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cis-Chloridobis(4,4′-dimethyl-2,2′-bipyridine)-nitrosylruthenium(II) bis(hexafluorophosphate)

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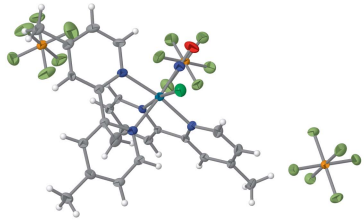
CCDC reference: 1557221

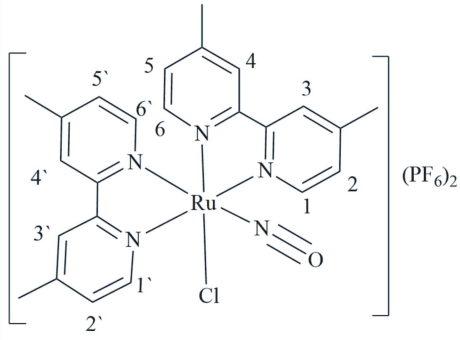
Structural data: full structural data are available from iucrdata.iucr.org

In the cation of the title complex, $[RuCl(NO)(C_{12}H_{12}N_2)_2](PF_6)_2$, the central Ru^{II} ion is sixfold coordinated by a chloride ion and a nitrosyl ligand, which are *cis* to one another, and by four N atoms of two 4,4′-dimethyl-2,2′-bipyridine ligands, in a slightly distorted octahedral geometry. One of the PF_6^- anions is located in a general position, while the other is composed of two half PF_6^- anions located on twofold rotation axes. The crystal packing is dominated by C—H···F hydrogen bonds, leading to the formation of a three-dimensional supramolecular structure. There are also C—H···Cl hydrogen bonds present.

3D view



Chemical scheme



Structure description

Ruthenium nitrosyl complexes have attracted significant attention over the last two decades, mainly due to their interesting photoreactivity properties. Ruthenium nitrosyl complexes can either induce photochromism (Schaniel *et al.*, 2007*b*) or NO photorelease (Rose & Mascharak, 2008). In the first case, photoisomerization along the ruthenium–nitrosyl bond (Ru—NO/Ru—ON) is observed in the solid state when the compounds are irradiated in the blue region (Schaniel *et al.*, 2007*a*; Cormary *et al.*, 2009, 2012). It offers technological applications as optical high-capacity storage devices (Imlau *et al.*, 1999). In the latter case, irradiation at room temperature in the UV–visible region of solutions of ruthenium nitrosyl complexes gives rise to NO photorelease (Tfouni *et al.*, 2003; Fry & Mascharak, 2011; Akl *et al.*, 2014). This is very appealing because the nitric oxide has a significant role in various biological processes. It shows an ability to induce apoptosis and is involved in blood-pressure control, and also has antimicrobial activity (Hirst & Robson, 2007). Ruthenium nitrosyl complexes based on bipyridine ligands are of great interest because of their photoreactive properties (Togniolo *et al.*, 2001). In the search for new systems, we have synthesized the title complex and report herein its crystal structure.

The cation of the title complex has a central Ru^{II} ion which is sixfold coordinated by a chloride ion and a nitrosyl ligand (Fig. 1), which are *cis* to one another, and by four N atoms of two 4,4′-dimethyl-2,2′-bipyridine ligands, in a slightly distorted octahedral

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Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···F2^{i}	0.95	2.54	3.328 (5)	141
C5—H5···F9^{ii}	0.95	2.51	3.310 (5)	141
C8—H8···Cl1	0.95	2.75	3.353 (4)	122
C15—H15···Cl1^{iii}	0.95	2.80	3.660 (4)	152
C18—H18···F11^{iv}	0.95	2.49	3.215 (5)	133
C18—H18···F4^{v}	0.95	2.32	3.064 (5)	135
C19—H19···F13^{vi}	0.95	2.54	3.208 (5)	127
C21—H21···F8^{vii}	0.95	2.53	3.433 (5)	159
C22—H22···F1^{ii}	0.95	2.32	3.253 (5)	166
C25—H25A···F4^{ii}	0.98	2.50	3.358 (6)	147
C25—H25C···F9	0.98	2.39	3.228 (6)	143

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+\frac{1}{2}, y, -z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $x, y+1, z$; (v) $x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (vi) $-x+\frac{1}{2}, y+1, -z+\frac{3}{2}$; (vii) $x, y-1, z$.

geometry. One of the PF_6^- anions is located in a general position, while the other is composed of two half PF_6^- anions located on twofold rotation axes. The Ru1—N1—O1 angle is 178.6 (3)°, which is close to 180°, in agreement with the Enemark–Feltham notation for {RuNO}6 with Ru^{II}—NO^+ and with the characteristic range of (NO) absorption around 1940 cm^{-1} (Lahiri & Kaim, 2010).

The crystal packing is dominated by C—H···F hydrogen bonds, leading to the formation of a three-dimensional supramolecular structure (Table 1 and Fig. 2). There are also C—H···Cl hydrogen bonds present (Table 1).

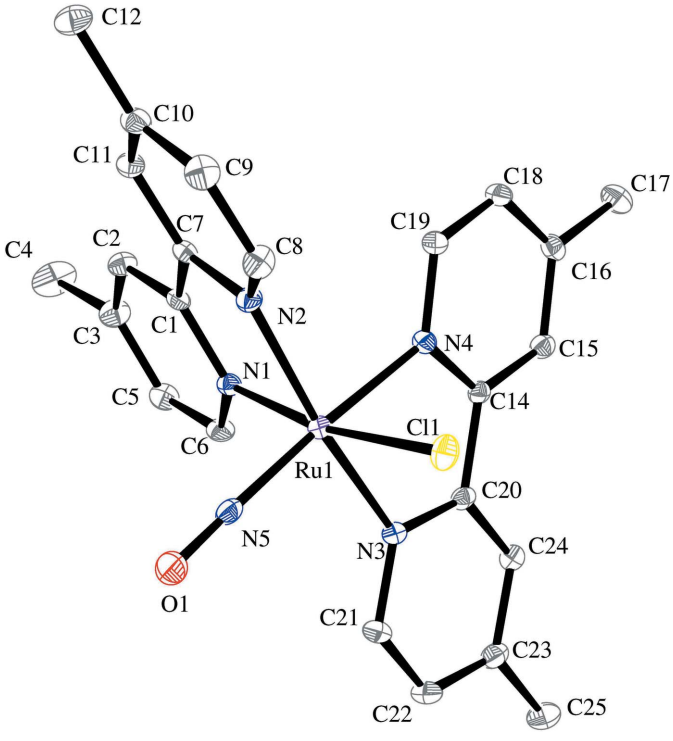

Figure 1
The molecular structure of the $[RuCl(NO)(dimethyl\text{-}2,2'\text{-}bipyridine)_2]^{2+}$ cation of the title complex, showing the atom labelling and 30% probability displacement ellipsoids.

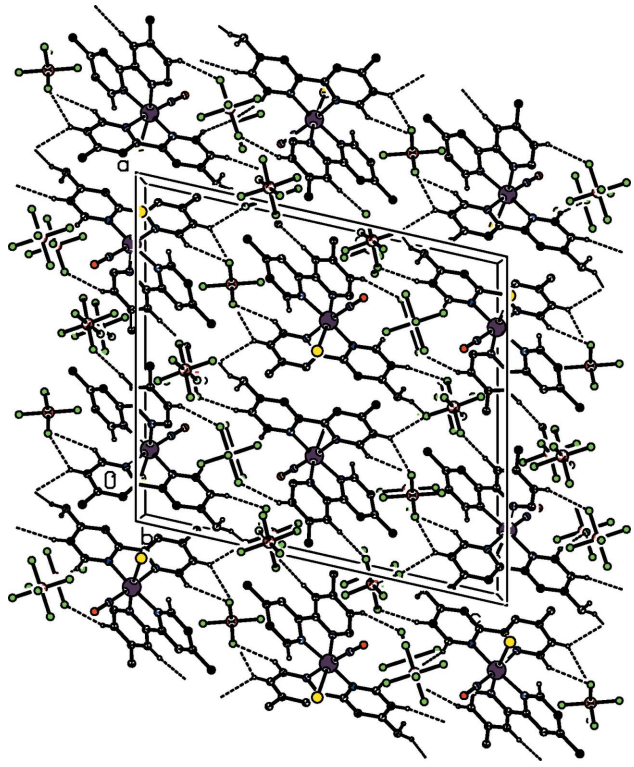

Figure 2
A view along the b axis of the crystal packing of the title complex. The C—H···F hydrogen bonds are shown as dashed lines (see Table 1) and only the H atoms involved in these interactions have been included.

Synthesis and crystallization

Synthesis of $[RuCl_2(4,4'\text{-dimethyl-}2,2'\text{-bipyridine})_2]$, (I)

DMF (15 ml) was bubbled with argon for 15 min. Lithium chloride (406 mg, 9.6 mmol) was dissolved in DMF (5 ml) with stirring for 15 min. Ruthenium chloride, (III) (250 mg, 1.2 mmol), was dissolved in DMF (5 ml). 4,4′-Dimethyl-2,2′-bipyridine (406 mg, 2.4 mmol) was dissolved in DMF (5 ml). The lithium chloride solution was added to the ruthenium chloride solution *via* cannula under argon. The 4,4′-dimethyl-2,2′-bipyridine solution was added to the above solution dropwise *via* cannula under argon. The reaction mixture was refluxed for 6 h at 413 K. The volume of the solution was decreased to half using a rotary evaporator. Acetone (50 ml) was added to the mixture and it was placed in an ice bath for 4 h. The dark-red precipitate that formed was filtered off, washed with distilled water and dried (yield 392 mg, 60.4%). Analysis calculated for $C_{24}H_{24}Cl_2N_4Ru$: C 53.34, H 4.48, N 10.37%; found: C 53.44, H 4.53, N 10.30%. IR (KBr) cm^{-1}: 3041 (C—H aromatic), 2910 (C—H aliphatic), 1615 (C═N), 1446 (C═C), 825 (C—H rock), 551 (C—H rock).

Synthesis of $[Ru(NO_2)_2(4,4'\text{-dimethyl-}2,2'\text{-bipyridine})_2]$, (II)

Compound (**I**) (305 mg, 0.56 mmol) was dissolved in distilled water (20 ml) and the red solution was refluxed for 30 min, then filtered. $NaNO_2$ (156.5 mg, 2.24 mmol) dissolved in distilled water (5 ml) and was added to the filtrate and the reaction mixture was refluxed for 1 h at 373 K. The mixture

Table 2
Experimental details.

Crystal data	
Chemical formula	$[RuCl(NO)(C_{12}H_{12}N_2)_2](PF_6)_2$
M_r	824.94
Crystal system, space group	Monoclinic, *P*2/*n*
Temperature (K)	173
a, *b*, *c* (Å)	18.6926 (12), 8.1573 (5), 20.3841 (13)
β (°)	102.828 (3)
V (Å^3)	3030.6 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.82
Crystal size (mm)	0.15 × 0.08 × 0.02
Data collection	
Diffractometer	Bruker Kappa APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.847, 0.989
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	127079, 6196, 5805
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.108, 1.32
No. of reflections	6196
No. of parameters	421
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.19, −0.73

Computer programs: *APEX2* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick 2008), *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *WinGX* (Farrugia, 2012).

was placed in an ice bath for 1 h then filtered. The brown precipitate obtained was washed with distilled water and dried (yield 203 mg, 64.5%). Analysis calculated for $C_{24}H_{24}N_6O_4Ru$: C 51.33, H 4.31, N 14.97%; found: C 51.41, H 4.37, N 14.84%. ^{1}H NMR (300 MHz, DMSO-d_6, 298 K): δ 9.50 (2H1,1′, *d*, *J* = 5.85 Hz), 8.51 (2H3,3′, *s*), 8.41 (2H4,4′, *s*), 7.64 (2H2,2′, *dd*, *J* = 5.71, 1.18 Hz), 7.32 (2H6,6′, *d*, *J* = 5.74 Hz), 7.08 (2H5,5′, *dd*, *J* = 5.72, 1.00 Hz), 2.61 (6H, *s*, 2CH$_3$), 2.38 (6H, *s*, 2CH$_3$). IR (KBr) cm^{-1}: 3071 (C—H aromatic), 2920 (C—H aliphatic), 1616 (C═N), 1447 (C═C), 1320 $(NO_2)_{asy}$, 1272 $(NO_2)_{sy}$, 1039, 825, 551 due to (C—H) deformation.

Synthesis of [RuCl(NO)(dimethyl-2,2′-bipyridine)$_2$](PF$_6$)$_2$

Compound (**II**) (170 mg, 0.30 mmol) was dissolved in HCl (24 ml, 37%). The reaction mixture was refluxed for 1 h at 373 K and then left to cool to room temperature. NH_4PF_6 (195 mg, 1.2 mmol) dissolved in distilled water (2 ml) was added to the reaction mixture. The orange precipitate that formed was filtered off, washed with distilled water, then diethyl ether and dried (yield 154 mg, 62%). IR (KBr) cm^{-1}: 3088 (C—H aromatic), 2905 (C—H aliphatic), 1940 (N═O), 1618 (C═N), 1428(C═C), 835 (P—F).

Analysis calculated for $C24H_{24}ClF_{12}N_5OP_2Ru$: C 34.94, H 2.93, N 8.49%; found: C 34.84, H 2.85, N 8.40%. ^{1}H NMR (300 MHz, DMSO-d_6, 298 K): δ 9.23 (1H1, *d*, *J* = 5.99 Hz), 9.11 (1H1‘, *d*, *J* = 5.92 Hz), 9.00 (1H3, *d*, 1.88), 8.92 (1H3‘, *d*, *J* = 1.76 Hz), 8.86 (1H4, *d*, *J* = 1.86 Hz), 8.82 (1H4‘, *d*, *J* = 1.82 Hz), 8.06 (1H2, *d*, *J* = 6.11 Hz), 8.00 (1H2‘, *d*, *J* = 6.22 Hz), 7.82 (1H6, *d*, *J* = 5.94 Hz), 7.54 (1H5, *dd*, *J* = 6.10, 1.77 Hz), 7.43 (1H5‘, *J* = 5.96, 1.80 Hz), 7.26 (1H6‘, *d*, *J* = 6.02 Hz), 2.76 (3H, *s*, CH$_3$), 2.74 (3H, *s*, CH$_3$), 2.56 (3H, *s*, CH$_3$), 2.53 (3H, *s*, CH$_3$). UV–Vis in acetonitrile at 298 K, λ nm (ε M^{-1} cm^{-1}): 295 (24273), 323 (16554). Yellow plate-like crystals of the title complex were obtained by slow diffusion of diethyl ether into an acetone solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2017). **2**, x171013 [https://doi.org/10.1107/S2414314617010136]

cis-Chloridobis(4,4′-dimethyl-2,2′-bipyridine)nitrosylruthenium(II) bis(hexafluorophosphate)

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cis-Chloridobis(4,4′-dimethyl-2,2′-bipyridine)nitrosylruthenium(II) bis(hexafluorophosphate)

Crystal data

$[RuCl(NO)(C_{12}H_{12}N_2)_2](PF_6)_2$
M_r = 824.94
Monoclinic, *P*2/*n*
Hall symbol: -P 2yac
a = 18.6926 (12) Å
b = 8.1573 (5) Å
c = 20.3841 (13) Å
β = 102.828 (3)°
V = 3030.6 (3) Å^3
Z = 4
F(000) = 1640
D_x = 1.808 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9501 reflections
θ = 2.7–29.2°
μ = 0.82 mm^{-1}
T = 173 K
Plate, yellow
0.15 × 0.08 × 0.02 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: microsource
Graphite monochromator
ω–*φ* scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.847, T_{max} = 0.989
127079 measured reflections
6196 independent reflections
5805 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.4°, θ_{min} = 2.2°
h = −23→23
k = −10→10
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.108
S = 1.32
6196 reflections
421 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 13.0961P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.73 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	0.295920 (17)	0.32238 (4)	0.480073 (15)	0.01836 (9)
F12	0.22407 (15)	−0.3588 (3)	0.66961 (12)	0.0334 (6)
Cl1	0.39303 (6)	0.13222 (13)	0.51121 (6)	0.0283 (2)
P2	0.25	0.8827 (2)	0.25	0.0294 (4)
P1	0.03457 (6)	0.28288 (15)	0.36131 (6)	0.0278 (3)
F8	0.25	1.0779 (5)	0.25	0.0410 (10)
F7	0.25	0.6866 (7)	0.25	0.094 (2)
F4	−0.00952 (17)	0.3316 (4)	0.28780 (14)	0.0471 (8)
F2	−0.03599 (15)	0.1995 (4)	0.37854 (14)	0.0390 (7)
F6	0.01399 (17)	0.4528 (4)	0.39047 (17)	0.0491 (8)
F1	0.10714 (15)	0.3653 (4)	0.34570 (13)	0.0402 (7)
F3	0.08241 (15)	0.2338 (4)	0.43490 (12)	0.0373 (7)
F5	0.05578 (15)	0.1119 (4)	0.33278 (14)	0.0375 (6)
F10	0.29501 (17)	0.8830 (5)	0.32644 (14)	0.0504 (8)
F9	0.32424 (18)	0.8816 (5)	0.22524 (15)	0.0562 (10)
N1	0.21674 (17)	0.5015 (4)	0.46897 (16)	0.0175 (7)
N2	0.24267 (18)	0.2538 (4)	0.55481 (16)	0.0197 (7)
N3	0.35224 (18)	0.4258 (4)	0.41381 (16)	0.0210 (7)
F11	0.30891 (14)	−0.2203 (3)	0.74521 (13)	0.0292 (6)
C1	0.1747 (2)	0.4963 (5)	0.51577 (18)	0.0177 (8)
C2	0.1216 (2)	0.6127 (5)	0.5158 (2)	0.0232 (8)
H2	0.0939	0.6094	0.5496	0.028*
C3	0.1077 (3)	0.7348 (6)	0.4675 (2)	0.0294 (10)
C4	0.0469 (3)	0.8564 (6)	0.4652 (3)	0.0465 (14)
H4A	0.0058	0.8029	0.4792	0.07*
H4B	0.0304	0.898	0.4192	0.07*
H4C	0.0648	0.9478	0.4956	0.07*
C5	0.1509 (2)	0.7352 (5)	0.4195 (2)	0.0257 (9)
H5	0.1428	0.8156	0.3849	0.031*
C6	0.2047 (2)	0.6205 (5)	0.42230 (19)	0.0227 (8)
H6	0.2346	0.625	0.3902	0.027*
C7	0.1877 (2)	0.3556 (5)	0.56216 (18)	0.0175 (8)
C8	0.2559 (2)	0.1187 (5)	0.5928 (2)	0.0264 (9)
H8	0.2944	0.0477	0.5873	0.032*
C9	0.2157 (2)	0.0788 (5)	0.6395 (2)	0.0283 (9)

H9	0.2259	−0.0193	0.665	0.034*
C10	0.1600 (2)	0.1834 (5)	0.6493 (2)	0.0237 (9)
C11	0.1467 (2)	0.3241 (5)	0.60945 (19)	0.0205 (8)
H11	0.1093	0.3984	0.6148	0.025*
C12	0.1158 (3)	0.1481 (6)	0.7011 (2)	0.0304 (10)
H12A	0.0719	0.2173	0.6926	0.046*
H12B	0.1456	0.1715	0.7461	0.046*
H12C	0.1013	0.0324	0.6984	0.046*
N4	0.36093 (17)	0.4951 (4)	0.54082 (16)	0.0193 (7)
C14	0.4023 (2)	0.5919 (5)	0.50946 (19)	0.0174 (8)
C15	0.4448 (2)	0.7176 (5)	0.5430 (2)	0.0216 (8)
H15	0.4735	0.7832	0.5201	0.026*
C16	0.4458 (2)	0.7483 (5)	0.6106 (2)	0.0224 (8)
C17	0.4901 (3)	0.8868 (6)	0.6478 (2)	0.0315 (10)
H17A	0.4575	0.9785	0.6519	0.047*
H17B	0.5266	0.9228	0.623	0.047*
H17C	0.5151	0.8496	0.6928	0.047*
C18	0.4032 (2)	0.6491 (5)	0.6418 (2)	0.0238 (9)
H18	0.4024	0.6663	0.6877	0.029*
C19	0.3620 (2)	0.5251 (5)	0.60590 (19)	0.0224 (8)
H19	0.3332	0.458	0.6281	0.027*
C20	0.3978 (2)	0.5509 (5)	0.43805 (19)	0.0193 (8)
C21	0.3468 (2)	0.3789 (6)	0.3495 (2)	0.0281 (10)
H21	0.3156	0.2896	0.3323	0.034*
C22	0.3850 (2)	0.4564 (6)	0.3079 (2)	0.0301 (10)
H22	0.3796	0.42	0.2628	0.036*
C23	0.4310 (2)	0.5868 (6)	0.3311 (2)	0.0262 (9)
C24	0.4372 (2)	0.6333 (5)	0.3981 (2)	0.0192 (8)
H24	0.4684	0.7217	0.4163	0.023*
C25	0.4720 (3)	0.6781 (6)	0.2869 (2)	0.0346 (11)
H25A	0.477	0.6081	0.2491	0.052*
H25B	0.5208	0.7081	0.3131	0.052*
H25C	0.4448	0.7776	0.2697	0.052*
N5	0.24250 (18)	0.1847 (5)	0.42268 (17)	0.0234 (7)
O1	0.20981 (18)	0.0961 (5)	0.38590 (18)	0.0408 (9)
P3	0.25	−0.35977 (18)	0.75	0.0220 (3)
F13	0.19066 (17)	−0.4991 (3)	0.75495 (14)	0.0386 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01756 (16)	0.02110 (17)	0.01676 (15)	−0.00024 (13)	0.00453 (11)	−0.00084 (13)
F12	0.0493 (16)	0.0329 (15)	0.0176 (12)	−0.0077 (12)	0.0071 (11)	−0.0033 (11)
Cl1	0.0214 (5)	0.0274 (5)	0.0362 (6)	0.0041 (4)	0.0063 (4)	0.0019 (4)
P2	0.0402 (9)	0.0288 (9)	0.0226 (8)	0	0.0140 (7)	0
P1	0.0281 (6)	0.0351 (7)	0.0197 (5)	−0.0093 (5)	0.0043 (4)	−0.0013 (5)
F8	0.047 (2)	0.029 (2)	0.044 (2)	0	0.0035 (19)	0
F7	0.159 (7)	0.030 (3)	0.094 (5)	0	0.030 (4)	0

F4	0.0472 (17)	0.054 (2)	0.0311 (15)	−0.0047 (15)	−0.0106 (13)	0.0051 (14)
F2	0.0322 (14)	0.0447 (17)	0.0429 (16)	−0.0089 (13)	0.0139 (12)	−0.0072 (14)
F6	0.0451 (17)	0.0423 (18)	0.060 (2)	−0.0054 (15)	0.0108 (15)	−0.0110 (16)
F1	0.0362 (15)	0.0566 (19)	0.0266 (13)	−0.0175 (14)	0.0047 (11)	0.0056 (13)
F3	0.0426 (16)	0.0464 (17)	0.0205 (13)	−0.0140 (13)	0.0021 (11)	0.0029 (12)
F5	0.0359 (15)	0.0420 (16)	0.0345 (15)	−0.0084 (13)	0.0076 (12)	−0.0046 (13)
F10	0.0526 (19)	0.076 (2)	0.0237 (14)	0.0191 (17)	0.0101 (13)	0.0109 (15)
F9	0.0478 (18)	0.090 (3)	0.0360 (16)	0.0260 (18)	0.0211 (14)	0.0060 (17)
N1	0.0207 (16)	0.0155 (16)	0.0168 (15)	−0.0032 (13)	0.0049 (13)	−0.0017 (13)
N2	0.0193 (16)	0.0222 (17)	0.0179 (16)	0.0005 (14)	0.0050 (13)	0.0031 (14)
N3	0.0197 (16)	0.0265 (19)	0.0179 (16)	−0.0011 (14)	0.0063 (13)	−0.0027 (14)
F11	0.0336 (14)	0.0253 (13)	0.0307 (13)	−0.0043 (11)	0.0115 (11)	−0.0003 (11)
C1	0.0210 (18)	0.0181 (19)	0.0131 (17)	−0.0046 (15)	0.0020 (14)	−0.0005 (15)
C2	0.027 (2)	0.022 (2)	0.022 (2)	0.0036 (17)	0.0086 (16)	0.0009 (16)
C3	0.034 (2)	0.027 (2)	0.028 (2)	0.0041 (19)	0.0089 (19)	0.0046 (18)
C4	0.063 (4)	0.031 (3)	0.051 (3)	0.025 (3)	0.024 (3)	0.017 (2)
C5	0.032 (2)	0.022 (2)	0.022 (2)	0.0008 (18)	0.0042 (17)	0.0041 (17)
C6	0.030 (2)	0.024 (2)	0.0153 (18)	−0.0055 (17)	0.0072 (16)	0.0050 (16)
C7	0.0203 (18)	0.0155 (19)	0.0151 (17)	−0.0018 (15)	0.0008 (14)	−0.0016 (14)
C8	0.026 (2)	0.023 (2)	0.030 (2)	0.0057 (17)	0.0066 (18)	0.0071 (18)
C9	0.034 (2)	0.021 (2)	0.029 (2)	0.0014 (18)	0.0058 (18)	0.0093 (18)
C10	0.029 (2)	0.024 (2)	0.0184 (19)	−0.0072 (18)	0.0054 (16)	0.0012 (17)
C11	0.0236 (19)	0.0180 (19)	0.0200 (19)	0.0002 (16)	0.0052 (15)	−0.0023 (16)
C12	0.040 (3)	0.028 (2)	0.027 (2)	−0.002 (2)	0.0149 (19)	0.0069 (18)
N4	0.0185 (16)	0.0229 (17)	0.0160 (15)	0.0018 (14)	0.0025 (13)	0.0005 (13)
C14	0.0161 (18)	0.0183 (19)	0.0181 (18)	0.0048 (15)	0.0043 (14)	0.0005 (15)
C15	0.0215 (19)	0.022 (2)	0.022 (2)	0.0022 (16)	0.0065 (16)	0.0000 (16)
C16	0.022 (2)	0.021 (2)	0.022 (2)	0.0030 (17)	−0.0006 (16)	−0.0015 (16)
C17	0.037 (3)	0.028 (2)	0.027 (2)	−0.002 (2)	0.0007 (19)	−0.0045 (19)
C18	0.024 (2)	0.031 (2)	0.0157 (18)	0.0029 (18)	0.0038 (15)	−0.0011 (17)
C19	0.022 (2)	0.029 (2)	0.0169 (18)	−0.0008 (17)	0.0048 (15)	0.0014 (17)
C20	0.0195 (19)	0.0193 (19)	0.0200 (19)	0.0061 (16)	0.0062 (15)	0.0006 (16)
C21	0.029 (2)	0.037 (3)	0.019 (2)	−0.0050 (19)	0.0058 (17)	−0.0088 (18)
C22	0.033 (2)	0.040 (3)	0.019 (2)	0.002 (2)	0.0089 (18)	−0.0045 (19)
C23	0.025 (2)	0.032 (2)	0.023 (2)	0.0056 (18)	0.0102 (17)	0.0041 (18)
C24	0.0215 (19)	0.0122 (18)	0.024 (2)	0.0010 (15)	0.0065 (16)	0.0010 (15)
C25	0.040 (3)	0.040 (3)	0.027 (2)	−0.001 (2)	0.017 (2)	0.003 (2)
N5	0.0200 (16)	0.0264 (19)	0.0266 (18)	0.0014 (15)	0.0111 (14)	−0.0050 (16)
O1	0.0331 (18)	0.045 (2)	0.047 (2)	−0.0143 (16)	0.0149 (16)	−0.0238 (18)
P3	0.0354 (8)	0.0145 (7)	0.0174 (7)	0	0.0087 (6)	0
F13	0.0607 (19)	0.0245 (14)	0.0369 (15)	−0.0149 (13)	0.0240 (14)	−0.0079 (12)

Geometric parameters (Å, º)

Ru1—N5	1.763 (4)	C8—H8	0.95
Ru1—N1	2.056 (3)	C9—C10	1.394 (6)
Ru1—N3	2.068 (3)	C9—H9	0.95
Ru1—N2	2.073 (3)	C10—C11	1.395 (6)

Ru1—N4	2.080 (3)	C10—C12	1.506 (6)
Ru1—Cl1	2.3648 (11)	C11—H11	0.95
F12—P3	1.602 (2)	C12—H12A	0.98
P2—F9	1.578 (3)	C12—H12B	0.98
P2—F9^{i}	1.578 (3)	C12—H12C	0.98
P2—F8	1.593 (4)	N4—C19	1.345 (5)
P2—F10^{i}	1.598 (3)	N4—C14	1.360 (5)
P2—F10	1.598 (3)	C14—C15	1.382 (6)
P2—F7	1.599 (6)	C14—C20	1.478 (5)
P1—F6	1.589 (3)	C15—C16	1.397 (6)
P1—F2	1.591 (3)	C15—H15	0.95
P1—F4	1.592 (3)	C16—C18	1.384 (6)
P1—F5	1.595 (3)	C16—C17	1.503 (6)
P1—F1	1.608 (3)	C17—H17A	0.98
P1—F3	1.617 (3)	C17—H17B	0.98
N1—C6	1.343 (5)	C17—H17C	0.98
N1—C1	1.365 (5)	C18—C19	1.379 (6)
N2—C8	1.337 (5)	C18—H18	0.95
N2—C7	1.355 (5)	C19—H19	0.95
N3—C21	1.348 (5)	C20—C24	1.387 (5)
N3—C20	1.350 (5)	C21—C22	1.377 (6)
F11—P3	1.601 (3)	C21—H21	0.95
C1—C2	1.373 (6)	C22—C23	1.384 (7)
C1—C7	1.473 (5)	C22—H22	0.95
C2—C3	1.384 (6)	C23—C24	1.397 (6)
C2—H2	0.95	C23—C25	1.502 (6)
C3—C5	1.400 (6)	C24—H24	0.95
C3—C4	1.502 (6)	C25—H25A	0.98
C4—H4A	0.98	C25—H25B	0.98
C4—H4B	0.98	C25—H25C	0.98
C4—H4C	0.98	N5—O1	1.120 (5)
C5—C6	1.365 (6)	P3—F11^{ii}	1.601 (3)
C5—H5	0.95	P3—F12^{ii}	1.602 (2)
C6—H6	0.95	P3—F13	1.607 (3)
C7—C11	1.383 (5)	P3—F13^{ii}	1.607 (3)
C8—C9	1.377 (6)		

N5—Ru1—N1	95.26 (14)	N2—C8—H8	118.8
N5—Ru1—N3	97.01 (14)	C9—C8—H8	118.8
N1—Ru1—N3	95.54 (13)	C8—C9—C10	119.5 (4)
N5—Ru1—N2	91.27 (15)	C8—C9—H9	120.2
N1—Ru1—N2	79.70 (13)	C10—C9—H9	120.2
N3—Ru1—N2	170.83 (14)	C9—C10—C11	117.6 (4)
N5—Ru1—N4	175.15 (15)	C9—C10—C12	121.7 (4)
N1—Ru1—N4	84.00 (13)	C11—C10—C12	120.7 (4)
N3—Ru1—N4	78.32 (13)	C7—C11—C10	120.2 (4)
N2—Ru1—N4	93.30 (13)	C7—C11—H11	119.9
N5—Ru1—Cl1	92.77 (12)	C10—C11—H11	119.9

N1—Ru1—Cl1	170.58 (9)	C10—C12—H12A	109.5
N3—Ru1—Cl1	88.35 (10)	C10—C12—H12B	109.5
N2—Ru1—Cl1	95.22 (10)	H12A—C12—H12B	109.5
N4—Ru1—Cl1	88.42 (9)	C10—C12—H12C	109.5
F9—P2—F9^{i}	179.4 (3)	H12A—C12—H12C	109.5
F9—P2—F8	90.31 (16)	H12B—C12—H12C	109.5
F9^{i}—P2—F8	90.31 (16)	C19—N4—C14	118.2 (3)
F9—P2—F10^{i}	89.89 (16)	C19—N4—Ru1	126.1 (3)
F9^{i}—P2—F10^{i}	90.11 (16)	C14—N4—Ru1	115.5 (2)
F8—P2—F10^{i}	89.90 (15)	N4—C14—C15	121.5 (4)
F9—P2—F10	90.11 (16)	N4—C14—C20	114.7 (3)
F9^{i}—P2—F10	89.89 (16)	C15—C14—C20	123.7 (4)
F8—P2—F10	89.90 (15)	C14—C15—C16	120.1 (4)
F10^{i}—P2—F10	179.8 (3)	C14—C15—H15	120
F9—P2—F7	89.69 (16)	C16—C15—H15	120
F9^{i}—P2—F7	89.69 (16)	C18—C16—C15	117.7 (4)
F8—P2—F7	180.000 (2)	C18—C16—C17	121.1 (4)
F10^{i}—P2—F7	90.10 (15)	C15—C16—C17	121.2 (4)
F10—P2—F7	90.10 (15)	C16—C17—H17A	109.5
F6—P1—F2	90.51 (17)	C16—C17—H17B	109.5
F6—P1—F4	90.95 (19)	H17A—C17—H17B	109.5
F2—P1—F4	91.90 (17)	C16—C17—H17C	109.5
F6—P1—F5	179.40 (19)	H17A—C17—H17C	109.5
F2—P1—F5	89.54 (16)	H17B—C17—H17C	109.5
F4—P1—F5	89.65 (17)	C19—C18—C16	119.8 (4)
F6—P1—F1	89.67 (18)	C19—C18—H18	120.1
F2—P1—F1	178.51 (17)	C16—C18—H18	120.1
F4—P1—F1	89.57 (16)	N4—C19—C18	122.8 (4)
F5—P1—F1	90.26 (16)	N4—C19—H19	118.6
F6—P1—F3	89.92 (17)	C18—C19—H19	118.6
F2—P1—F3	90.25 (15)	N3—C20—C24	121.7 (4)
F4—P1—F3	177.67 (17)	N3—C20—C14	115.1 (3)
F5—P1—F3	89.47 (16)	C24—C20—C14	123.2 (4)
F1—P1—F3	88.28 (15)	N3—C21—C22	122.1 (4)
C6—N1—C1	119.0 (3)	N3—C21—H21	119
C6—N1—Ru1	126.5 (3)	C22—C21—H21	119
C1—N1—Ru1	114.4 (3)	C21—C22—C23	120.7 (4)
C8—N2—C7	119.2 (3)	C21—C22—H22	119.6
C8—N2—Ru1	126.2 (3)	C23—C22—H22	119.6
C7—N2—Ru1	114.5 (3)	C22—C23—C24	116.9 (4)
C21—N3—C20	118.4 (4)	C22—C23—C25	122.5 (4)
C21—N3—Ru1	125.5 (3)	C24—C23—C25	120.6 (4)
C20—N3—Ru1	116.1 (3)	C20—C24—C23	120.3 (4)
N1—C1—C2	120.5 (4)	C20—C24—H24	119.9
N1—C1—C7	115.7 (3)	C23—C24—H24	119.9
C2—C1—C7	123.8 (4)	C23—C25—H25A	109.5
C1—C2—C3	121.3 (4)	C23—C25—H25B	109.5
C1—C2—H2	119.4	H25A—C25—H25B	109.5

C3—C2—H2	119.4	C23—C25—H25C	109.5
C2—C3—C5	116.9 (4)	H25A—C25—H25C	109.5
C2—C3—C4	121.3 (4)	H25B—C25—H25C	109.5
C5—C3—C4	121.8 (4)	O1—N5—Ru1	178.6 (3)
C3—C4—H4A	109.5	F11—P3—F11^{ii}	89.5 (2)
C3—C4—H4B	109.5	F11—P3—F12	89.43 (14)
H4A—C4—H4B	109.5	F11^{ii}—P3—F12	90.16 (14)
C3—C4—H4C	109.5	F11—P3—F12^{ii}	90.16 (14)
H4A—C4—H4C	109.5	F11^{ii}—P3—F12^{ii}	89.43 (14)
H4B—C4—H4C	109.5	F12—P3—F12^{ii}	179.4 (2)
C6—C5—C3	120.2 (4)	F11—P3—F13	179.73 (16)
C6—C5—H5	119.9	F11^{ii}—P3—F13	90.28 (14)
C3—C5—H5	119.9	F12—P3—F13	90.66 (14)
N1—C6—C5	122.1 (4)	F12^{ii}—P3—F13	89.75 (15)
N1—C6—H6	119	F11—P3—F13^{ii}	90.28 (14)
C5—C6—H6	119	F11^{ii}—P3—F13^{ii}	179.73 (17)
N2—C7—C11	121.0 (4)	F12—P3—F13^{ii}	89.75 (15)
N2—C7—C1	115.4 (3)	F12^{ii}—P3—F13^{ii}	90.66 (14)
C11—C7—C1	123.6 (4)	F13—P3—F13^{ii}	90.0 (2)
N2—C8—C9	122.4 (4)		

Symmetry codes: (i) −*x*+1/2, *y*, −*z*+1/2; (ii) −*x*+1/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···F2^{iii}	0.95	2.54	3.328 (5)	141
C5—H5···F9^{i}	0.95	2.51	3.310 (5)	141
C8—H8···Cl1	0.95	2.75	3.353 (4)	122
C15—H15···Cl1^{iv}	0.95	2.80	3.660 (4)	152
C18—H18···F11^{v}	0.95	2.49	3.215 (5)	133
C18—H18···F4^{vi}	0.95	2.32	3.064 (5)	135
C19—H19···F13^{vii}	0.95	2.54	3.208 (5)	127
C21—H21···F8^{viii}	0.95	2.53	3.433 (5)	159
C22—H22···F1^{i}	0.95	2.32	3.253 (5)	166
C25—H25*A*···F4^{i}	0.98	2.50	3.358 (6)	147
C25—H25*C*···F9	0.98	2.39	3.228 (6)	143

Symmetry codes: (i) −*x*+1/2, *y*, −*z*+1/2; (iii) −*x*, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, *y*+1, *z*; (vi) *x*+1/2, −*y*+1, *z*+1/2; (vii) −*x*+1/2, *y*+1, −*z*+3/2; (viii) *x*, *y*−1, *z*.