Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of acetonitrile[η^6-1-methyl-4-(1-methylethyl)benzene][1-(pyrimidin-2-yl)-3*H*-indol-1-ium-2-yl-κ^2N,C]ruthenium(II) bis(hexafluoridoantimonate)

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Received 12 August 2015
Accepted 7 September 2015

Edited by H. Ishida, Okayama University, Japan

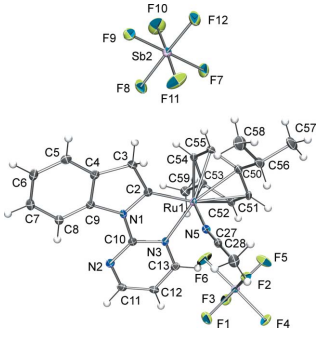
Keywords: crystal structure; cyclometalated Ru^{II}; pyrimidyl-3*H*-indole; *para*-cymene; C—H···F hydrogen bonds

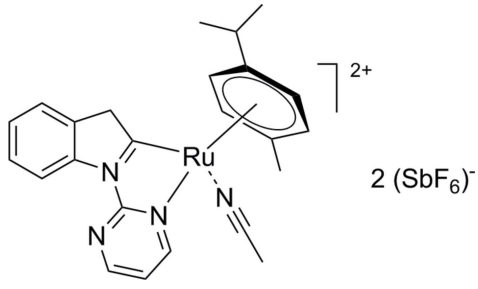
CCDC reference: 1027878
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $[Ru(C_{10}H_{14})(C_{12}H_9N_3)(CH_3CN)][SbF_6]_2$, the ruthenium(II) cation is η^6-coordinated by the *para*-cymene ligand with a Ru–centroid(η^6-benzene) distance of 1.746 (2) Å. Furthermore, ruthenium coordinations to the C and N atoms of the pyrimidyl indole ligand are found to be 1.986 (4) and 2.082 (3) Å, respectively. The typical piano-stool coordination environment is saturated with an acetonitrile solvent molecule with a Ru—N distance of 2.044 (3) Å. The indolyl ligand is protonated at the C3 position with the N=C imine bond length appropriate to that of related 3*H*-indole-based complexes. In the crystal, the complex cation is linked to the SbF_6^- ions through weak C—H···F hydrogen bonds.

1. Chemical context

Cyclometalated ruthenium compounds are well known catalytic intermediates in the C—H activation of various substrates (Arockiam *et al.*, 2012; Li *et al.*, 2012; Ferrer Flegeau *et al.*, 2011). In a recent study on oxidative Ru-catalysed heteroarene C—H arylation (Wang *et al.*, 2015; Ackermann & Lygin, 2011), we demonstrated that [{$RuCl_2$(*p*-cymene)}$_2$] in the presence of $AgSbF_6$ selectively ruthenates the C2—H bond of *N*-pyrimidine-substituted pyrroles and indoles (Sollert *et al.*, 2015). We concluded that in our catalytic system, the resulting ruthenacyclic species likely act as precursors rather than on-cycle intermediates. In the course of our studies we observed the unusual formation of the title complex, which shows protonation at the C3 position. The title compound and related cyclometalated ruthenium complexes are shown schematically in Fig. 1.





2. Structural commentary

In the title compound (Fig. 2), the ruthenium(II) cation is coordinated in an η^6 fashion by a *para*-cymene unit. The Ru—

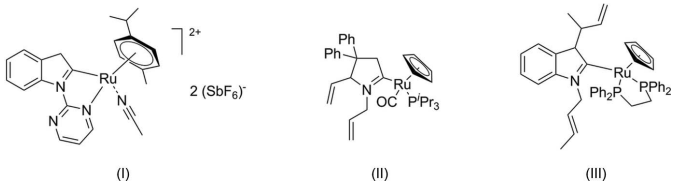


Figure 1
The title compound (I) and related cyclometalated ruthenium complexes (II) (Sollert *et al.*, 2015) and (III) (Chiang *et al.*, 2010).

$C_{p\text{-cymene}}$ distances range from 2.197 (4) to 2.298 (4) Å. The centroid of the *para*-cymene benzene ring (*Cg*) shows an Ru1—*Cg* distance of 1.746 (2) Å. Furthermore, ruthenium coordinations to C2 and N3 of the pyrimidyl indole are found to be 1.986 (4) and 2.082 (3) Å, respectively. The coordination environment is saturated with one acetonitrile solvent molecule, with an Ru1—N5 distance of 2.044 (3) Å. The least-squares planes of the 3*H*-indole ring system [r.m.s. deviation = 0.026 (4) Å] and the pyrimidine heterocycle [r.m.s. deviation = 0.013 (4) Å] are almost co-planar, making a dihedral angle of 2.6 (2)°. The Ru atom deviates by only 0.056 (1) Å from the 3*H*-indole plane. The 3*H*-indole shows a clear C2—N1 double bond of 1.345 (5) Å in the typical range for this class of compounds. The coordinating acetonitrile solvent molecule shows slight deviation from a linear arrangement [C27—N5—Ru1 = 170.4 (3)°].

3. Supramolecular features

The packing allows no direct interaction of equivalent ruthenium complexes. The crystal packing shows a complex pattern in which two crystallographically independent SbF_6^- counter-ions occupy a void formed by symmetry-equivalent metal complexes. C—H hydrogen bonds of the pyrimidylindole and *para*-cymene ligands with the SbF_6^- ions mainly account for the observed packing pattern (Table 1).

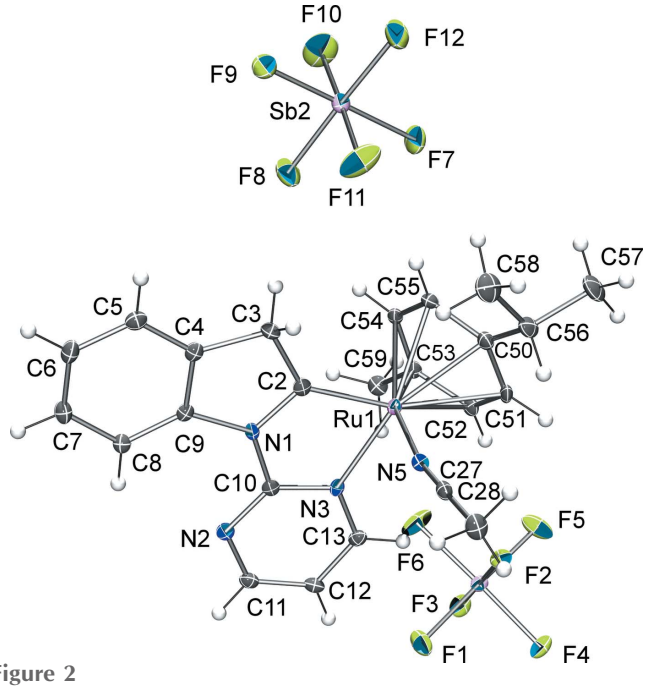


Figure 2
ORTEP representation of the molecular components of the title compound, showing 50% probability displacement ellipsoids.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···F7^{i}	0.95	2.54	3.398 (6)	151
C12—H12···F1	0.95	2.39	3.157 (5)	138
C13—H13···F2	0.95	2.30	3.229 (5)	167
C51—H51···F11^{ii}	0.95	2.54	3.485 (6)	174
C52—H52···F2	0.95	2.50	3.337 (5)	147
C54—H54···F5^{iii}	0.95	2.26	3.110 (5)	148
C59—H59*B*···F6	0.98	2.32	3.253 (6)	158
C59—H59*C*···F5^{iii}	0.98	2.45	3.270 (6)	141

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

4. Database survey

This structure is related to chloro(η^6-*para*-cymene)[κ^2-*N*,*C*-1-(pyrimidin-2-yl)-1*H*-indole]ruthenium (Sollert *et al.*, 2015), in which the double bond is at C2═C3. The Ru1—C2 and Ru1-cymene distances, however, are almost unaltered. This is consistent with the development of a positive charge at N1 to effect the C3 protonation rather than at the Ru^{II} atom. The C2 atom in the title compound is therefore formally an anionic ligand, and not a carbene carbon. A similar cyclometalated pyrrolinyl complex (2) Buil *et al.*, 2015; Fig. 1) was obtained through HBF_4-mediated rearrangement of *N*-allylic substituents. The Ru—C distances of 2.077 (4) Å (Buil *et al.*, 2003) are comparable to the Ru1—C2 distance of the title compound. The Ru-catalysed rearrangement of a 1,7-eneyne afforded the C2-cyclometalated 3*H*-indole (3) (Chiang *et al.*, 2010; Fig. 1). Structural parameters of this cyclopentadienyl-coordinated ruthenium complex are in good agreement with the title compound.

5. Synthesis and crystallization

A pre-dried Young's tube was charged with chlorido(η^6-*para*-cymene)[κ^2-*N*,*C*-1-(pyrimidin-2-yl)-1*H*-indole]ruthenium (50 mg, 1.0 equiv., 0.11 mmol) and $AgSbF_6$ (76 mg, 2.0 equiv., 0.22 mmol). The tube was evacuated and backfilled with argon three times. The tube was equipped with a rubber septum and anhydrous MeCN (2 mL) was added *via* a syringe. The septum was removed, the tube sealed and wrapped in aluminium foil to protect the reaction mixture from light. The mixture was left stirring at room temperature for 18 h, after which the resulting precipitate was filtered off rapidly under air and the filtrate transferred immediately into a pre-dried round-bottom flask under argon. The solvent was evaporated under reduced pressure and a green solid was obtained. The solid was dissolved in d_8-THF and transferred into a NMR tube under argon. The title compound was obtained as green crystals upon slow evaporation of the solvent.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ru(C_{10}H_{14})(C_{12}H_9N_3)(C_2H_3N)][SbF_6]_2$
M_r	943.06
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	100
a, *b*, *c* (Å)	16.6046 (8), 15.5955 (7), 23.2786 (12)
V (Å^3)	6028.2 (5)
Z	8
Radiation type	Mo *Kα*
μ (mm^{-1})	2.37
Crystal size (mm)	0.18 × 0.17 × 0.08
Data collection	
Diffractometer	BrukerAPEXII with CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.578, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27645, 6643, 4920
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.643
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.074, 1.01
No. of reflections	6643
No. of parameters	392
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.89, −1.00

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

6. Refinement

Crystal data, data collection and refinement details are summarized in Table 2. All H atoms on carbon were placed at calculated positions [C—H = 0.95 (aromatic), 0.98 (methyl), 0.99 (methylene) and 1.00 (methine) Å] using a riding model with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). The Ru—C bonds were ignored in the ideal placement of the aromatic H atoms.

Acknowledgements

The authors would like to thank the Swedish research council (Vetenskapsrådet) for support.

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supporting information

Acta Cryst. (2015). E**71**, 1190-1192 [doi:10.1107/S2056989015016710]

Crystal structure of acetonitrile[η^6-1-methyl-4-(1-methylethyl)benzene][1-(pyrimidin-2-yl)-3*H*-indol-1-ium-2-yl-κ^2N,C]ruthenium(II) bis(hexafluoridoantimonate)

Carina Sollert, Andreas Orthaber and Lukasz T. Pilarski

Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

Acetonitrile[η^6-1-methyl-4-(1-methylethyl)benzene][1-(pyrimidin-2-yl)-3*H*-indol-1-ium-2-yl-κ^2N,C]ruthenium(II) bis(hexafluoridoantimonate)

Crystal data

$[Ru(C_{10}H_{14})(C_{12}H_9N_3)(C_2H_3N)][SbF_6]_2$
M_r = 943.06
Orthorhombic, *Pbca*
a = 16.6046 (8) Å
b = 15.5955 (7) Å
c = 23.2786 (12) Å
V = 6028.2 (5) Å^3
Z = 8
F(000) = 3616
D_x = 2.078 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4920 reflections
θ = 1.8–25.2°
μ = 2.37 mm^{-1}
T = 100 K
Plate, green
0.18 × 0.17 × 0.08 mm

Data collection

BrukerAPEXII with CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.578, T_{max} = 0.746
27645 measured reflections
6643 independent reflections
4920 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 27.2°, θ_{min} = 1.8°
h = −21→20
k = −19→19
l = −29→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.074
S = 1.01
6643 reflections
392 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 2.8958P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.89$ e Å^{-3}
$\Delta\rho_{min} = -1.00$ e Å^{-3}

Special details

Experimental. X-ray crystallographic data for I were collected from a single-crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker smart diffractometer equipped with an *APEX* II CCD Detector, a graphite monochromator. The crystal-to-detector distance was 5.0 cm, and the data collection was carried out in 512 *x* 512 pixel mode.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ru1	0.93384 (2)	0.22549 (2)	0.87058 (2)	0.01738 (8)
Sb1	0.85442 (2)	−0.13417 (2)	0.94657 (2)	0.02309 (8)
Sb2	0.78888 (2)	0.49530 (2)	0.71378 (2)	0.02768 (8)
F1	0.89213 (19)	−0.09995 (16)	1.01877 (12)	0.0461 (8)
F2	0.93350 (14)	−0.06576 (14)	0.91106 (11)	0.0282 (6)
F3	0.77479 (15)	−0.20299 (16)	0.98028 (12)	0.0372 (7)
F4	0.92598 (15)	−0.22620 (14)	0.94954 (12)	0.0359 (6)
F5	0.82004 (18)	−0.1684 (2)	0.87420 (12)	0.0517 (8)
F6	0.78500 (18)	−0.04062 (18)	0.94558 (15)	0.0605 (10)
F7	0.7883 (2)	0.37969 (16)	0.69278 (13)	0.0558 (9)
F8	0.7572 (2)	0.46434 (18)	0.78733 (12)	0.0538 (9)
F9	0.79200 (19)	0.61114 (16)	0.73516 (12)	0.0464 (8)
F10	0.6831 (2)	0.5081 (2)	0.69167 (18)	0.0795 (12)
F11	0.89525 (19)	0.48324 (19)	0.73806 (18)	0.0732 (11)
F12	0.8239 (3)	0.5243 (2)	0.64075 (14)	0.0872 (14)
N1	0.90603 (19)	0.3372 (2)	0.96626 (14)	0.0178 (7)
N2	0.8897 (2)	0.2522 (2)	1.04905 (14)	0.0214 (8)
N3	0.91640 (18)	0.1923 (2)	0.95632 (14)	0.0176 (7)
N5	1.0551 (2)	0.2291 (2)	0.88533 (14)	0.0210 (8)
C2	0.9204 (2)	0.3380 (3)	0.90940 (18)	0.0206 (9)
C3	0.9232 (3)	0.4290 (2)	0.89077 (17)	0.0223 (9)
H3A	0.8806	0.4410	0.8621	0.027*
H3B	0.9762	0.4431	0.8738	0.027*
C4	0.9091 (2)	0.4796 (2)	0.94537 (18)	0.0214 (9)
C5	0.9052 (3)	0.5662 (3)	0.95639 (19)	0.0263 (10)
H5	0.9146	0.6068	0.9267	0.032*
C6	0.8872 (3)	0.5931 (3)	1.0116 (2)	0.0281 (10)
H6	0.8847	0.6528	1.0196	0.034*

C7	0.8729 (3)	0.5347 (3)	1.05571 (19)	0.0267 (10)
H7	0.8592	0.5549	1.0930	0.032*
C8	0.8786 (2)	0.4467 (3)	1.04542 (18)	0.0223 (9)
H8	0.8703	0.4058	1.0751	0.027*
C9	0.8969 (2)	0.4222 (2)	0.99016 (18)	0.0191 (9)
C10	0.9032 (2)	0.2573 (2)	0.99352 (18)	0.0190 (9)
C11	0.8882 (2)	0.1724 (3)	1.07022 (18)	0.0230 (9)
H11	0.8788	0.1650	1.1102	0.028*
C12	0.8997 (2)	0.1006 (3)	1.03687 (17)	0.0229 (9)
H12	0.8967	0.0446	1.0527	0.028*
C13	0.9157 (2)	0.1136 (2)	0.97918 (18)	0.0219 (9)
H13	0.9264	0.0655	0.9553	0.026*
C27	1.1233 (3)	0.2267 (2)	0.88601 (17)	0.0209 (9)
C28	1.2103 (3)	0.2249 (3)	0.8860 (2)	0.0326 (11)
H28A	1.2303	0.2392	0.8476	0.049*
H28B	1.2289	0.1674	0.8967	0.049*
H28C	1.2307	0.2668	0.9138	0.049*
C50	0.9606 (3)	0.2124 (3)	0.77680 (18)	0.0247 (10)
C51	0.9519 (3)	0.1264 (3)	0.79863 (17)	0.0241 (10)
H51	0.9932	0.0856	0.7916	0.029*
C52	0.8850 (3)	0.1023 (3)	0.82936 (17)	0.0227 (9)
H52	0.8808	0.0452	0.8433	0.027*
C53	0.8216 (2)	0.1625 (3)	0.84050 (18)	0.0207 (9)
C54	0.8274 (2)	0.2446 (2)	0.81594 (16)	0.0196 (9)
H54	0.7842	0.2840	0.8205	0.024*
C55	0.8962 (2)	0.2697 (3)	0.78465 (17)	0.0213 (9)
H55	0.8990	0.3257	0.7687	0.026*
C56	1.0360 (3)	0.2354 (3)	0.74384 (19)	0.0305 (11)
H56	1.0823	0.2063	0.7631	0.037*
C57	1.0281 (3)	0.1975 (3)	0.6834 (2)	0.0448 (14)
H57A	1.0189	0.1355	0.6861	0.067*
H57B	1.0777	0.2083	0.6617	0.067*
H57C	0.9825	0.2244	0.6635	0.067*
C58	1.0541 (3)	0.3316 (3)	0.7425 (2)	0.0445 (13)
H58A	1.1071	0.3411	0.7252	0.067*
H58C	1.0539	0.3542	0.7818	0.067*
H58B	1.0129	0.3611	0.7198	0.067*
C59	0.7499 (3)	0.1355 (3)	0.87511 (19)	0.0283 (10)
H59C	0.7273	0.1855	0.8949	0.042*
H59B	0.7664	0.0926	0.9035	0.042*
H59A	0.7091	0.1108	0.8496	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01528 (16)	0.01805 (16)	0.01879 (17)	0.00165 (13)	0.00117 (14)	−0.00097 (14)
Sb1	0.01982 (15)	0.02391 (15)	0.02554 (16)	0.00207 (12)	0.00044 (13)	0.00291 (13)
Sb2	0.03160 (17)	0.02741 (16)	0.02404 (16)	0.00270 (13)	0.00236 (13)	−0.00066 (13)

F1	0.073 (2)	0.0358 (15)	0.0295 (16)	−0.0094 (15)	0.0018 (15)	−0.0053 (13)
F2	0.0254 (13)	0.0237 (12)	0.0354 (15)	−0.0027 (11)	−0.0018 (12)	0.0042 (11)
F3	0.0258 (15)	0.0391 (15)	0.0466 (17)	−0.0006 (12)	0.0117 (13)	0.0062 (13)
F4	0.0288 (15)	0.0234 (13)	0.0557 (18)	0.0042 (11)	0.0071 (13)	0.0046 (13)
F5	0.0488 (19)	0.075 (2)	0.0315 (17)	−0.0302 (17)	−0.0106 (14)	0.0072 (15)
F6	0.0386 (18)	0.0464 (17)	0.096 (3)	0.0217 (14)	0.0235 (18)	0.0285 (18)
F7	0.089 (3)	0.0309 (16)	0.0481 (19)	0.0035 (15)	0.0041 (18)	−0.0131 (14)
F8	0.082 (2)	0.0438 (17)	0.0351 (17)	−0.0126 (17)	0.0162 (16)	0.0004 (14)
F9	0.068 (2)	0.0297 (15)	0.0410 (17)	−0.0018 (14)	0.0026 (16)	0.0015 (13)
F10	0.055 (2)	0.085 (3)	0.098 (3)	0.011 (2)	−0.041 (2)	−0.017 (2)
F11	0.0317 (18)	0.0531 (19)	0.135 (4)	0.0016 (15)	−0.007 (2)	0.019 (2)
F12	0.164 (4)	0.055 (2)	0.042 (2)	0.028 (2)	0.048 (2)	0.0118 (17)
N1	0.0178 (17)	0.0198 (17)	0.0158 (18)	0.0006 (14)	0.0008 (14)	−0.0036 (14)
N2	0.0171 (18)	0.0243 (19)	0.023 (2)	0.0003 (15)	−0.0010 (15)	−0.0002 (16)
N3	0.0131 (17)	0.0188 (17)	0.0209 (19)	0.0016 (13)	0.0025 (14)	−0.0020 (15)
N5	0.023 (2)	0.0222 (18)	0.0182 (19)	0.0017 (15)	0.0006 (15)	−0.0003 (15)
C2	0.010 (2)	0.027 (2)	0.024 (2)	0.0011 (16)	−0.0018 (17)	−0.0022 (18)
C3	0.025 (2)	0.025 (2)	0.017 (2)	−0.0011 (18)	0.0005 (18)	0.0039 (18)
C4	0.014 (2)	0.023 (2)	0.027 (2)	0.0000 (16)	−0.0017 (18)	−0.0017 (19)
C5	0.022 (2)	0.027 (2)	0.030 (3)	0.0025 (18)	−0.0022 (19)	0.003 (2)
C6	0.024 (2)	0.025 (2)	0.035 (3)	0.0022 (19)	−0.002 (2)	−0.009 (2)
C7	0.025 (2)	0.028 (2)	0.027 (2)	−0.0029 (19)	0.002 (2)	−0.008 (2)
C8	0.020 (2)	0.022 (2)	0.024 (2)	−0.0023 (17)	0.0005 (18)	−0.0019 (19)
C9	0.013 (2)	0.019 (2)	0.025 (2)	−0.0008 (16)	−0.0015 (17)	−0.0016 (18)
C10	0.0097 (19)	0.025 (2)	0.022 (2)	−0.0012 (16)	−0.0028 (17)	−0.0017 (18)
C11	0.020 (2)	0.030 (2)	0.020 (2)	0.0007 (18)	−0.0028 (18)	0.0031 (19)
C12	0.022 (2)	0.027 (2)	0.020 (2)	−0.0031 (18)	−0.0039 (18)	0.0056 (19)
C13	0.020 (2)	0.020 (2)	0.026 (2)	0.0012 (16)	−0.0060 (19)	−0.0006 (18)
C27	0.026 (2)	0.020 (2)	0.017 (2)	−0.0017 (18)	0.0019 (18)	−0.0029 (17)
C28	0.020 (2)	0.043 (3)	0.035 (3)	−0.005 (2)	0.002 (2)	−0.007 (2)
C50	0.024 (2)	0.032 (2)	0.018 (2)	0.0057 (19)	−0.0021 (18)	−0.0047 (19)
C51	0.026 (2)	0.027 (2)	0.019 (2)	0.0100 (18)	−0.0035 (18)	−0.0101 (19)
C52	0.027 (2)	0.022 (2)	0.020 (2)	−0.0010 (18)	−0.0037 (19)	−0.0070 (18)
C53	0.015 (2)	0.025 (2)	0.022 (2)	0.0001 (17)	−0.0028 (18)	−0.0043 (18)
C54	0.016 (2)	0.025 (2)	0.017 (2)	0.0041 (17)	−0.0047 (17)	−0.0043 (18)
C55	0.024 (2)	0.024 (2)	0.016 (2)	0.0022 (18)	−0.0005 (18)	0.0024 (18)
C56	0.026 (2)	0.043 (3)	0.022 (2)	0.005 (2)	0.007 (2)	0.003 (2)
C57	0.045 (3)	0.066 (4)	0.023 (3)	0.003 (3)	0.014 (2)	0.001 (3)
C58	0.036 (3)	0.053 (3)	0.045 (3)	−0.004 (3)	0.013 (3)	0.001 (3)
C59	0.026 (2)	0.028 (2)	0.031 (3)	−0.006 (2)	−0.001 (2)	−0.001 (2)

Geometric parameters (Å, º)

Ru1—C2	1.986 (4)	C56—C57	1.533 (6)
Ru1—N3	2.082 (3)	C56—H56	1.0000
Ru1—N5	2.044 (3)	C57—H57A	0.9800
Ru1—C50	2.237 (4)	C57—H57B	0.9800
Ru1—C51	2.298 (4)	C57—H57C	0.9800

Ru1—C52	2.296 (4)	C58—H58A	0.9800
Ru1—C53	2.220 (4)	C58—H58C	0.9800
Ru1—C54	2.197 (4)	C58—H58B	0.9800
Ru1—C55	2.206 (4)	C2—N1	1.345 (5)
Sb1—F1	1.871 (3)	C2—C3	1.485 (5)
Sb1—F2	1.883 (2)	C3—C4	1.514 (5)
Sb1—F3	1.875 (2)	C3—H3A	0.9900
Sb1—F4	1.865 (2)	C3—H3B	0.9900
Sb1—F5	1.857 (3)	C4—C5	1.377 (6)
Sb1—F6	1.860 (3)	C4—C9	1.388 (5)
Sb2—F7	1.868 (3)	C9—C8	1.376 (5)
Sb2—F8	1.855 (3)	C9—N1	1.447 (5)
Sb2—F9	1.875 (3)	C8—C7	1.396 (6)
Sb2—F10	1.841 (3)	C8—H8	0.9500
Sb2—F11	1.864 (3)	C7—C6	1.393 (6)
Sb2—F12	1.853 (3)	C7—H7	0.9500
N5—C27	1.133 (5)	C6—C5	1.384 (6)
C50—C55	1.406 (6)	C6—H6	0.9500
C50—C51	1.442 (6)	C5—H5	0.9500
C50—C56	1.510 (6)	N1—C10	1.399 (5)
C51—C52	1.373 (6)	C10—N2	1.314 (5)
C51—H51	0.9500	C10—N3	1.351 (5)
C52—C53	1.436 (5)	N2—C11	1.339 (5)
C52—H52	0.9500	C11—C12	1.375 (6)
C53—C54	1.406 (5)	C11—H11	0.9500
C53—C59	1.497 (6)	C12—C13	1.384 (6)
C54—C55	1.409 (6)	C12—H12	0.9500
C54—H54	0.9500	C13—N3	1.338 (5)
C55—H55	0.9500	C13—H13	0.9500
C59—H59C	0.9800	C27—C28	1.444 (6)
C59—H59B	0.9800	C28—H28A	0.9800
C59—H59A	0.9800	C28—H28B	0.9800
C56—C58	1.531 (6)	C28—H28C	0.9800

C2—Ru1—N5	90.54 (14)	C59—C53—Ru1	128.4 (3)
C2—Ru1—N3	76.59 (15)	C53—C54—C55	121.3 (4)
N5—Ru1—N3	89.02 (13)	C53—C54—Ru1	72.3 (2)
C2—Ru1—C54	93.05 (15)	C55—C54—Ru1	71.7 (2)
N5—Ru1—C54	152.34 (14)	C53—C54—H54	119.4
N3—Ru1—C54	118.48 (14)	C55—C54—H54	119.4
C2—Ru1—C55	96.00 (16)	Ru1—C54—H54	129.0
N5—Ru1—C55	115.03 (14)	C50—C55—C54	120.5 (4)
N3—Ru1—C55	155.13 (14)	C50—C55—Ru1	72.7 (2)
C54—Ru1—C55	37.32 (15)	C54—C55—Ru1	71.0 (2)
C2—Ru1—C53	116.11 (15)	C50—C55—H55	119.8
N5—Ru1—C53	153.20 (14)	C54—C55—H55	119.8
N3—Ru1—C53	94.33 (14)	Ru1—C55—H55	128.8
C54—Ru1—C53	37.11 (14)	C53—C59—H59C	109.5

C55—Ru1—C53	67.31 (15)	C53—C59—H59B	109.5
C2—Ru1—C50	123.14 (16)	H59C—C59—H59B	109.5
N5—Ru1—C50	88.31 (14)	C53—C59—H59A	109.5
N3—Ru1—C50	160.11 (14)	H59C—C59—H59A	109.5
C54—Ru1—C50	66.88 (15)	H59B—C59—H59A	109.5
C55—Ru1—C50	36.89 (14)	C50—C56—C58	113.9 (4)
C53—Ru1—C50	79.57 (15)	C50—C56—C57	107.7 (4)
C2—Ru1—C52	152.89 (15)	C58—C56—C57	112.1 (4)
N5—Ru1—C52	116.18 (14)	C50—C56—H56	107.6
N3—Ru1—C52	98.24 (14)	C58—C56—H56	107.6
C54—Ru1—C52	65.67 (15)	C57—C56—H56	107.6
C55—Ru1—C52	77.44 (15)	C56—C57—H57A	109.5
C53—Ru1—C52	37.03 (14)	C56—C57—H57B	109.5
C50—Ru1—C52	65.55 (15)	H57A—C57—H57B	109.5
C2—Ru1—C51	160.05 (16)	C56—C57—H57C	109.5
N5—Ru1—C51	90.72 (14)	H57A—C57—H57C	109.5
N3—Ru1—C51	123.34 (14)	H57B—C57—H57C	109.5
C54—Ru1—C51	76.96 (15)	C56—C58—H58A	109.5
C55—Ru1—C51	65.56 (15)	C56—C58—H58C	109.5
C53—Ru1—C51	65.32 (15)	H58A—C58—H58C	109.5
C50—Ru1—C51	37.03 (15)	C56—C58—H58B	109.5
C52—Ru1—C51	34.79 (15)	H58A—C58—H58B	109.5
F5—Sb1—F6	91.35 (15)	H58C—C58—H58B	109.5
F5—Sb1—F4	90.48 (13)	N1—C2—C3	107.6 (3)
F6—Sb1—F4	178.08 (15)	N1—C2—Ru1	117.3 (3)
F5—Sb1—F1	178.35 (14)	C3—C2—Ru1	135.1 (3)
F6—Sb1—F1	89.71 (14)	C2—C3—C4	104.4 (3)
F4—Sb1—F1	88.45 (12)	C2—C3—H3A	110.9
F5—Sb1—F3	89.91 (12)	C4—C3—H3A	110.9
F6—Sb1—F3	90.98 (12)	C2—C3—H3B	110.9
F4—Sb1—F3	89.61 (11)	C4—C3—H3B	110.9
F1—Sb1—F3	91.34 (12)	H3A—C3—H3B	108.9
F5—Sb1—F2	88.79 (11)	C5—C4—C9	119.0 (4)
F6—Sb1—F2	88.99 (11)	C5—C4—C3	132.5 (4)
F4—Sb1—F2	90.46 (10)	C9—C4—C3	108.5 (3)
F1—Sb1—F2	89.96 (12)	C8—C9—C4	123.8 (4)
F3—Sb1—F2	178.70 (12)	C8—C9—N1	129.6 (4)
F10—Sb2—F12	91.0 (2)	C4—C9—N1	106.7 (3)
F10—Sb2—F8	90.89 (17)	C9—C8—C7	116.6 (4)
F12—Sb2—F8	178.02 (18)	C9—C8—H8	121.7
F10—Sb2—F11	178.52 (18)	C7—C8—H8	121.7
F12—Sb2—F11	90.30 (19)	C6—C7—C8	120.3 (4)
F8—Sb2—F11	87.86 (17)	C6—C7—H7	119.8
F10—Sb2—F7	91.56 (15)	C8—C7—H7	119.8
F12—Sb2—F7	89.85 (14)	C5—C6—C7	121.5 (4)
F8—Sb2—F7	89.37 (13)	C5—C6—H6	119.2
F11—Sb2—F7	89.24 (15)	C7—C6—H6	119.2
F10—Sb2—F9	89.76 (14)	C4—C5—C6	118.7 (4)

F12—Sb2—F9	89.97 (14)	C4—C5—H5	120.6
F8—Sb2—F9	90.77 (12)	C6—C5—H5	120.6
F11—Sb2—F9	89.45 (14)	C2—N1—C10	117.4 (3)
F7—Sb2—F9	178.67 (15)	C2—N1—C9	112.8 (3)
C27—N5—Ru1	170.4 (3)	C10—N1—C9	129.7 (3)
C55—C50—C51	117.9 (4)	N2—C10—N3	127.8 (4)
C55—C50—C56	123.1 (4)	N2—C10—N1	120.4 (4)
C51—C50—C56	118.9 (4)	N3—C10—N1	111.8 (3)
C55—C50—Ru1	70.4 (2)	C10—N2—C11	114.9 (4)
C51—C50—Ru1	73.8 (2)	N2—C11—C12	123.1 (4)
C56—C50—Ru1	129.7 (3)	N2—C11—H11	118.5
C52—C51—C50	121.4 (4)	C12—C11—H11	118.5
C52—C51—Ru1	72.5 (2)	C11—C12—C13	117.1 (4)
C50—C51—Ru1	69.2 (2)	C11—C12—H12	121.4
C52—C51—H51	119.3	C13—C12—H12	121.4
C50—C51—H51	119.3	N3—C13—C12	121.4 (4)
Ru1—C51—H51	132.0	N3—C13—H13	119.3
C51—C52—C53	120.5 (4)	C12—C13—H13	119.3
C51—C52—Ru1	72.7 (2)	C13—N3—C10	115.6 (3)
C53—C52—Ru1	68.6 (2)	C13—N3—Ru1	127.6 (3)
C51—C52—H52	119.7	C10—N3—Ru1	116.8 (3)
C53—C52—H52	119.7	N5—C27—C28	178.9 (5)
Ru1—C52—H52	131.8	C27—C28—H28A	109.5
C54—C53—C52	118.2 (4)	C27—C28—H28B	109.5
C54—C53—C59	122.0 (4)	H28A—C28—H28B	109.5
C52—C53—C59	119.8 (4)	C27—C28—H28C	109.5
C54—C53—Ru1	70.6 (2)	H28A—C28—H28C	109.5
C52—C53—Ru1	74.3 (2)	H28B—C28—H28C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···F7^{i}	0.95	2.54	3.398 (6)	151
C12—H12···F1	0.95	2.39	3.157 (5)	138
C13—H13···F2	0.95	2.30	3.229 (5)	167
C51—H51···F11^{ii}	0.95	2.54	3.485 (6)	174
C52—H52···F2	0.95	2.50	3.337 (5)	147
C54—H54···F5^{iii}	0.95	2.26	3.110 (5)	148
C59—H59*B*···F6	0.98	2.32	3.253 (6)	158
C59—H59*C*···F5^{iii}	0.98	2.45	3.270 (6)	141

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$; (ii) $-x+2$, $y-1/2$, $-z+3/2$; (iii) $-x+3/2$, $y+1/2$, z.