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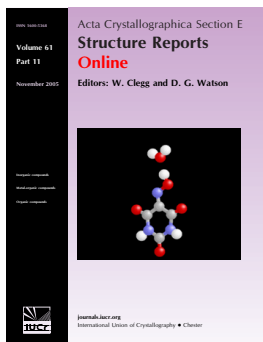
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(Glycol- $\kappa^2 O, O'$)nitrosyl(η^5 -pentamethylcyclopentadienyl)ruthenium(II)
bis(trifluoromethanesulfonate)

Semeret Munie, Anna Larsen and Milan Gembicky

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(Glycol- κ^2 O,O')nitrosyl(η^5 -pentamethylcyclopentadienyl)ruthenium(II) bis(trifluoromethanesulfonate)Semeret Munie,^a Anna Larsen^a and Milan Gembicky^{b*}^aDepartment of Chemistry, CNS 359, Ithaca College, Ithaca, NY 14850, USA, and^bDepartment of Chemistry, State University of New York at Buffalo, 732 NSC

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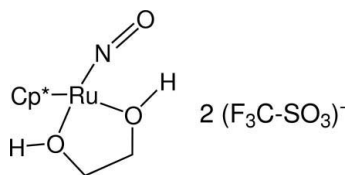
Received 11 December 2007; accepted 17 December 2007

Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.021; wR factor = 0.049; data-to-parameter ratio = 16.4.

The title compound, $[\text{Ru}(\text{C}_{10}\text{H}_{15})(\text{NO})(\text{HOCH}_2\text{CH}_2\text{OH})](\text{CF}_3\text{SO}_3)_2$, possesses a three-legged piano-stool geometry around the Ru atom, with an average Ru—O distance of 2.120 (6) Å and an Ru—N—O angle of 159.45 (14)°. The ethyleneglycol ligand forms a non-planar metallacyclic ring by chelating the Ru atom *via* the O atoms. The O...O distances of 2.554 (2) and 2.568 (2) Å are indicative of hydrogen bonding between coordinated ethyleneglycol and outer-sphere trifluoromethanesulfonate fragments. The crystal packing is stabilized by ionic forces and several $\text{CH}_3 \cdots \text{F}$ (2.585 and 2.640 Å) and $\text{CH}_3 \cdots \text{O}$ interactions (2.391, 2.678, 2.694 and 2.699 Å) between the pentamethylcyclopentadienyl ligand and trifluoromethanesulfonate anion. There is noticeable short intermolecular contact [2.9039 (16) Å], between an O atom of the SO_3 group and a C atom of the pentamethylcyclopentadienyl ligand.

Related literature

For closely related ruthenium diol- and alkyloxy-chelated structures, see: Hubbard & McVicar (1992); Yang *et al.* (1995, 1997). For chemically related complexes, see: Burns & Hubbard (1994); Pearsal *et al.* (2007); Svetlanova-Larsen *et al.* (1996).

**Experimental***Crystal data* $[\text{Ru}(\text{C}_{10}\text{H}_{15})(\text{NO})(\text{C}_2\text{H}_6\text{O}_2)]\cdot$ $(\text{CF}_3\text{O}_3\text{S})_2$ $M_r = 626.51$ Monoclinic, $P2_1/n$ $a = 8.5593$ (2) Å $b = 30.5443$ (7) Å $c = 8.8608$ (2) Å $\beta = 91.295$ (1)° $V = 2315.96$ (9) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.95$ mm⁻¹ $T = 90$ (1) K $0.20 \times 0.20 \times 0.04$ mm*Data collection*

Bruker SMART APEX2

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

 $T_{\min} = 0.833$, $T_{\max} = 0.963$

30259 measured reflections

5102 independent reflections

4513 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.027$ *Refinement* $R[F^2 > 2\sigma(F^2)] = 0.021$ $wR(F^2) = 0.049$ $S = 1.04$

5102 reflections

311 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.52$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O}2-H2 \cdots \text{O}5$	0.80 (3)	1.76 (3)	2.568 (2)	176 (3)
$\text{O}3-H3 \cdots \text{O}7$	0.80 (3)	1.76 (3)	2.554 (2)	169 (3)

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *pubCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2160).

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supplementary materials

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**(Glycol-²O,O')nitrosyl(⁵-pentamethylcyclopentadienyl)ruthenium(II)
bis(trifluoromethanesulfonate)**

S. Munie, A. Larsen and M. Gembicky

Comment

The electrophilic ruthenium center in trifluoromethanesulfonate complexes with a [(C₁₀H₁₅)Ru(NO)] core is reactive towards small molecular nucleophiles, such as water (Svetlanova-Larsen *et al.*, 1996), unsaturated hydrocarbons (Burns & Hubbard, 1994), and alcohols, and results in their binding and activation. For primary and secondary alcohols, after initial coordination, the reaction leads to alcohol oxidation and reduction of the ruthenium center to the Ru=O dimer, (Pearsal *et al.*, 2007). In the case of ethylene glycol, however, the coordination product is stabilized by a chelate ring formation and can be isolated and characterized.

Experimental

The compound was obtained as a product of reaction between Cp*Ru(NO)(SO₃CF₃)₂ and 3 equivalents of ethylene glycol in chloroform solution (Cp* = C₅Me₅). The single crystals for the X-ray diffraction studies were grown by solvent diffusion from a dichloromethane/hexane mixture at ambient temperature under inert atmosphere (Pearsal *et al.*, 2007).

All synthetic procedures were carried out in inert atmosphere. 10 μL of HO—CH₂CH₂—OH (0.213 mmol, 3 equiv) was added to a stirred solution of 40 mg of Cp*Ru(NO)(SO₃CF₃)₂ (0.071 mmol) in 10 ml of CHCl₃. A red precipitate formed within 5 min and the initially purple solution became almost colorless. The supernatant was decanted and the precipitate was recrystallized from a CH₂Cl₂/hexane mixture at -40 °C yielding 30 mg (0.050 mmol, 70%) of analytically pure complex[(C₁₀H₁₅)Ru(NO)(OH—CH₂—CH₂—OH)]²⁺ 2[SO₃CF₃]⁻. X-ray quality crystals were grown by slow diffusion of hexane into dichloromethane solution. The CHCl₃ solvent used in this reaction should be completely ethanol-free, otherwise isolation of the crystalline product becomes difficult due to the reaction between Cp*Ru(NO)(SO₃CF₃) and ethanol used routinely for stabilization of commercially available chloroform. The compound was characterized by ¹H, ¹⁹F, ¹³C NMR, and by IR spectroscopy. ¹H NMR(CH₂Cl₂): δ 1.93 (s) (15H, Cp*); δ 1.88 (s) (15H, Cp* minor amounts of starting material existing in equilibrium with product); δ 4.23 (broad) (2H, HO-CHH_a—CHH_a—OH); δ 3.35 (broad) (2H, HO—CH_bH—CH_bH—OH); δ 11.13 (2H, 2OH); ¹³C NMR (CH₂Cl₂): δ 119.8 (q, SO₃CF₃, JC—F = 318.4 Hz); δ 113.9 (C₅Me₅); δ 67.8 (HO—CH₂—CH₂—OH), δ 9.6 (C₅Me₅); ¹⁹F {¹H} NMR (CH₂Cl₂): δ -78.6; IR (nujol) νNO 1820 cm⁻¹ (*versus*); mp 131 °C; Anal. Calcd for C₁₄H₂₁NO₉RuS₂F₆ (626): C, 26.80; H, 3.40; N, 2.20; Found: C, 26.82; H, 3.43; N, 2.18.

Refinement

All non-hydrogen atoms were refined anisotropically. Positions of hydrogen atoms were found from difference Fourier maps, but placed in calculated position and refined in the riding approximation. CH₃ H atoms were treated as part of idealized methyl group with torsion angles from electron density. Displacement factors were assigned as *U*_{iso}=1.5*U*_{eq} (for CH~3~) and *U*_{iso}=1.2*U*_{eq} for the CH₂. Hydroxyl hydrogen atoms were freely refined.

Figures

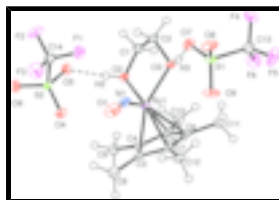


Fig. 1. , Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds shown in broken lines.

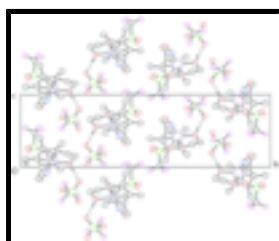


Fig. 2. A packing diagram of the title compound, showing the columns of Ru-complex and trifluoromethanesulfonate anion (coming out of the plane) along the *a*-axis direction. Only hydrogen atoms involved in H-bonding are present. Hydrogen bonds are shown as dashed lines.

(Glycol- κ^2O,O')nitrosyl(η^5 -pentamethylcyclopentadienyl)ruthenium(II) bis(trifluoromethanesulfonate)

Crystal data

[Ru(C₁₀H₁₅)(NO)(C₂H₆O₂)](CF₃O₃S)₂

$M_r = 626.51$

Monoclinic, $P2_1/n$

Hall symbol: -P2yn

$a = 8.5593$ (2) Å

$b = 30.5443$ (7) Å

$c = 8.8608$ (2) Å

$\beta = 91.295$ (1)°

$V = 2315.96$ (9) Å³

$Z = 4$

$F_{000} = 1256$

$D_x = 1.797$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6176 reflections

$\theta = 2.4$ – 27.1 °

$\mu = 0.95$ mm⁻¹

$T = 90$ (1) K

Plate, red

$0.20 \times 0.20 \times 0.04$ mm

Data collection

Bruker SMART APEX2
diffractometer

Radiation source: rotating anode

Monochromator: graphite

Detector resolution: 8.33 pixels mm⁻¹

$T = 90$ (1) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

$T_{\min} = 0.833$, $T_{\max} = 0.963$

30259 measured reflections

5102 independent reflections

4513 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\text{max}} = 27.1$ °

$\theta_{\text{min}} = 1.3$ °

$h = -10 \rightarrow 10$

$k = -39 \rightarrow 39$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.021$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0207P)^2 + 1.8018P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
5102 reflections	$(\Delta/\sigma)_{\max} = 0.003$
311 parameters	$\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Experimental. X-ray diffraction data on the title compound were collected at 90 (1) K using a Bruker *SMART APEX2* CCD diffractometer installed at a Rigaku rotating anode source (Mo K α radiation, $\lambda = 0.71073 \text{ \AA}$), and equipped with an Oxford Cryosystems nitrogen gas-flow apparatus. Data collection was performed with four runs at $\varphi = 0.00^\circ$, at $\varphi = 90.00^\circ$, at $\varphi = 180^\circ$ and at $\varphi = 270^\circ$ (600 frames each). Frame width = 0.30° in ω . Data were merged, corrected for decay, and treated with multi-scan absorption corrections, Bruker (2004).

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ru1	0.363831 (15)	0.628334 (4)	0.422226 (16)	0.01184 (4)
S1	0.79684 (5)	0.553701 (14)	0.22295 (5)	0.01778 (10)
S2	0.38389 (5)	0.708576 (14)	0.83367 (5)	0.01840 (10)
F1	0.64453 (14)	0.67755 (4)	0.72541 (14)	0.0311 (3)
F2	0.67642 (15)	0.72972 (4)	0.88629 (16)	0.0364 (3)
F3	0.61548 (16)	0.66532 (4)	0.96169 (15)	0.0373 (3)
F4	0.92738 (16)	0.58431 (5)	-0.01957 (15)	0.0393 (3)
F5	0.83784 (17)	0.51903 (5)	-0.04318 (17)	0.0461 (4)
F6	0.67972 (15)	0.57350 (4)	-0.04329 (14)	0.0350 (3)
O1	0.51407 (19)	0.58134 (5)	0.66923 (18)	0.0359 (4)
O2	0.37028 (15)	0.69766 (4)	0.44193 (15)	0.0160 (3)
H2	0.372 (3)	0.7089 (8)	0.524 (3)	0.038 (7)*
O3	0.54090 (15)	0.64795 (4)	0.27409 (15)	0.0183 (3)

supplementary materials

H3	0.611 (3)	0.6309 (8)	0.263 (3)	0.035 (7)*
O4	0.30874 (16)	0.66687 (4)	0.81175 (16)	0.0237 (3)
O5	0.38100 (16)	0.73629 (4)	0.69937 (15)	0.0219 (3)
O6	0.35026 (17)	0.73178 (4)	0.97014 (16)	0.0265 (3)
O7	0.78423 (16)	0.59968 (4)	0.26746 (16)	0.0252 (3)
O8	0.94083 (15)	0.53366 (4)	0.27126 (18)	0.0268 (3)
O9	0.65658 (16)	0.52870 (5)	0.24344 (18)	0.0311 (3)
N1	0.47625 (18)	0.60542 (5)	0.57437 (19)	0.0210 (3)
C1	0.4776 (2)	0.71981 (6)	0.3435 (2)	0.0193 (4)
H1A	0.5153	0.7473	0.3911	0.023*
H1B	0.4246	0.7271	0.2463	0.023*
C2	0.6118 (2)	0.68950 (6)	0.3177 (2)	0.0207 (4)
H2A	0.6785	0.7007	0.2365	0.025*
H2B	0.6765	0.6861	0.4111	0.025*
C3	0.16359 (19)	0.58780 (5)	0.4961 (2)	0.0152 (3)
C4	0.10739 (19)	0.62820 (6)	0.4356 (2)	0.0155 (3)
C5	0.15106 (19)	0.63035 (6)	0.2792 (2)	0.0148 (3)
C6	0.23367 (19)	0.59090 (5)	0.2443 (2)	0.0144 (3)
C7	0.24397 (19)	0.56501 (5)	0.3780 (2)	0.0139 (3)
C8	0.1359 (2)	0.57127 (6)	0.6516 (2)	0.0216 (4)
H8A	0.1364	0.5959	0.7225	0.032*
H8B	0.2187	0.5506	0.6808	0.032*
H8C	0.0344	0.5565	0.6537	0.032*
C9	0.0145 (2)	0.66126 (6)	0.5195 (2)	0.0203 (4)
H9A	-0.0942	0.6514	0.5248	0.030*
H9B	0.0183	0.6894	0.4668	0.030*
H9C	0.0586	0.6646	0.6219	0.030*
C10	0.1076 (2)	0.66649 (6)	0.1722 (2)	0.0194 (4)
H10A	0.1765	0.6657	0.0854	0.029*
H10B	0.1190	0.6947	0.2238	0.029*
H10C	-0.0010	0.6627	0.1373	0.029*
C11	0.3010 (2)	0.57971 (6)	0.0955 (2)	0.0188 (4)
H11A	0.3937	0.5613	0.1113	0.028*
H11B	0.3304	0.6067	0.0433	0.028*
H11C	0.2231	0.5638	0.0341	0.028*
C12	0.3081 (2)	0.51965 (5)	0.3901 (2)	0.0177 (4)
H12A	0.2255	0.4985	0.3648	0.027*
H12B	0.3467	0.5144	0.4935	0.027*
H12C	0.3942	0.5162	0.3200	0.027*
C13	0.8112 (2)	0.55776 (6)	0.0190 (2)	0.0236 (4)
C14	0.5906 (2)	0.69451 (6)	0.8531 (2)	0.0226 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru1	0.01021 (7)	0.00918 (6)	0.01600 (7)	-0.00059 (5)	-0.00226 (5)	0.00032 (5)
S1	0.0120 (2)	0.0172 (2)	0.0240 (2)	-0.00174 (16)	-0.00207 (17)	0.00336 (17)
S2	0.0227 (2)	0.0141 (2)	0.0184 (2)	-0.00311 (17)	0.00084 (18)	-0.00146 (17)

F1	0.0254 (6)	0.0365 (7)	0.0316 (7)	0.0005 (5)	0.0013 (5)	-0.0093 (5)
F2	0.0304 (7)	0.0261 (6)	0.0523 (9)	-0.0110 (5)	-0.0083 (6)	-0.0085 (6)
F3	0.0422 (8)	0.0333 (7)	0.0360 (7)	0.0061 (6)	-0.0080 (6)	0.0122 (6)
F4	0.0344 (7)	0.0521 (8)	0.0320 (7)	-0.0134 (6)	0.0114 (6)	0.0015 (6)
F5	0.0525 (9)	0.0368 (8)	0.0487 (9)	0.0094 (7)	-0.0059 (7)	-0.0262 (7)
F6	0.0314 (7)	0.0446 (8)	0.0286 (7)	0.0055 (6)	-0.0079 (5)	0.0077 (6)
O1	0.0407 (9)	0.0254 (8)	0.0406 (9)	-0.0040 (7)	-0.0226 (7)	0.0138 (7)
O2	0.0181 (6)	0.0113 (6)	0.0186 (7)	-0.0010 (5)	0.0008 (5)	-0.0002 (5)
O3	0.0142 (6)	0.0135 (6)	0.0274 (7)	-0.0029 (5)	0.0030 (5)	-0.0051 (5)
O4	0.0263 (7)	0.0178 (6)	0.0270 (7)	-0.0073 (5)	0.0003 (6)	-0.0023 (5)
O5	0.0304 (7)	0.0149 (6)	0.0204 (7)	0.0015 (5)	0.0002 (6)	-0.0003 (5)
O6	0.0361 (8)	0.0229 (7)	0.0206 (7)	-0.0018 (6)	0.0047 (6)	-0.0045 (6)
O7	0.0201 (7)	0.0249 (7)	0.0307 (8)	0.0029 (6)	0.0003 (6)	-0.0092 (6)
O8	0.0158 (7)	0.0213 (7)	0.0428 (9)	-0.0010 (5)	-0.0103 (6)	0.0069 (6)
O9	0.0161 (7)	0.0361 (8)	0.0410 (9)	-0.0091 (6)	-0.0044 (6)	0.0166 (7)
N1	0.0195 (8)	0.0156 (8)	0.0274 (9)	-0.0028 (6)	-0.0098 (7)	0.0008 (6)
C1	0.0215 (9)	0.0137 (8)	0.0227 (10)	-0.0035 (7)	0.0014 (7)	0.0017 (7)
C2	0.0166 (9)	0.0159 (9)	0.0296 (10)	-0.0072 (7)	0.0017 (8)	-0.0034 (7)
C3	0.0101 (8)	0.0140 (8)	0.0213 (9)	-0.0039 (6)	-0.0013 (7)	0.0010 (7)
C4	0.0094 (8)	0.0140 (8)	0.0230 (9)	-0.0024 (6)	-0.0024 (7)	-0.0004 (7)
C5	0.0110 (8)	0.0133 (8)	0.0200 (9)	-0.0022 (6)	-0.0047 (7)	0.0004 (7)
C6	0.0102 (8)	0.0129 (8)	0.0200 (9)	-0.0041 (6)	-0.0038 (7)	-0.0017 (7)
C7	0.0104 (8)	0.0111 (8)	0.0201 (9)	-0.0036 (6)	-0.0032 (7)	-0.0002 (6)
C8	0.0215 (10)	0.0214 (9)	0.0221 (10)	-0.0016 (7)	0.0037 (8)	0.0047 (8)
C9	0.0159 (9)	0.0185 (9)	0.0264 (10)	0.0026 (7)	0.0004 (8)	-0.0010 (7)
C10	0.0182 (9)	0.0161 (8)	0.0237 (10)	0.0010 (7)	-0.0052 (7)	0.0035 (7)
C11	0.0212 (9)	0.0168 (8)	0.0183 (9)	-0.0010 (7)	-0.0022 (7)	-0.0009 (7)
C12	0.0163 (9)	0.0115 (8)	0.0252 (10)	-0.0008 (7)	-0.0022 (7)	0.0018 (7)
C13	0.0199 (9)	0.0226 (9)	0.0283 (11)	0.0005 (8)	0.0001 (8)	-0.0047 (8)
C14	0.0261 (10)	0.0161 (9)	0.0255 (10)	-0.0049 (7)	-0.0031 (8)	-0.0022 (7)

Geometric parameters (Å, °)

Ru1—N1	1.7817 (16)	C1—H1B	0.9900
Ru1—O3	2.1139 (13)	C2—H2A	0.9900
Ru1—O2	2.1252 (12)	C2—H2B	0.9900
Ru1—C5	2.1961 (17)	C3—C4	1.425 (2)
Ru1—C4	2.2008 (17)	C3—C7	1.444 (2)
Ru1—C7	2.2201 (16)	C3—C8	1.492 (3)
Ru1—C3	2.2247 (17)	C4—C5	1.445 (3)
Ru1—C6	2.2257 (17)	C4—C9	1.494 (2)
S1—O8	1.4328 (14)	C5—C6	1.434 (2)
S1—O9	1.4379 (14)	C5—C10	1.497 (2)
S1—O7	1.4633 (14)	C6—C7	1.425 (2)
S1—C13	1.818 (2)	C6—C11	1.491 (3)
S2—O6	1.4364 (14)	C7—C12	1.493 (2)
S2—O4	1.4383 (13)	C8—H8A	0.9800
S2—O5	1.4601 (14)	C8—H8B	0.9800
S2—C14	1.825 (2)	C8—H8C	0.9800

supplementary materials

F1—C14	1.336 (2)	C9—H9A	0.9800
F2—C14	1.332 (2)	C9—H9B	0.9800
F3—C14	1.326 (2)	C9—H9C	0.9800
F4—C13	1.333 (2)	C10—H10A	0.9800
F5—C13	1.327 (2)	C10—H10B	0.9800
F6—C13	1.332 (2)	C10—H10C	0.9800
O1—N1	1.158 (2)	C11—H11A	0.9800
O2—C1	1.448 (2)	C11—H11B	0.9800
O2—H2	0.80 (3)	C11—H11C	0.9800
O3—C2	1.455 (2)	C12—H12A	0.9800
O3—H3	0.80 (3)	C12—H12B	0.9800
C1—C2	1.497 (3)	C12—H12C	0.9800
C1—H1A	0.9900		
N1—Ru1—O3	101.46 (7)	C3—C4—C9	125.28 (17)
N1—Ru1—O2	108.46 (6)	C5—C4—C9	126.77 (16)
O3—Ru1—O2	75.58 (5)	C3—C4—Ru1	72.14 (10)
N1—Ru1—C5	150.97 (7)	C5—C4—Ru1	70.63 (9)
O3—Ru1—C5	103.33 (6)	C9—C4—Ru1	124.63 (12)
O2—Ru1—C5	92.24 (6)	C6—C5—C4	107.92 (15)
N1—Ru1—C4	118.73 (7)	C6—C5—C10	126.86 (16)
O3—Ru1—C4	139.81 (6)	C4—C5—C10	125.12 (16)
O2—Ru1—C4	91.23 (6)	C6—C5—Ru1	72.20 (9)
C5—Ru1—C4	38.38 (7)	C4—C5—Ru1	70.99 (9)
N1—Ru1—C7	91.78 (7)	C10—C5—Ru1	125.25 (12)
O3—Ru1—C7	118.17 (6)	C7—C6—C5	107.99 (15)
O2—Ru1—C7	153.32 (6)	C7—C6—C11	126.16 (16)
C5—Ru1—C7	63.16 (6)	C5—C6—C11	125.82 (16)
C4—Ru1—C7	63.29 (6)	C7—C6—Ru1	71.09 (10)
N1—Ru1—C3	88.01 (7)	C5—C6—Ru1	69.95 (9)
O3—Ru1—C3	155.28 (6)	C11—C6—Ru1	123.16 (12)
O2—Ru1—C3	123.30 (6)	C6—C7—C3	108.26 (15)
C5—Ru1—C3	63.32 (6)	C6—C7—C12	126.20 (16)
C4—Ru1—C3	37.55 (6)	C3—C7—C12	125.11 (16)
C7—Ru1—C3	37.93 (6)	C6—C7—Ru1	71.52 (9)
N1—Ru1—C6	125.96 (7)	C3—C7—Ru1	71.21 (9)
O3—Ru1—C6	93.46 (6)	C12—C7—Ru1	128.90 (12)
O2—Ru1—C6	125.57 (6)	C3—C8—H8A	109.5
C5—Ru1—C6	37.85 (6)	C3—C8—H8B	109.5
C4—Ru1—C6	63.48 (6)	H8A—C8—H8B	109.5
C7—Ru1—C6	37.38 (6)	C3—C8—H8C	109.5
C3—Ru1—C6	62.99 (7)	H8A—C8—H8C	109.5
O8—S1—O9	116.77 (9)	H8B—C8—H8C	109.5
O8—S1—O7	113.43 (8)	C4—C9—H9A	109.5
O9—S1—O7	114.13 (9)	C4—C9—H9B	109.5
O8—S1—C13	104.41 (9)	H9A—C9—H9B	109.5
O9—S1—C13	103.72 (9)	C4—C9—H9C	109.5
O7—S1—C13	102.10 (9)	H9A—C9—H9C	109.5
O6—S2—O4	116.95 (9)	H9B—C9—H9C	109.5
O6—S2—O5	113.58 (8)	C5—C10—H10A	109.5

O4—S2—O5	113.84 (8)	C5—C10—H10B	109.5
O6—S2—C14	104.43 (9)	H10A—C10—H10B	109.5
O4—S2—C14	103.53 (9)	C5—C10—H10C	109.5
O5—S2—C14	102.23 (9)	H10A—C10—H10C	109.5
C1—O2—Ru1	115.58 (10)	H10B—C10—H10C	109.5
C1—O2—H2	110.5 (19)	C6—C11—H11A	109.5
Ru1—O2—H2	119.9 (19)	C6—C11—H11B	109.5
C2—O3—Ru1	112.50 (11)	H11A—C11—H11B	109.5
C2—O3—H3	106.7 (18)	C6—C11—H11C	109.5
Ru1—O3—H3	116.0 (18)	H11A—C11—H11C	109.5
O1—N1—Ru1	159.45 (14)	H11B—C11—H11C	109.5
O2—C1—C2	107.59 (14)	C7—C12—H12A	109.5
O2—C1—H1A	110.2	C7—C12—H12B	109.5
C2—C1—H1A	110.2	H12A—C12—H12B	109.5
O2—C1—H1B	110.2	C7—C12—H12C	109.5
C2—C1—H1B	110.2	H12A—C12—H12C	109.5
H1A—C1—H1B	108.5	H12B—C12—H12C	109.5
O3—C2—C1	105.25 (14)	F5—C13—F6	107.53 (16)
O3—C2—H2A	110.7	F5—C13—F4	107.46 (17)
C1—C2—H2A	110.7	F6—C13—F4	107.59 (17)
O3—C2—H2B	110.7	F5—C13—S1	111.59 (15)
C1—C2—H2B	110.7	F6—C13—S1	111.14 (14)
H2A—C2—H2B	108.8	F4—C13—S1	111.33 (14)
C4—C3—C7	107.89 (15)	F3—C14—F2	107.70 (16)
C4—C3—C8	125.55 (16)	F3—C14—F1	107.53 (16)
C7—C3—C8	126.49 (16)	F2—C14—F1	107.52 (16)
C4—C3—Ru1	70.32 (9)	F3—C14—S2	111.48 (14)
C7—C3—Ru1	70.86 (9)	F2—C14—S2	111.06 (13)
C8—C3—Ru1	126.86 (13)	F1—C14—S2	111.37 (13)
C3—C4—C5	107.92 (15)		
N1—Ru1—O2—C1	101.00 (13)	C6—Ru1—C5—C4	116.90 (14)
O3—Ru1—O2—C1	3.51 (11)	N1—Ru1—C5—C10	167.29 (15)
C5—Ru1—O2—C1	-99.68 (12)	O3—Ru1—C5—C10	-44.76 (16)
C4—Ru1—O2—C1	-138.07 (12)	O2—Ru1—C5—C10	30.94 (15)
C7—Ru1—O2—C1	-121.52 (15)	C4—Ru1—C5—C10	120.10 (19)
C3—Ru1—O2—C1	-158.89 (12)	C7—Ru1—C5—C10	-159.84 (18)
C6—Ru1—O2—C1	-80.33 (13)	C3—Ru1—C5—C10	157.47 (17)
N1—Ru1—O3—C2	-80.50 (13)	C6—Ru1—C5—C10	-123.0 (2)
O2—Ru1—O3—C2	25.85 (11)	C4—C5—C6—C7	-1.15 (18)
C5—Ru1—O3—C2	114.74 (12)	C10—C5—C6—C7	-177.64 (16)
C4—Ru1—O3—C2	100.18 (14)	Ru1—C5—C6—C7	61.24 (11)
C7—Ru1—O3—C2	-178.80 (11)	C4—C5—C6—C11	-179.44 (16)
C3—Ru1—O3—C2	168.68 (14)	C10—C5—C6—C11	4.1 (3)
C6—Ru1—O3—C2	151.73 (12)	Ru1—C5—C6—C11	-117.05 (17)
O3—Ru1—N1—O1	-137.2 (5)	C4—C5—C6—Ru1	-62.39 (11)
O2—Ru1—N1—O1	144.4 (5)	C10—C5—C6—Ru1	121.12 (17)
C5—Ru1—N1—O1	11.0 (6)	N1—Ru1—C6—C7	27.59 (13)
C4—Ru1—N1—O1	42.3 (5)	O3—Ru1—C6—C7	134.43 (10)
C7—Ru1—N1—O1	-17.9 (5)	O2—Ru1—C6—C7	-150.85 (9)

supplementary materials

C3—Ru1—N1—O1	19.8 (5)	C5—Ru1—C6—C7	-118.20 (14)
C6—Ru1—N1—O1	-34.3 (5)	C4—Ru1—C6—C7	-79.96 (11)
Ru1—O2—C1—C2	-30.53 (18)	C3—Ru1—C6—C7	-37.71 (10)
Ru1—O3—C2—C1	-49.10 (17)	N1—Ru1—C6—C5	145.78 (11)
O2—C1—C2—O3	49.83 (19)	O3—Ru1—C6—C5	-107.38 (10)
N1—Ru1—C3—C4	146.55 (11)	O2—Ru1—C6—C5	-32.66 (12)
O3—Ru1—C3—C4	-99.89 (16)	C4—Ru1—C6—C5	38.23 (10)
O2—Ru1—C3—C4	35.68 (13)	C7—Ru1—C6—C5	118.20 (15)
C5—Ru1—C3—C4	-38.20 (10)	C3—Ru1—C6—C5	80.49 (11)
C7—Ru1—C3—C4	-118.00 (15)	N1—Ru1—C6—C11	-93.83 (15)
C6—Ru1—C3—C4	-80.83 (11)	O3—Ru1—C6—C11	13.01 (14)
N1—Ru1—C3—C7	-95.46 (11)	O2—Ru1—C6—C11	87.72 (15)
O3—Ru1—C3—C7	18.11 (19)	C5—Ru1—C6—C11	120.38 (18)
O2—Ru1—C3—C7	153.68 (9)	C4—Ru1—C6—C11	158.62 (16)
C5—Ru1—C3—C7	79.80 (11)	C7—Ru1—C6—C11	-121.42 (18)
C4—Ru1—C3—C7	118.00 (15)	C3—Ru1—C6—C11	-159.13 (16)
C6—Ru1—C3—C7	37.17 (10)	C5—C6—C7—C3	1.59 (18)
N1—Ru1—C3—C8	26.31 (16)	C11—C6—C7—C3	179.87 (16)
O3—Ru1—C3—C8	139.88 (15)	Ru1—C6—C7—C3	62.10 (11)
O2—Ru1—C3—C8	-84.55 (16)	C5—C6—C7—C12	174.32 (16)
C5—Ru1—C3—C8	-158.44 (17)	C11—C6—C7—C12	-7.4 (3)
C4—Ru1—C3—C8	-120.2 (2)	Ru1—C6—C7—C12	-125.16 (17)
C7—Ru1—C3—C8	121.77 (19)	C5—C6—C7—Ru1	-60.52 (11)
C6—Ru1—C3—C8	158.94 (17)	C11—C6—C7—Ru1	117.77 (17)
C7—C3—C4—C5	0.71 (19)	C4—C3—C7—C6	-1.43 (19)
C8—C3—C4—C5	-176.24 (16)	C8—C3—C7—C6	175.48 (16)
Ru1—C3—C4—C5	61.94 (11)	Ru1—C3—C7—C6	-62.30 (11)
C7—C3—C4—C9	178.56 (16)	C4—C3—C7—C12	-174.26 (16)
C8—C3—C4—C9	1.6 (3)	C8—C3—C7—C12	2.6 (3)
Ru1—C3—C4—C9	-120.22 (17)	Ru1—C3—C7—C12	124.86 (17)
C7—C3—C4—Ru1	-61.23 (11)	C4—C3—C7—Ru1	60.88 (11)
C8—C3—C4—Ru1	121.83 (17)	C8—C3—C7—Ru1	-122.21 (18)
N1—Ru1—C4—C3	-38.92 (13)	N1—Ru1—C7—C6	-157.97 (11)
O3—Ru1—C4—C3	140.32 (10)	O3—Ru1—C7—C6	-53.96 (11)
O2—Ru1—C4—C3	-150.82 (11)	O2—Ru1—C7—C6	61.92 (17)
C5—Ru1—C4—C3	117.13 (14)	C5—Ru1—C7—C6	37.30 (10)
C7—Ru1—C4—C3	37.41 (10)	C4—Ru1—C7—C6	80.51 (11)
C6—Ru1—C4—C3	79.42 (11)	C3—Ru1—C7—C6	117.56 (14)
N1—Ru1—C4—C5	-156.05 (10)	N1—Ru1—C7—C3	84.47 (11)
O3—Ru1—C4—C5	23.19 (14)	O3—Ru1—C7—C3	-171.52 (9)
O2—Ru1—C4—C5	92.06 (10)	O2—Ru1—C7—C3	-55.63 (17)
C7—Ru1—C4—C5	-79.71 (11)	C5—Ru1—C7—C3	-80.25 (11)
C3—Ru1—C4—C5	-117.13 (14)	C4—Ru1—C7—C3	-37.04 (10)
C6—Ru1—C4—C5	-37.70 (10)	C6—Ru1—C7—C3	-117.56 (14)
N1—Ru1—C4—C9	82.06 (16)	N1—Ru1—C7—C12	-35.94 (17)
O3—Ru1—C4—C9	-98.70 (16)	O3—Ru1—C7—C12	68.07 (17)
O2—Ru1—C4—C9	-29.83 (15)	O2—Ru1—C7—C12	-176.04 (13)
C5—Ru1—C4—C9	-121.89 (19)	C5—Ru1—C7—C12	159.34 (18)
C7—Ru1—C4—C9	158.40 (18)	C4—Ru1—C7—C12	-157.45 (18)

C3—Ru1—C4—C9	121.0 (2)	C3—Ru1—C7—C12	-120.4 (2)
C6—Ru1—C4—C9	-159.59 (17)	C6—Ru1—C7—C12	122.0 (2)
C3—C4—C5—C6	0.26 (19)	O8—S1—C13—F5	56.05 (16)
C9—C4—C5—C6	-177.54 (16)	O9—S1—C13—F5	-66.73 (16)
Ru1—C4—C5—C6	63.17 (11)	O7—S1—C13—F5	174.41 (14)
C3—C4—C5—C10	176.83 (16)	O8—S1—C13—F6	176.07 (13)
C9—C4—C5—C10	-1.0 (3)	O9—S1—C13—F6	53.29 (16)
Ru1—C4—C5—C10	-120.26 (17)	O7—S1—C13—F6	-65.57 (15)
C3—C4—C5—Ru1	-62.91 (12)	O8—S1—C13—F4	-64.01 (15)
C9—C4—C5—Ru1	119.29 (17)	O9—S1—C13—F4	173.21 (14)
N1—Ru1—C5—C6	-69.71 (18)	O7—S1—C13—F4	54.35 (15)
O3—Ru1—C5—C6	78.24 (10)	O6—S2—C14—F3	67.93 (15)
O2—Ru1—C5—C6	153.94 (10)	O4—S2—C14—F3	-54.97 (15)
C4—Ru1—C5—C6	-116.90 (14)	O5—S2—C14—F3	-173.49 (13)
C7—Ru1—C5—C6	-36.85 (10)	O6—S2—C14—F2	-52.17 (16)
C3—Ru1—C5—C6	-79.53 (11)	O4—S2—C14—F2	-175.08 (14)
N1—Ru1—C5—C4	47.19 (18)	O5—S2—C14—F2	66.40 (15)
O3—Ru1—C5—C4	-164.86 (9)	O6—S2—C14—F1	-171.97 (13)
O2—Ru1—C5—C4	-89.15 (10)	O4—S2—C14—F1	65.12 (15)
C7—Ru1—C5—C4	80.06 (10)	O5—S2—C14—F1	-53.40 (14)
C3—Ru1—C5—C4	37.37 (10)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O2—H2...O5	0.80 (3)	1.76 (3)	2.568 (2)	176 (3)
O3—H3...O7	0.80 (3)	1.76 (3)	2.554 (2)	169 (3)

Fig. 2

