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Hydrogen bonding in thiacrown complexes: chlorobis(nicotinamide-κN)(1,4,7-trithiacyclononane-$\kappa^3 S$)ruthenium(II) hexafluorophosphate monohydrate

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The title complex, $[RuClL_2([9]\text{-ane-}S_3)]PF_6\cdot H_2O$ [where L is nicotinamide, $C_7H_6N_2O$) and [9]-ane-S_3 is 1,4,7-trithiacyclononane ($C_6H_{12}S_3$)], was isolated as a hexafluorophosphate salt from water–ethanol. The structure confirms that the amide moieties are available for possible mutual hydrogen-bond interactions. However, from aqueous solvents, these sites are involved in networks of interactions with water molecules.

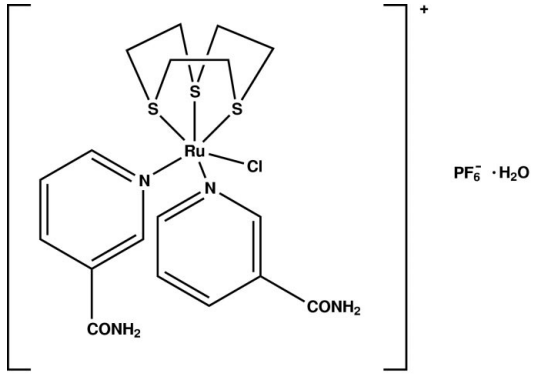
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 17.8

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

Comment

The targeted design of long-range solid-state structures, or crystal engineering, is a fast-emerging area of chemistry. In this context, structures mediated by ligand coordination to specific metal centres has been particularly well pursued. However, comparatively less work has involved coordination complexes with coordinated ligands containing hydrogen-bonding sites. As part of a programme investigating the possible assembly of solution- and solid-phase host structures, we are synthesizing complexes based on Ru^{II} centres facially capped by thiacrown ligands. Coordination of two or three nicotinic and isonicotinic acid derivatives to these centres provides two- and three-dimensional synthons for crystal engineering. Solubility properties of the resultant complexes can be modulated by a change of counter-ion. The complexes are initially synthesized as chloride salts, but anion metathesis affords a route to a variety of other counter-ions. Using this methodology, we are exploring the effect of the counter-ion on the long-range structure of the resulting crystallographic architecture. We report here the structure of $[RuClL_2([9]\text{-ane-}S_3)]PF_6$ (where L is nicotinamide and [9]-ane-S_3 is 1,4,7-trithiacyclononane) grown from water–ethanol as a monohydrate, (I), in which the water molecules interact with the projecting amide groups.



(I)

Experimental

$[RuCl_2(DMSO)([9]\text{-ane-}S_3)]$ (0.215 g, 0.5 mmol) and nicotinamide (0.122 g, 1.0 mmol) were heated at reflux for 4 h under a nitrogen atmosphere in 30 ml of a water–ethanol mixture (1:1). The reaction mixture was allowed to cool and any insoluble material was removed by filtration. Addition of ammonium hexafluorophosphate (0.163 g, 1.0 mmol) led to the crystallization of the final product. This was collected by filtration, washed with (3 × 10 ml) portions of water, ethanol and diethyl ether, and allowed to dry *in vacuo*. The product was obtained as a yellow powder (yield 0.178 g, 50%). Crystals suitable for X-ray crystallography were obtained via slow evaporation from the water:ethanol mother liquor.

Crystal data

$[RuCl(C_6H_6N_2O)_2(C_6H_{12}S_3)](PF_6)\cdot H_2O$
$M_r = 724.10$
Monoclinic, $P2_1/c$
$a = 12.9575\ (15)$ Å
$b = 9.8190\ (11)$ Å
$c = 20.974\ (3)$ Å
$\beta = 101.483\ (2)^\circ$
$V = 2615.1\ (5)$ Å^3
$Z = 4$
$D_x = 1.839$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4801 reflections
$\theta = 5.2$–54.6°
$\mu = 1.08$ mm^{-1}
$T = 150\ (2)$ K
Block, yellow
0.32 × 0.28 × 0.21 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.725$, $T_{max} = 0.806$
28 430 measured reflections
5948 independent reflections
4693 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.6^\circ$
$h = -16 \rightarrow 16$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.116$
$S = 1.05$
5948 reflections
334 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 5.2806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.49$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}

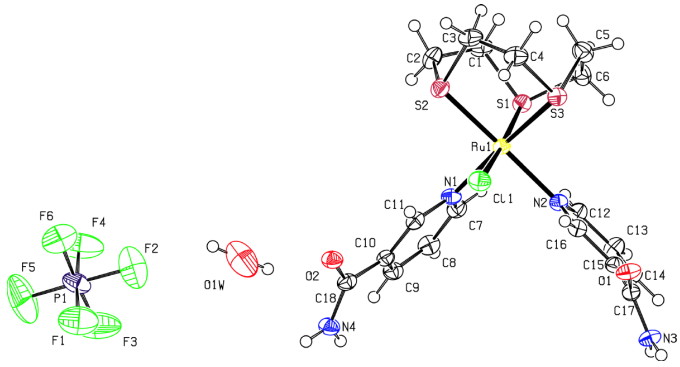


Figure 1
View of (I), showing the numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

The H atoms were introduced at calculated positions and treated as riding atoms, with bond lengths of NH_2 0.88 Å, 0.95(C—H aromatic), and 0.99 Å (CH_2). The exception being the water O—H lengths which were found by low-theta difference Fourier, then restrained to 0.85 (1) Å. The thermal displacement parameters were all made equal to 1.2 times U_{eq} (parent N, O or C atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

supporting information

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Monoclinic, $P2_1/c$
$a = 12.9575\ (15)$ Å
$b = 9.8190\ (11)$ Å
$c = 20.974\ (3)$ Å
$\beta = 101.483\ (2)°$
$V = 2615.1\ (5)$ Å^3
$Z = 4$
$F(000) = 1456$
$D_x = 1.839$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4801 reflections
$\theta = 5.2–54.6°$
$\mu = 1.08$ mm^{-1}
$T = 150$ K
Block, yellow
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Radiation source: fine-focus sealed tube
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$R_{int} = 0.039$
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$h = -16\rightarrow16$
$k = -12\rightarrow12$
$l = -27\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.116$
$S = 1.05$
5948 reflections
334 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 5.2806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.49$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ 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}
Ru1	0.19776 (2)	0.70164 (3)	0.319483 (13)	0.01861 (10)
Cl1	0.01847 (8)	0.66421 (10)	0.25691 (5)	0.0308 (2)
S1	0.36636 (7)	0.70302 (9)	0.37723 (4)	0.0231 (2)
S2	0.16145 (8)	0.53152 (10)	0.38655 (5)	0.0273 (2)
S3	0.24794 (7)	0.54053 (9)	0.25309 (4)	0.0243 (2)
N1	0.1462 (2)	0.8586 (3)	0.37745 (15)	0.0223 (6)
N2	0.2257 (2)	0.8604 (3)	0.25466 (14)	0.0214 (6)
N3	0.0935 (3)	1.0985 (3)	0.05677 (15)	0.0253 (7)
H3A	0.0523	1.1057	0.0181	0.030*
H3B	0.1261	1.1708	0.0757	0.030*
N4	−0.1557 (3)	1.1008 (3)	0.41734 (16)	0.0282 (7)
H4A	−0.2235	1.1055	0.4174	0.034*
H4B	−0.1166	1.1746	0.4241	0.034*
O1	0.0627 (2)	0.8725 (3)	0.06244 (13)	0.0280 (6)
O2	−0.1639 (2)	0.8741 (3)	0.39730 (14)	0.0285 (6)
C1	0.3748 (3)	0.5650 (4)	0.43730 (19)	0.0297 (9)
H1A	0.3978	0.4801	0.4188	0.036*
H1B	0.4281	0.5887	0.4765	0.036*
C2	0.2704 (3)	0.5417 (4)	0.45614 (19)	0.0335 (9)
H2A	0.2568	0.6170	0.4848	0.040*
H2B	0.2737	0.4560	0.4813	0.040*
C3	0.1930 (3)	0.3712 (4)	0.3486 (2)	0.0336 (9)
H3D	0.2651	0.3421	0.3691	0.040*
H3E	0.1435	0.2993	0.3565	0.040*
C4	0.1861 (3)	0.3866 (4)	0.2762 (2)	0.0331 (9)
H4D	0.1109	0.3867	0.2544	0.040*
H4E	0.2197	0.3066	0.2601	0.040*
C5	0.3877 (3)	0.5047 (4)	0.2854 (2)	0.0325 (9)
H5A	0.3940	0.4260	0.3155	0.039*
H5B	0.4230	0.4807	0.2492	0.039*
C6	0.4411 (3)	0.6278 (4)	0.3213 (2)	0.0315 (9)
H6A	0.4519	0.6974	0.2891	0.038*
H6B	0.5113	0.6004	0.3460	0.038*
C7	0.2126 (3)	0.9549 (4)	0.40857 (18)	0.0268 (8)
H7	0.2858	0.9455	0.4095	0.032*

C8	0.1787 (3)	1.0653 (4)	0.43880 (19)	0.0280 (8)
H8	0.2277	1.1311	0.4597	0.034*
C9	0.0724 (3)	1.0800 (4)	0.43849 (18)	0.0264 (8)
H9	0.0470	1.1568	0.4582	0.032*
C10	0.0037 (3)	0.9801 (4)	0.40874 (18)	0.0229 (7)
C11	0.0435 (3)	0.8715 (4)	0.37911 (17)	0.0232 (8)
H11	−0.0039	0.8031	0.3591	0.028*
C12	0.3037 (3)	0.9516 (4)	0.27140 (19)	0.0259 (8)
H12	0.3473	0.9451	0.3134	0.031*
C13	0.3237 (3)	1.0540 (4)	0.23064 (19)	0.0291 (9)
H13	0.3800	1.1159	0.2446	0.035*
C14	0.2612 (3)	1.0660 (4)	0.16944 (19)	0.0269 (8)
H14	0.2731	1.1361	0.1405	0.032*
C15	0.1807 (3)	0.9730 (4)	0.15150 (17)	0.0217 (7)
C16	0.1654 (3)	0.8718 (4)	0.19481 (17)	0.0224 (7)
H16	0.1102	0.8080	0.1817	0.027*
C17	0.1064 (3)	0.9782 (4)	0.08650 (18)	0.0231 (8)
C18	−0.1124 (3)	0.9821 (4)	0.40712 (17)	0.0249 (8)
P1	0.46884 (10)	0.82344 (13)	0.59334 (6)	0.0403 (3)
F1	0.3684 (3)	0.8500 (4)	0.53914 (19)	0.0786 (11)
F2	0.5298 (3)	0.7825 (4)	0.5370 (2)	0.0902 (13)
F3	0.5049 (3)	0.9775 (4)	0.5886 (2)	0.0944 (14)
F4	0.5742 (3)	0.7989 (3)	0.6439 (2)	0.0896 (14)
F5	0.4085 (4)	0.8643 (6)	0.6465 (2)	0.1205 (18)
F6	0.4388 (3)	0.6669 (3)	0.59625 (16)	0.0671 (9)
O1W	0.6483 (3)	0.7735 (4)	0.4320 (3)	0.0801 (14)
H2W	0.6918	0.8241	0.4177	0.096*
H1W	0.6300	0.7398	0.4654	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01819 (16)	0.01820 (15)	0.01811 (16)	−0.00032 (11)	0.00040 (11)	0.00068 (11)
Cl1	0.0258 (5)	0.0314 (5)	0.0331 (5)	0.0003 (4)	0.0007 (4)	−0.0005 (4)
S1	0.0199 (4)	0.0265 (5)	0.0211 (5)	−0.0004 (3)	−0.0003 (3)	0.0007 (3)
S2	0.0281 (5)	0.0257 (5)	0.0288 (5)	−0.0024 (4)	0.0072 (4)	0.0045 (4)
S3	0.0267 (5)	0.0230 (5)	0.0222 (5)	0.0003 (4)	0.0022 (4)	−0.0019 (3)
N1	0.0216 (15)	0.0190 (15)	0.0237 (16)	0.0014 (12)	−0.0015 (12)	−0.0002 (12)
N2	0.0219 (15)	0.0203 (15)	0.0210 (16)	0.0002 (12)	0.0016 (12)	−0.0002 (12)
N3	0.0309 (17)	0.0191 (15)	0.0223 (16)	−0.0008 (13)	−0.0034 (13)	0.0025 (12)
N4	0.0291 (17)	0.0198 (16)	0.0355 (19)	0.0016 (13)	0.0063 (14)	−0.0016 (13)
O1	0.0343 (15)	0.0175 (13)	0.0267 (14)	−0.0008 (11)	−0.0072 (11)	0.0012 (11)
O2	0.0281 (14)	0.0205 (13)	0.0364 (16)	0.0002 (11)	0.0055 (12)	0.0008 (11)
C1	0.033 (2)	0.029 (2)	0.0222 (19)	0.0042 (16)	−0.0065 (16)	0.0018 (15)
C2	0.040 (2)	0.038 (2)	0.022 (2)	−0.0009 (19)	0.0062 (17)	0.0072 (17)
C3	0.039 (2)	0.0176 (19)	0.043 (2)	−0.0002 (16)	0.0047 (19)	0.0028 (17)
C4	0.036 (2)	0.0218 (19)	0.040 (2)	−0.0028 (16)	0.0035 (18)	−0.0024 (17)
C5	0.027 (2)	0.036 (2)	0.034 (2)	0.0044 (17)	0.0051 (17)	−0.0008 (18)

C6	0.0220 (19)	0.042 (2)	0.031 (2)	0.0015 (17)	0.0068 (16)	−0.0005 (18)
C7	0.025 (2)	0.028 (2)	0.026 (2)	−0.0007 (15)	0.0020 (16)	0.0030 (15)
C8	0.032 (2)	0.0240 (19)	0.026 (2)	−0.0046 (16)	−0.0007 (16)	−0.0032 (15)
C9	0.033 (2)	0.0224 (19)	0.0227 (19)	0.0022 (15)	0.0032 (16)	−0.0019 (14)
C10	0.0262 (19)	0.0222 (18)	0.0201 (18)	0.0029 (14)	0.0037 (15)	0.0040 (14)
C11	0.0283 (19)	0.0210 (18)	0.0196 (18)	−0.0031 (15)	0.0030 (15)	0.0007 (14)
C12	0.0226 (19)	0.031 (2)	0.0224 (19)	−0.0030 (15)	−0.0007 (15)	0.0022 (15)
C13	0.029 (2)	0.029 (2)	0.029 (2)	−0.0108 (16)	0.0015 (16)	0.0008 (16)
C14	0.028 (2)	0.0259 (19)	0.026 (2)	−0.0031 (15)	0.0043 (16)	0.0072 (15)
C15	0.0243 (18)	0.0195 (17)	0.0208 (18)	0.0025 (14)	0.0034 (14)	−0.0006 (14)
C16	0.0217 (18)	0.0216 (18)	0.0227 (19)	0.0001 (14)	0.0014 (14)	−0.0021 (14)
C17	0.0222 (18)	0.0247 (19)	0.0219 (19)	0.0021 (14)	0.0031 (15)	0.0018 (14)
C18	0.030 (2)	0.0229 (19)	0.0198 (18)	0.0015 (15)	0.0017 (15)	0.0022 (14)
P1	0.0343 (6)	0.0412 (7)	0.0387 (7)	0.0108 (5)	−0.0085 (5)	−0.0105 (5)
F1	0.062 (2)	0.079 (2)	0.079 (2)	0.0124 (19)	−0.0238 (18)	0.002 (2)
F2	0.076 (3)	0.113 (3)	0.091 (3)	−0.015 (2)	0.039 (2)	−0.030 (2)
F3	0.080 (3)	0.046 (2)	0.138 (4)	0.0003 (18)	−0.027 (2)	−0.002 (2)
F4	0.079 (3)	0.059 (2)	0.102 (3)	0.0182 (18)	−0.052 (2)	−0.020 (2)
F5	0.115 (4)	0.178 (5)	0.079 (3)	0.011 (3)	0.045 (3)	−0.060 (3)
F6	0.078 (2)	0.0551 (19)	0.059 (2)	−0.0091 (17)	−0.0070 (17)	0.0022 (15)
O1W	0.062 (3)	0.062 (3)	0.125 (4)	−0.018 (2)	0.040 (3)	−0.035 (3)

Geometric parameters (Å, º)

Ru1—N2	2.146 (3)	C4—H4E	0.9900
Ru1—N1	2.150 (3)	C5—C6	1.516 (6)
Ru1—S1	2.2773 (9)	C5—H5A	0.9900
Ru1—S3	2.2859 (10)	C5—H5B	0.9900
Ru1—S2	2.2922 (10)	C6—H6A	0.9900
Ru1—Cl1	2.4579 (10)	C6—H6B	0.9900
S1—C6	1.820 (4)	C7—C8	1.372 (5)
S1—C1	1.839 (4)	C7—H7	0.9500
S2—C2	1.820 (4)	C8—C9	1.384 (6)
S2—C3	1.847 (4)	C8—H8	0.9500
S3—C4	1.821 (4)	C9—C10	1.386 (5)
S3—C5	1.837 (4)	C9—H9	0.9500
N1—C11	1.344 (5)	C10—C11	1.384 (5)
N1—C7	1.355 (5)	C10—C18	1.498 (5)
N2—C12	1.344 (5)	C11—H11	0.9500
N2—C16	1.344 (5)	C12—C13	1.377 (5)
N3—C17	1.330 (5)	C12—H12	0.9500
N3—H3A	0.8800	C13—C14	1.380 (5)
N3—H3B	0.8800	C13—H13	0.9500
N4—C18	1.329 (5)	C14—C15	1.381 (5)
N4—H4A	0.8800	C14—H14	0.9500
N4—H4B	0.8800	C15—C16	1.387 (5)
O1—C17	1.240 (4)	C15—C17	1.505 (5)
O2—C18	1.248 (4)	C16—H16	0.9500

C1—C2	1.501 (6)	P1—F5	1.537 (4)
C1—H1A	0.9900	P1—F1	1.570 (3)
C1—H1B	0.9900	P1—F4	1.571 (3)
C2—H2A	0.9900	P1—F6	1.590 (3)
C2—H2B	0.9900	P1—F3	1.592 (4)
C3—C4	1.510 (6)	P1—F2	1.598 (4)
C3—H3D	0.9900	O1W—H2W	0.8501
C3—H3E	0.9900	O1W—H1W	0.8499
C4—H4D	0.9900		

N2—Ru1—N1	87.02 (12)	S3—C5—H5A	109.6
N2—Ru1—S1	93.89 (8)	C6—C5—H5B	109.6
N1—Ru1—S1	93.92 (8)	S3—C5—H5B	109.6
N2—Ru1—S3	90.72 (8)	H5A—C5—H5B	108.1
N1—Ru1—S3	176.98 (8)	C5—C6—S1	113.2 (3)
S1—Ru1—S3	88.23 (4)	C5—C6—H6A	108.9
N2—Ru1—S2	177.81 (8)	S1—C6—H6A	108.9
N1—Ru1—S2	92.89 (9)	C5—C6—H6B	108.9
S1—Ru1—S2	88.30 (4)	S1—C6—H6B	108.9
S3—Ru1—S2	89.29 (4)	H6A—C6—H6B	107.7
N2—Ru1—Cl1	91.77 (8)	N1—C7—C8	122.9 (4)
N1—Ru1—Cl1	92.40 (8)	N1—C7—H7	118.6
S1—Ru1—Cl1	171.72 (4)	C8—C7—H7	118.6
S3—Ru1—Cl1	85.67 (3)	C7—C8—C9	119.4 (4)
S2—Ru1—Cl1	86.05 (4)	C7—C8—H8	120.3
C6—S1—C1	100.11 (19)	C9—C8—H8	120.3
C6—S1—Ru1	103.42 (13)	C8—C9—C10	118.5 (3)
C1—S1—Ru1	106.27 (13)	C8—C9—H9	120.8
C2—S2—C3	101.0 (2)	C10—C9—H9	120.8
C2—S2—Ru1	103.34 (14)	C11—C10—C9	119.0 (3)
C3—S2—Ru1	105.44 (14)	C11—C10—C18	117.5 (3)
C4—S3—C5	101.3 (2)	C9—C10—C18	123.5 (3)
C4—S3—Ru1	102.61 (14)	N1—C11—C10	122.9 (3)
C5—S3—Ru1	106.66 (14)	N1—C11—H11	118.5
C11—N1—C7	117.3 (3)	C10—C11—H11	118.5
C11—N1—Ru1	120.2 (2)	N2—C12—C13	123.3 (3)
C7—N1—Ru1	122.2 (3)	N2—C12—H12	118.4
C12—N2—C16	117.1 (3)	C13—C12—H12	118.4
C12—N2—Ru1	122.1 (2)	C12—C13—C14	119.5 (4)
C16—N2—Ru1	120.8 (2)	C12—C13—H13	120.3
C17—N3—H3A	120.0	C14—C13—H13	120.3
C17—N3—H3B	120.0	C13—C14—C15	118.0 (3)
H3A—N3—H3B	120.0	C13—C14—H14	121.0
C18—N4—H4A	120.0	C15—C14—H14	121.0
C18—N4—H4B	120.0	C14—C15—C16	119.6 (3)
H4A—N4—H4B	120.0	C14—C15—C17	122.4 (3)
C2—C1—S1	111.0 (3)	C16—C15—C17	118.0 (3)
C2—C1—H1A	109.4	N2—C16—C15	122.6 (3)

S1—C1—H1A	109.4	N2—C16—H16	118.7
C2—C1—H1B	109.4	C15—C16—H16	118.7
S1—C1—H1B	109.4	O1—C17—N3	123.2 (3)
H1A—C1—H1B	108.0	O1—C17—C15	119.9 (3)
C1—C2—S2	113.2 (3)	N3—C17—C15	116.9 (3)
C1—C2—H2A	108.9	O2—C18—N4	122.8 (4)
S2—C2—H2A	108.9	O2—C18—C10	119.6 (3)
C1—C2—H2B	108.9	N4—C18—C10	117.5 (3)
S2—C2—H2B	108.9	F5—P1—F1	90.8 (3)
H2A—C2—H2B	107.8	F5—P1—F4	92.9 (3)
C4—C3—S2	112.0 (3)	F1—P1—F4	176.0 (3)
C4—C3—H3D	109.2	F5—P1—F6	93.5 (3)
S2—C3—H3D	109.2	F1—P1—F6	90.8 (2)
C4—C3—H3E	109.2	F4—P1—F6	90.70 (19)
S2—C3—H3E	109.2	F5—P1—F3	89.8 (3)
H3D—C3—H3E	107.9	F1—P1—F3	90.4 (2)
C3—C4—S3	114.2 (3)	F4—P1—F3	87.8 (2)
C3—C4—H4D	108.7	F6—P1—F3	176.5 (2)
S3—C4—H4D	108.7	F5—P1—F2	178.8 (3)
C3—C4—H4E	108.7	F1—P1—F2	88.1 (2)
S3—C4—H4E	108.7	F4—P1—F2	88.3 (3)
H4D—C4—H4E	107.6	F6—P1—F2	86.9 (2)
C6—C5—S3	110.4 (3)	F3—P1—F2	89.9 (3)
C6—C5—H5A	109.6	H2W—O1W—H1W	146.2