Nieuwenhuyzen and Graham C. Saunders

(Acetonitrile-κN){1,2-bis[bis(pentafluorophenyl)phosphino]ethane- κ^2P,P}(η^5-pentamethylcyclopentadienyl)ruthenium(II) hexafluorophosphate

Crystal data

$[Ru(C_{10}H_{15})(C_{26}H_4F_{20}P_2)(C_2H_3N)]PF_6$
$M_r = 1180.55$
Monoclinic, $P2_1/n$
$a = 12.5380$ (9) Å
$b = 10.6157$ (8) Å
$c = 31.760$ (2) Å
$\beta = 98.062$ (2)°
$V = 4185.4$ (5) Å^3
$Z = 4$
$F(000) = 2320$
$D_x = 1.874$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5351 reflections
$\theta = 4$–50°
$\mu = 0.64$ mm^{-1}
$T = 153$ K
Needle, red
0.42 × 0.10 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.775$, $T_{max} = 0.951$
37499 measured reflections
9479 independent reflections
5575 reflections with $I > 2\sigma(I)$
$R_{int} = 0.098$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.3°$
$h = -16 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -41 \rightarrow 40$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.109$
$S = 0.94$
9479 reflections
627 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: geom and difmap for Me H
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ e Å^{-3}

Special details

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Ru1	0.11895 (3)	0.13441 (3)	0.386595 (10)	0.02476 (9)
C1	0.1383 (4)	0.0814 (4)	0.45544 (13)	0.0344 (10)
C2	0.0259 (3)	0.0940 (4)	0.44016 (13)	0.0338 (10)
C3	−0.0011 (3)	0.0004 (4)	0.40794 (13)	0.0334 (10)
C4	0.0929 (3)	−0.0680 (4)	0.40313 (13)	0.0326 (10)
C5	0.1810 (3)	−0.0172 (4)	0.43183 (13)	0.0310 (10)
C6	0.1987 (4)	0.1406 (4)	0.49471 (13)	0.0426 (11)
H6A	0.1538	0.2057	0.5053	0.064*
H6B	0.2653	0.1788	0.4878	0.064*
H6C	0.2163	0.0760	0.5166	0.064*
C7	−0.0529 (4)	0.1709 (4)	0.46049 (15)	0.0489 (13)
H7A	−0.0822	0.1200	0.4819	0.073*
H7B	−0.1117	0.1981	0.4388	0.073*
H7C	−0.0164	0.2451	0.4741	0.073*
C8	−0.1145 (4)	−0.0332 (5)	0.38813 (15)	0.0479 (13)
H8A	−0.1122	−0.0757	0.3609	0.072*
H8B	−0.1577	0.0437	0.3834	0.072*
H8C	−0.1470	−0.0895	0.4073	0.072*
C9	0.1016 (4)	−0.1771 (4)	0.37448 (15)	0.0471 (13)
H9A	0.1719	−0.1755	0.3644	0.071*
H9B	0.0444	−0.1721	0.3501	0.071*
H9C	0.0941	−0.2556	0.3901	0.071*
C10	0.2918 (4)	−0.0755 (4)	0.43991 (16)	0.0464 (12)
H10A	0.2859	−0.1630	0.4491	0.070*
H10B	0.3367	−0.0279	0.4622	0.070*
H10C	0.3247	−0.0734	0.4137	0.070*
P1	0.01571 (8)	0.27318 (10)	0.34249 (3)	0.0243 (2)
C11A	−0.0839 (3)	0.3702 (4)	0.36536 (11)	0.0253 (8)
C12A	−0.1899 (3)	0.3329 (4)	0.36497 (13)	0.0322 (10)
F12A	−0.22560 (18)	0.2244 (2)	0.34630 (8)	0.0442 (7)
C13A	−0.2644 (3)	0.4012 (4)	0.38383 (14)	0.0368 (11)
F13A	−0.3655 (2)	0.3605 (3)	0.38198 (9)	0.0552 (8)
C14A	−0.2331 (4)	0.5123 (4)	0.40430 (14)	0.0396 (11)
F14A	−0.3026 (2)	0.5787 (3)	0.42356 (9)	0.0577 (8)
C15A	−0.1292 (4)	0.5530 (4)	0.40570 (13)	0.0361 (11)

F15A	−0.0969 (2)	0.6597 (2)	0.42586 (9)	0.0553 (8)
C16A	−0.0572 (3)	0.4830 (4)	0.38573 (13)	0.0305 (10)
F16A	0.04305 (19)	0.5298 (2)	0.38709 (8)	0.0406 (6)
C11B	−0.0692 (3)	0.2292 (4)	0.29221 (12)	0.0284 (9)
C12B	−0.0825 (3)	0.1088 (4)	0.27615 (13)	0.0314 (10)
F12B	−0.03597 (19)	0.0094 (2)	0.29806 (8)	0.0391 (6)
C13B	−0.1424 (4)	0.0806 (5)	0.23738 (14)	0.0396 (11)
F13B	−0.1493 (2)	−0.0382 (3)	0.22318 (8)	0.0530 (7)
C14B	−0.1957 (4)	0.1761 (5)	0.21398 (14)	0.0428 (12)
F14B	−0.2558 (2)	0.1507 (3)	0.17677 (8)	0.0633 (9)
C15B	−0.1852 (3)	0.2980 (5)	0.22826 (14)	0.0396 (11)
F15B	−0.2360 (2)	0.3917 (3)	0.20523 (8)	0.0529 (7)
C16B	−0.1228 (3)	0.3231 (4)	0.26627 (13)	0.0304 (10)
F16B	−0.11495 (18)	0.4451 (2)	0.27840 (7)	0.0378 (6)
C1A	0.1091 (3)	0.3770 (4)	0.31799 (12)	0.0283 (9)
H1A1	0.1290	0.3347	0.2924	0.034*
H1A2	0.0708	0.4558	0.3086	0.034*
C2A	0.2128 (3)	0.4109 (4)	0.34770 (12)	0.0291 (9)
H2A1	0.2033	0.4932	0.3613	0.035*
H2A2	0.2731	0.4190	0.3308	0.035*
P2	0.24670 (8)	0.28992 (10)	0.38917 (3)	0.0261 (2)
C21A	0.3767 (3)	0.2212 (4)	0.38038 (14)	0.0314 (10)
C22A	0.4452 (3)	0.1681 (4)	0.41375 (15)	0.0371 (11)
F22A	0.4254 (2)	0.1869 (2)	0.45387 (8)	0.0457 (7)
C23A	0.5333 (4)	0.0951 (4)	0.40833 (19)	0.0478 (13)
F23A	0.5957 (2)	0.0465 (3)	0.44162 (11)	0.0735 (10)
C24A	0.5545 (4)	0.0730 (5)	0.3679 (2)	0.0575 (16)
F24A	0.6387 (2)	−0.0004 (3)	0.36203 (13)	0.0825 (11)
C25A	0.4908 (4)	0.1249 (5)	0.33344 (18)	0.0527 (14)
F25A	0.5121 (2)	0.1039 (3)	0.29398 (11)	0.0774 (10)
C26A	0.4045 (4)	0.1966 (4)	0.34006 (15)	0.0394 (11)
F26A	0.3439 (2)	0.2439 (3)	0.30516 (8)	0.0481 (7)
C21B	0.2839 (3)	0.4011 (4)	0.43330 (13)	0.0311 (10)
C22B	0.2068 (4)	0.4407 (4)	0.45785 (13)	0.0349 (10)
F22B	0.1089 (2)	0.3850 (2)	0.45389 (8)	0.0421 (6)
C23B	0.2221 (4)	0.5382 (4)	0.48622 (14)	0.0423 (12)
F23B	0.1433 (3)	0.5716 (3)	0.50852 (9)	0.0625 (8)
C24B	0.3180 (5)	0.6007 (5)	0.49196 (15)	0.0523 (14)
F24B	0.3349 (3)	0.6951 (3)	0.52025 (9)	0.0691 (9)
C25B	0.3983 (4)	0.5671 (4)	0.46856 (15)	0.0453 (13)
F25B	0.4913 (2)	0.6298 (3)	0.47281 (9)	0.0606 (8)
C26B	0.3801 (4)	0.4689 (4)	0.43942 (14)	0.0356 (11)
F26B	0.45955 (19)	0.4423 (2)	0.41727 (9)	0.0454 (7)
N1S	0.1761 (3)	0.0731 (3)	0.33285 (10)	0.0268 (8)
C2S	0.2011 (3)	0.0292 (4)	0.30337 (14)	0.0310 (10)
C3S	0.2301 (4)	−0.0259 (5)	0.26464 (14)	0.0494 (13)
H3S1	0.1680	−0.0223	0.2422	0.074*
H3S2	0.2515	−0.1139	0.2699	0.074*

H3S3	0.2902	0.0213	0.2557	0.074*
P3	0.40845 (10)	0.71325 (12)	0.33113 (4)	0.0385 (3)
F31	0.5311 (2)	0.7346 (3)	0.34824 (10)	0.0687 (9)
F32	0.3840 (2)	0.6759 (3)	0.37704 (9)	0.0632 (9)
F33	0.4288 (2)	0.7504 (3)	0.28423 (9)	0.0632 (8)
F34	0.2840 (2)	0.6917 (3)	0.31325 (9)	0.0564 (8)
F35	0.4325 (2)	0.5692 (3)	0.32137 (9)	0.0564 (8)
F36	0.3821 (2)	0.8565 (2)	0.34084 (9)	0.0568 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.02723 (18)	0.02367 (17)	0.02360 (17)	0.00175 (15)	0.00433 (12)	0.00038 (15)
C1	0.044 (3)	0.029 (2)	0.031 (2)	0.003 (2)	0.010 (2)	0.0092 (19)
C2	0.039 (3)	0.035 (3)	0.031 (2)	0.004 (2)	0.015 (2)	0.0086 (19)
C3	0.040 (3)	0.027 (2)	0.035 (2)	−0.004 (2)	0.009 (2)	0.0088 (19)
C4	0.041 (3)	0.025 (2)	0.033 (2)	−0.003 (2)	0.010 (2)	0.0072 (19)
C5	0.040 (3)	0.023 (2)	0.031 (2)	0.0073 (19)	0.0073 (19)	0.0087 (18)
C6	0.057 (3)	0.040 (3)	0.030 (2)	0.006 (2)	0.006 (2)	0.005 (2)
C7	0.057 (3)	0.047 (3)	0.049 (3)	0.010 (2)	0.027 (3)	0.015 (2)
C8	0.041 (3)	0.052 (3)	0.052 (3)	−0.011 (2)	0.010 (2)	0.010 (3)
C9	0.066 (3)	0.025 (2)	0.052 (3)	−0.007 (2)	0.013 (3)	−0.003 (2)
C10	0.048 (3)	0.041 (3)	0.048 (3)	0.010 (2)	0.002 (2)	0.012 (2)
P1	0.0239 (5)	0.0253 (6)	0.0234 (5)	0.0000 (4)	0.0026 (4)	−0.0004 (4)
C11A	0.031 (2)	0.025 (2)	0.0198 (19)	−0.0004 (19)	0.0047 (16)	0.0007 (17)
C12A	0.035 (2)	0.032 (3)	0.029 (2)	−0.0006 (19)	0.0029 (19)	0.0005 (19)
F12A	0.0308 (14)	0.0447 (16)	0.0590 (18)	−0.0066 (12)	0.0125 (12)	−0.0120 (13)
C13A	0.032 (3)	0.047 (3)	0.034 (3)	0.004 (2)	0.011 (2)	0.005 (2)
F13A	0.0368 (16)	0.0644 (19)	0.069 (2)	0.0035 (14)	0.0224 (14)	−0.0053 (16)
C14A	0.051 (3)	0.040 (3)	0.031 (2)	0.021 (2)	0.017 (2)	0.005 (2)
F14A	0.0669 (19)	0.0585 (19)	0.0531 (18)	0.0256 (16)	0.0272 (15)	−0.0024 (15)
C15A	0.054 (3)	0.027 (2)	0.028 (2)	0.007 (2)	0.006 (2)	−0.0041 (19)
F15A	0.076 (2)	0.0374 (17)	0.0552 (18)	0.0038 (14)	0.0175 (15)	−0.0154 (13)
C16A	0.034 (2)	0.027 (2)	0.030 (2)	0.0031 (19)	0.0033 (19)	0.0042 (18)
F16A	0.0422 (15)	0.0306 (14)	0.0495 (16)	−0.0077 (12)	0.0074 (12)	−0.0119 (12)
C11B	0.025 (2)	0.035 (2)	0.025 (2)	−0.0009 (19)	0.0061 (17)	0.0017 (19)
C12B	0.027 (2)	0.035 (3)	0.033 (2)	−0.0014 (19)	0.0066 (18)	−0.0035 (19)
F12B	0.0419 (15)	0.0299 (14)	0.0446 (15)	−0.0034 (11)	0.0023 (12)	−0.0075 (12)
C13B	0.037 (3)	0.045 (3)	0.037 (3)	−0.009 (2)	0.007 (2)	−0.018 (2)
F13B	0.0536 (17)	0.0562 (18)	0.0483 (17)	−0.0147 (14)	0.0044 (14)	−0.0241 (14)
C14B	0.036 (3)	0.063 (3)	0.027 (2)	−0.006 (2)	−0.005 (2)	−0.011 (2)
F14B	0.0589 (18)	0.091 (2)	0.0336 (15)	−0.0083 (17)	−0.0157 (13)	−0.0137 (16)
C15B	0.030 (2)	0.056 (3)	0.031 (2)	0.000 (2)	0.002 (2)	0.008 (2)
F15B	0.0505 (17)	0.071 (2)	0.0330 (15)	0.0090 (15)	−0.0099 (12)	0.0093 (14)
C16B	0.027 (2)	0.036 (3)	0.027 (2)	−0.0011 (18)	0.0010 (18)	−0.0025 (19)
F16B	0.0409 (15)	0.0375 (15)	0.0333 (14)	0.0045 (12)	−0.0008 (11)	0.0050 (11)
C1A	0.026 (2)	0.033 (2)	0.027 (2)	−0.0014 (19)	0.0031 (17)	−0.0009 (19)
C2A	0.025 (2)	0.032 (2)	0.030 (2)	−0.0053 (18)	0.0023 (17)	0.0031 (18)

P2	0.0255 (6)	0.0279 (6)	0.0242 (5)	0.0011 (5)	0.0006 (4)	−0.0017 (5)
C21A	0.028 (2)	0.028 (2)	0.037 (2)	−0.0032 (18)	0.0010 (19)	−0.0030 (19)
C22A	0.033 (2)	0.032 (3)	0.043 (3)	−0.0020 (19)	−0.006 (2)	−0.012 (2)
F22A	0.0494 (16)	0.0422 (15)	0.0407 (16)	0.0090 (13)	−0.0100 (13)	−0.0002 (13)
C23A	0.029 (3)	0.032 (3)	0.077 (4)	0.003 (2)	−0.009 (3)	−0.009 (3)
F23A	0.0492 (18)	0.0526 (19)	0.108 (3)	0.0183 (15)	−0.0249 (18)	−0.0076 (18)
C24A	0.025 (3)	0.050 (3)	0.098 (5)	−0.004 (2)	0.013 (3)	−0.028 (3)
F24A	0.0333 (17)	0.066 (2)	0.151 (3)	0.0068 (15)	0.0211 (19)	−0.034 (2)
C25A	0.034 (3)	0.061 (4)	0.068 (4)	−0.008 (3)	0.024 (3)	−0.027 (3)
F25A	0.063 (2)	0.092 (3)	0.087 (2)	−0.0090 (18)	0.0463 (18)	−0.037 (2)
C26A	0.033 (3)	0.046 (3)	0.040 (3)	−0.006 (2)	0.008 (2)	−0.010 (2)
F26A	0.0518 (17)	0.0596 (18)	0.0351 (15)	−0.0093 (14)	0.0142 (13)	−0.0070 (13)
C21B	0.038 (3)	0.024 (2)	0.030 (2)	0.0091 (18)	−0.0005 (19)	0.0008 (18)
C22B	0.048 (3)	0.031 (3)	0.025 (2)	0.003 (2)	0.001 (2)	0.0052 (19)
F22B	0.0463 (16)	0.0408 (16)	0.0423 (15)	0.0066 (13)	0.0167 (12)	−0.0004 (12)
C23B	0.067 (3)	0.035 (3)	0.025 (2)	0.008 (3)	0.007 (2)	−0.002 (2)
F23B	0.098 (2)	0.0507 (18)	0.0421 (17)	0.0231 (17)	0.0206 (16)	−0.0075 (14)
C24B	0.085 (4)	0.035 (3)	0.031 (3)	0.009 (3)	−0.013 (3)	−0.007 (2)
F24B	0.120 (3)	0.0408 (17)	0.0405 (17)	0.0050 (18)	−0.0106 (17)	−0.0196 (14)
C25B	0.052 (3)	0.036 (3)	0.040 (3)	−0.001 (2)	−0.020 (2)	−0.003 (2)
F25B	0.0635 (19)	0.0414 (16)	0.066 (2)	−0.0095 (15)	−0.0272 (15)	−0.0095 (15)
C26B	0.038 (3)	0.033 (3)	0.032 (2)	0.003 (2)	−0.009 (2)	−0.002 (2)
F26B	0.0336 (15)	0.0391 (16)	0.0623 (18)	−0.0032 (12)	0.0020 (13)	−0.0102 (13)
N1S	0.0259 (18)	0.0254 (19)	0.0288 (19)	0.0000 (15)	0.0032 (15)	−0.0003 (15)
C2S	0.032 (2)	0.030 (2)	0.030 (2)	0.0037 (19)	0.0029 (19)	0.0013 (19)
C3S	0.067 (3)	0.055 (3)	0.029 (3)	0.018 (3)	0.012 (2)	−0.004 (2)
P3	0.0419 (7)	0.0380 (7)	0.0364 (7)	−0.0014 (6)	0.0082 (5)	−0.0006 (6)
F31	0.0456 (18)	0.079 (2)	0.076 (2)	−0.0089 (16)	−0.0086 (16)	−0.0006 (18)
F32	0.090 (2)	0.0595 (19)	0.0432 (17)	0.0183 (17)	0.0217 (16)	0.0120 (14)
F33	0.064 (2)	0.084 (2)	0.0467 (18)	0.0111 (17)	0.0239 (15)	0.0129 (16)
F34	0.0431 (17)	0.0541 (18)	0.072 (2)	0.0021 (14)	0.0070 (14)	−0.0095 (15)
F35	0.0586 (19)	0.0450 (17)	0.065 (2)	0.0121 (14)	0.0082 (15)	−0.0106 (15)
F36	0.078 (2)	0.0362 (16)	0.0549 (18)	−0.0010 (15)	0.0062 (15)	−0.0038 (14)

Geometric parameters (Å, º)

Ru1—N1S	2.048 (3)	C12B—C13B	1.382 (6)
Ru1—C5	2.224 (4)	C13B—F13B	1.339 (5)
Ru1—C2	2.235 (4)	C13B—C14B	1.374 (6)
Ru1—C1	2.238 (4)	C14B—F14B	1.337 (5)
Ru1—C3	2.244 (4)	C14B—C15B	1.371 (6)
Ru1—C4	2.247 (4)	C15B—F15B	1.342 (5)
Ru1—P2	2.2936 (11)	C15B—C16B	1.370 (6)
Ru1—P1	2.3019 (11)	C16B—F16B	1.351 (5)
C1—C2	1.432 (6)	C1A—C2A	1.538 (5)
C1—C5	1.434 (6)	C1A—H1A1	0.9900
C1—C6	1.503 (6)	C1A—H1A2	0.9900
C2—C3	1.433 (6)	C2A—P2	1.846 (4)

C2—C7	1.496 (6)	C2A—H2A1	0.9900
C3—C4	1.410 (6)	C2A—H2A2	0.9900
C3—C8	1.515 (6)	P2—C21B	1.841 (4)
C4—C5	1.435 (6)	P2—C21A	1.842 (4)
C4—C9	1.486 (6)	C21A—C22A	1.387 (6)
C5—C10	1.510 (6)	C21A—C26A	1.398 (6)
C6—H6A	0.9800	C22A—F22A	1.347 (5)
C6—H6B	0.9800	C22A—C23A	1.380 (6)
C6—H6C	0.9800	C23A—F23A	1.328 (5)
C7—H7A	0.9800	C23A—C24A	1.367 (7)
C7—H7B	0.9800	C24A—F24A	1.347 (5)
C7—H7C	0.9800	C24A—C25A	1.377 (8)
C8—H8A	0.9800	C25A—F25A	1.336 (6)
C8—H8B	0.9800	C25A—C26A	1.363 (6)
C8—H8C	0.9800	C26A—F26A	1.349 (5)
C9—H9A	0.9800	C21B—C22B	1.389 (6)
C9—H9B	0.9800	C21B—C26B	1.395 (6)
C9—H9C	0.9800	C22B—F22B	1.352 (5)
C10—H10A	0.9800	C22B—C23B	1.368 (6)
C10—H10B	0.9800	C23B—F23B	1.341 (5)
C10—H10C	0.9800	C23B—C24B	1.363 (7)
P1—C11A	1.844 (4)	C24B—F24B	1.342 (5)
P1—C11B	1.851 (4)	C24B—C25B	1.379 (7)
P1—C1A	1.857 (4)	C25B—F25B	1.333 (5)
C11A—C16A	1.380 (5)	C25B—C26B	1.391 (6)
C11A—C12A	1.385 (5)	C26B—F26B	1.328 (5)
C12A—F12A	1.343 (4)	N1S—C2S	1.129 (5)
C12A—C13A	1.384 (6)	C2S—C3S	1.453 (6)
C13A—F13A	1.333 (5)	C3S—H3S1	0.9800
C13A—C14A	1.377 (6)	C3S—H3S2	0.9800
C14A—F14A	1.334 (5)	C3S—H3S3	0.9800
C14A—C15A	1.367 (6)	P3—F31	1.574 (3)
C15A—F15A	1.335 (5)	P3—F32	1.582 (3)
C15A—C16A	1.389 (6)	P3—F36	1.595 (3)
C16A—F16A	1.347 (5)	P3—F33	1.596 (3)
C11B—C12B	1.378 (6)	P3—F35	1.598 (3)
C11B—C16B	1.403 (5)	P3—F34	1.600 (3)
C12B—F12B	1.351 (5)		

N1S—Ru1—C5	100.19 (14)	F14A—C14A—C13A	120.5 (4)
N1S—Ru1—C2	148.71 (15)	C15A—C14A—C13A	119.6 (4)
C5—Ru1—C2	62.76 (15)	F15A—C15A—C14A	120.5 (4)
N1S—Ru1—C1	137.05 (14)	F15A—C15A—C16A	120.0 (4)
C5—Ru1—C1	37.50 (14)	C14A—C15A—C16A	119.5 (4)
C2—Ru1—C1	37.33 (15)	F16A—C16A—C11A	119.9 (4)
N1S—Ru1—C3	112.23 (14)	F16A—C16A—C15A	116.8 (4)
C5—Ru1—C3	62.26 (15)	C11A—C16A—C15A	123.3 (4)
C2—Ru1—C3	37.31 (15)	C12B—C11B—C16B	114.7 (4)

C1—Ru1—C3	62.03 (16)	C12B—C11B—P1	125.4 (3)
N1S—Ru1—C4	88.30 (14)	C16B—C11B—P1	119.8 (3)
C5—Ru1—C4	37.45 (15)	F12B—C12B—C11B	120.9 (4)
C2—Ru1—C4	61.98 (16)	F12B—C12B—C13B	115.5 (4)
C1—Ru1—C4	62.01 (16)	C11B—C12B—C13B	123.6 (4)
C3—Ru1—C4	36.61 (15)	F13B—C13B—C14B	120.6 (4)
N1S—Ru1—P2	86.22 (9)	F13B—C13B—C12B	120.4 (4)
C5—Ru1—P2	108.57 (11)	C14B—C13B—C12B	119.0 (4)
C2—Ru1—P2	123.19 (12)	F14B—C14B—C15B	119.9 (4)
C1—Ru1—P2	99.58 (12)	F14B—C14B—C13B	120.0 (4)
C3—Ru1—P2	160.09 (11)	C15B—C14B—C13B	120.0 (4)
C4—Ru1—P2	143.49 (11)	F15B—C15B—C16B	120.3 (4)
N1S—Ru1—P1	85.92 (9)	F15B—C15B—C14B	120.2 (4)
C5—Ru1—P1	166.43 (11)	C16B—C15B—C14B	119.5 (4)
C2—Ru1—P1	106.06 (11)	F16B—C16B—C15B	116.6 (4)
C1—Ru1—P1	136.89 (11)	F16B—C16B—C11B	120.3 (3)
C3—Ru1—P1	104.24 (11)	C15B—C16B—C11B	123.1 (4)
C4—Ru1—P1	131.80 (11)	C2A—C1A—P1	114.2 (3)
P2—Ru1—P1	83.77 (4)	C2A—C1A—H1A1	108.7
C2—C1—C5	108.2 (4)	P1—C1A—H1A1	108.7
C2—C1—C6	126.8 (4)	C2A—C1A—H1A2	108.7
C5—C1—C6	123.9 (4)	P1—C1A—H1A2	108.7
C2—C1—Ru1	71.3 (2)	H1A1—C1A—H1A2	107.6
C5—C1—Ru1	70.7 (2)	C1A—C2A—P2	111.4 (3)
C6—C1—Ru1	132.8 (3)	C1A—C2A—H2A1	109.3
C1—C2—C3	107.4 (4)	P2—C2A—H2A1	109.3
C1—C2—C7	125.7 (4)	C1A—C2A—H2A2	109.3
C3—C2—C7	125.7 (4)	P2—C2A—H2A2	109.3
C1—C2—Ru1	71.4 (2)	H2A1—C2A—H2A2	108.0
C3—C2—Ru1	71.7 (2)	C21B—P2—C21A	103.55 (19)
C7—C2—Ru1	132.1 (3)	C21B—P2—C2A	96.07 (18)
C4—C3—C2	108.5 (4)	C21A—P2—C2A	106.68 (19)
C4—C3—C8	125.7 (4)	C21B—P2—Ru1	126.11 (15)
C2—C3—C8	125.0 (4)	C21A—P2—Ru1	109.75 (13)
C4—C3—Ru1	71.8 (2)	C2A—P2—Ru1	112.68 (13)
C2—C3—Ru1	71.0 (2)	C22A—C21A—C26A	114.6 (4)
C8—C3—Ru1	130.9 (3)	C22A—C21A—P2	120.8 (3)
C3—C4—C5	108.5 (4)	C26A—C21A—P2	123.5 (3)
C3—C4—C9	127.0 (4)	F22A—C22A—C23A	117.3 (4)
C5—C4—C9	124.5 (4)	F22A—C22A—C21A	119.1 (4)
C3—C4—Ru1	71.6 (2)	C23A—C22A—C21A	123.6 (5)
C5—C4—Ru1	70.4 (2)	F23A—C23A—C24A	120.7 (5)
C9—C4—Ru1	125.2 (3)	F23A—C23A—C22A	120.7 (5)
C1—C5—C4	107.2 (4)	C24A—C23A—C22A	118.5 (5)
C1—C5—C10	127.8 (4)	F24A—C24A—C23A	119.3 (5)
C4—C5—C10	124.2 (4)	F24A—C24A—C25A	120.0 (5)
C1—C5—Ru1	71.8 (2)	C23A—C24A—C25A	120.7 (5)
C4—C5—Ru1	72.1 (2)	F25A—C25A—C26A	120.4 (5)

C10—C5—Ru1	129.5 (3)	F25A—C25A—C24A	120.6 (5)
C1—C6—H6A	109.5	C26A—C25A—C24A	119.0 (5)
C1—C6—H6B	109.5	F26A—C26A—C25A	116.6 (4)
H6A—C6—H6B	109.5	F26A—C26A—C21A	120.0 (4)
C1—C6—H6C	109.5	C25A—C26A—C21A	123.4 (5)
H6A—C6—H6C	109.5	C22B—C21B—C26B	114.9 (4)
H6B—C6—H6C	109.5	C22B—C21B—P2	120.0 (3)
C2—C7—H7A	109.5	C26B—C21B—P2	124.0 (3)
C2—C7—H7B	109.5	F22B—C22B—C23B	115.7 (4)
H7A—C7—H7B	109.5	F22B—C22B—C21B	120.6 (4)
C2—C7—H7C	109.5	C23B—C22B—C21B	123.7 (5)
H7A—C7—H7C	109.5	F23B—C23B—C24B	120.1 (4)
H7B—C7—H7C	109.5	F23B—C23B—C22B	120.1 (5)
C3—C8—H8A	109.5	C24B—C23B—C22B	119.8 (5)
C3—C8—H8B	109.5	F24B—C24B—C23B	120.4 (5)
H8A—C8—H8B	109.5	F24B—C24B—C25B	119.7 (5)
C3—C8—H8C	109.5	C23B—C24B—C25B	119.9 (4)
H8A—C8—H8C	109.5	F25B—C25B—C24B	120.7 (4)
H8B—C8—H8C	109.5	F25B—C25B—C26B	120.0 (5)
C4—C9—H9A	109.5	C24B—C25B—C26B	119.2 (5)
C4—C9—H9B	109.5	F26B—C26B—C25B	116.5 (4)
H9A—C9—H9B	109.5	F26B—C26B—C21B	121.0 (4)
C4—C9—H9C	109.5	C25B—C26B—C21B	122.5 (5)
H9A—C9—H9C	109.5	C2S—N1S—Ru1	173.1 (3)
H9B—C9—H9C	109.5	N1S—C2S—C3S	178.2 (5)
C5—C10—H10A	109.5	C2S—C3S—H3S1	109.5
C5—C10—H10B	109.5	C2S—C3S—H3S2	109.5
H10A—C10—H10B	109.5	H3S1—C3S—H3S2	109.5
C5—C10—H10C	109.5	C2S—C3S—H3S3	109.5
H10A—C10—H10C	109.5	H3S1—C3S—H3S3	109.5
H10B—C10—H10C	109.5	H3S2—C3S—H3S3	109.5
C11A—P1—C11B	98.14 (17)	F31—P3—F32	91.37 (18)
C11A—P1—C1A	109.58 (18)	F31—P3—F36	90.86 (17)
C11B—P1—C1A	96.20 (17)	F32—P3—F36	89.35 (16)
C11A—P1—Ru1	118.14 (12)	F31—P3—F33	90.61 (17)
C11B—P1—Ru1	124.75 (14)	F32—P3—F33	178.02 (18)
C1A—P1—Ru1	107.53 (13)	F36—P3—F33	90.52 (16)
C16A—C11A—C12A	114.9 (4)	F31—P3—F35	90.16 (17)
C16A—C11A—P1	122.4 (3)	F32—P3—F35	90.29 (16)
C12A—C11A—P1	122.6 (3)	F36—P3—F35	178.93 (17)
F12A—C12A—C13A	115.9 (4)	F33—P3—F35	89.80 (17)
F12A—C12A—C11A	120.6 (4)	F31—P3—F34	179.43 (18)
C13A—C12A—C11A	123.5 (4)	F32—P3—F34	89.20 (17)
F13A—C13A—C14A	120.3 (4)	F36—P3—F34	89.24 (16)
F13A—C13A—C12A	120.5 (4)	F33—P3—F34	88.83 (16)
C14A—C13A—C12A	119.2 (4)	F35—P3—F34	89.75 (15)
F14A—C14A—C15A	119.9 (4)		