Electronic Supplementary Material (ESI) for Dalton Transactions.
This journal is © The Royal Society of Chemistry 2015

Supporting Information:

Fixation of atmospheric carbon dioxide by ruthenium complexes bearing an NHC-based pincer ligand: formation of a methylcarbonato complex and its methylation

Yasuhiro Arikawa,* Takuo Nakamura, Shinji Ogushi, Kazushige Eguchi and Keisuke Umakoshi

Division of Chemistry and Materials Science, Graduate School of Engineering, Nagasaki University, Bunkyo-machi 1-14, Nagasaki 852-8521, Japan.

Experimental Procedures

General Procedures: All reactions and subsequent work-up manipulations were performed in air unless otherwise noted. Organic solvents and all other reagents were commercially available and used without further purification. Deaerated solvents were prepared from bubbling of Ar into the solution for ca. 20 min. NMR spectra in CD_3CN were recorded on a Varian Gemini-300 and a JEOL JNM-AL-400 spectrometers. ^1H and $^{13}C\{^1H\}$ NMR chemical shifts are quoted with respect to the solvent signals. Infrared spectra in KBr pellets were obtained on JASCO FT-IR-4100 spectrometers. Electrospray mass spectroscopies (ESI-MS) were carried out on a Waters ACQUITY SQD MS system. Elemental analyses (C, H, N) were performed on a Perkin Elmer 2400II elemental analyzer. $H[Ru(bpy)Cl_4]$[1] and the carbene ligand (CNC) precursor (pyridine-bridged bis(imidazolium) chloride)[2] were prepared according to literature methods.

Preparation of $[(CNC)Ru(bpy)Cl]PF_6$ (1): This complex was prepared according to a literature method[3] with slight modifications. A mixture of $H[Ru(bpy)Cl_4]$ (300 mg, 0.750 mmol) and 2,6-bis(3-*tert*-butylimidazolium-1-yl)pyridine dichloride (327 mg, 0.825 mmol) in deaerated ethylene glycol (15 mL) was heated at 190 ºC under Ar for 90 min. After concentration of the mixture, NH_4PF_6 (489 mg, 3.00 mmol) in H_2O (10 mL) was added. The precipitated brown powder was filtered, washed with H_2O, and dried. The resulting brown solid was then refluxed

with Zn (65.7 mg, 1.00 mmol) in MeOH (40 mL) under Ar for 1 h. The mixture was cooled to room temperature and evaporated to dryness. The residue was column-chromatographed with a silica gel, eluting with CH_2Cl_2/MeOH (30/1 v/v) to give a red brown solid, followed by washing with a small amount of MeOH to afford [(CNC)Ru(bpy)Cl]PF_6 (**1**) (230 mg, 40%). From the washings, a trace amount of [$(CNC)_2$Ru]$(PF_6)_2$ was obtained.

1: IR (KBr, pellet): ν(PF) 847 (s) cm^{-1}. ^{1}H NMR (CD_3CN): δ 10.02 (d, *J* = 5.8 Hz, 1H, bpy), 8.40 (d, *J* = 7.8 Hz, 1H, bpy), 8.19 (d, *J* = 7.9 Hz, 1H, bpy), 8.10 (t, *J* = 8.2 Hz, 1H, 4-py), 8.03 (ddd, *J* = 8.1, 7.6, 1.5 Hz, 1H, bpy), 7.99 (d, *J* = 2.4 Hz, 2H, imid), 7.79–7.74 (m, 3H, 3,5-py + bpy), 7.59 (ddd, *J* = 8.1, 7.5, 1.4 Hz, 1H, bpy), 7.32 (d, *J* = 2.4 Hz, 2H, imid), 7.26 (d, *J* = 5.7 Hz, 1H, bpy), 6.89 (ddd, *J* = 7.4, 5.9, 1.5 Hz, 1H, bpy), 1.07 (s, 18H, ^{t}Bu). ^{13}C{^{1}H} NMR (CD_3CN): 191.8 (Ru-C_{NHC}), 158.9 (bpy), 158.7 (bpy), 155.6 (bpy), 155.1 (2,6-py), 152.7 (bpy), 139.2 (4-py), 135.9 (bpy), 134.9 (bpy), 126.2 (bpy), 126.2 (bpy), 123.7 (bpy), 123.7 (bpy), 122.3 (imid), 116.5 (imid), 107.0 (3,5-py), 58.8 (CMe$_3$), 31.5 (CMe_3). ESI-MS (*m/z*): 616.2 $[M–PF_6]^+$. Elemental analysis (%) calcd for $C_{29}H_{33}N_7ClRuPF_6$: C 45.76; H 4.37, N 12.88; Found: C 45.60; H 4.20, N 12.78.

[$(CNC)_2$Ru]$(PF_6)_2$: IR (KBr, pellet): ν(PF) 844 (s) cm^{-1}. ^{1}H NMR (CD_3CN): δ 8.27 (t, *J* = 8.2 Hz, 2H, 4-py), 8.04 (d, *J* = 2.5 Hz, 4H, imid), 7.95 (d, *J* = 8.2 Hz, 4H, 3,5-py), 7.34 (d, *J* = 2.4, 4H, imid), 0.71 (s, 36H, ^{t}Bu). ^{13}C{^{1}H} NMR (CD_3CN): 185.4 (Ru-C_{NHC}), 153.2 (2,6-py), 139.7 (4-py), 124.2 (imid), 116.5 (imid), 108.2 (3,5-py), 58.7 (CMe$_3$), 30.1 (CMe_3). ESI-MS (*m/z*): 893.2 $[M\text{-}PF_6]^+$, 374.1 $[M\text{-}(PF_6)_2]^{2+}$. Elemental analysis (%) calcd for $C_{38}H_{50}N_{10}RuP_2F_{12}\cdot C_4H_{10}O$: C 45.36; H 5.44, N 12.60; Found: C 45.31; H 5.39, N 12.85.

Preparation of [(CNC)Ru(bpy)(OMe)]PF_6 (2): To a solution of [(CNC)Ru(bpy)Cl]PF_6 (**1**) (38.7 mg, 0.0508 mmol) in deaerated MeOH (10 mL) was added a NaOMe solution (4.0 mL, 2.0 mmol; 0.5 M in MeOH) under Ar. The mixture was refluxed under Ar for 24 h and evacuated to dryness. In a glovebox, the residue was extracted with MeCN, and the extract was evacuated to dryness. Crystallization from MeCN/ether gave [(CNC)Ru(bpy)(OMe)]PF_6 (**2**) as dark brown crystals (25 mg, 65%).

2: IR (KBr, pellet): ν(PF) 845 (s) cm^{-1}. ^{1}H NMR (CD_3CN): δ 9.77 (d, *J* = 5.8 Hz, 1H, bpy), 8.36 (d, *J* = 8.0 Hz, 1H, bpy), 8.15 (d, *J* = 8.0 Hz, 1H, bpy), 8.04–7.96 (m, 2H, 4-py + bpy), 7.99 (d, *J* = 2.3 Hz, 2H, imid), 7.79 (ddd, *J* = 7.4, 5.8, 1.5 Hz, 1H, bpy), 7.77 (d, *J* = 8.2 Hz, 2H, 3,5-py), 7.49

(ddd, J = 8.1, 7.5, 1.4 Hz, 1H, bpy), 7.31 (d, J = 2.3 Hz, 2H, imid), 6.99 (d, J = 5.8 Hz,, 1H, bpy), 6.80 (ddd, J = 7.4, 5.8, 1.5 Hz, 1H, bpy), 2.38 (s, 3H, OMe), 1.06 (s, 18H, ^{t}Bu). $^{13}C\{^1H\}$ NMR (CD_3CN): 192.7 (Ru-C_{NHC}), 158.6 (bpy), 158.3 (bpy), 154.9 (2,6-py), 153.3 (bpy), 151.8 (bpy), 136.3 (4-py), 135.3 (bpy), 133.8 (bpy), 126.0 (bpy), 125.4 (bpy), 123.5 (bpy), 123.3 (bpy), 121.8 (imid), 116.0 (imid), 106.4 (3,5-py), 60.5 (O*Me*), 58.5 (*C*Me$_3$), 31.4 (C*Me*$_3$). ESI-MS (*m/z*): 612.3 $[M–PF_6]^+$. Elemental analysis (%) calcd for $C_{30}H_{36}N_7ORuPF_6$: C 47.62; H 4.80, N 12.96; Found: C 47.20; H 5.06, N 12.57.

Preparation of [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (3):

From complex **1**; A KOH solution (0.5 mL, 0.05 mmol; 0.1 M in MeOH) was added to a solution of [(CNC)Ru(bpy)Cl]PF$_6$ (**1**) (38.2 mg, 0.0502 mmol) in MeOH/H_2O (9/1 v/v, 20 mL). In air, the mixture was stirred for 24 h at room temperature. After evaporation, the residue was crystallized from MeOH/ether to give [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (**3**) as dark red crystals (22.3 mg, 56%).

From complex **2** (in air); In air, a MeCN (20 mL) solution of [(CNC)Ru(bpy)(OMe)]PF$_6$ (**2**) (19.1 mg, 0.0252 mmol) was stirred for 1 h at room temperature. After evaporation, the residue was crystallized from MeCN/ether to give [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (**3**) as dark red crystals (17.2 mg, 85%).

From complex **2** (CO_2 bubbling); After bubbling of CO_2 into a MeCN (20 mL) solution of [(CNC)Ru(bpy)(OMe)]PF$_6$ (**2**) (19.0 mg, 0.0251 mmol) for 20 min, the resulting solution was evaporated to dryness. Crystallization from MeCN/ether gave [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (**3**) as dark red crystals (16.1 mg, 80%).

From complex **4**; The mixture of [(CNC)Ru(bpy)(NCMe)](PF$_6$)$_2$ (**4**) (22.8 mg, 0.025 mmol) and a KOH solution (0.5 mL, 0.05 mmol; 0.1 M in MeOH) in MeOH (20 mL) was stirred at room temperature in air for 41 h. After evaporation, the residue was extracted with MeCN and filtered. Crystallization from MeOH/ether gave [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (**3**) as dark red crystals (19.3 mg, 96%).

3: IR (KBr, pellet): ν(PF) 847 (s), ν(CO) 1649 (s) cm^{-1}. ^{1}H NMR (CD_3CN): δ 9.65 (d, J = 5.2 Hz, 1H, bpy), 8.42 (d, J = 8.1 Hz, 1H, bpy), 8.18 (d, J = 8.1 Hz, 1H, bpy), 8.09 (t, J = 8.2 Hz, 1H, 4-py), 8.05 (ddd, J = 7.8, 7.8, 1.3 Hz, 1H, bpy), 7.98 (d, J = 2.3 Hz, 2H, imid), 7.87 (ddd, J = 7.3, 5.9, 1.3

Hz, 1H, bpy), 7.76 (d, 8.2 Hz, 2H, 3,5-py), 7.59–7.53 (m, 1H, bpy), 7.30 (d, J = 2.4 Hz, 2H, imid), 7.02 (d, J = 5.6 Hz, 1H, bpy), 6.89 (ddd, J = 7.4, 5.9, 1.4 Hz, 1H, bpy), 3.05 (s, 3H, OMe), 1.05 (s, 18H, ^{t}Bu). $^{13}C\{^1H\}$ NMR (CD_3CN): 191.5 (Ru-C_{NHC}), 160.1 (O*C*O), 158.7 (bpy), 158.2 (bpy), 155.6 (2,6-py), 153.2 (bpy), 152.3 (bpy), 138.8 (4-py), 136.0 (bpy), 134.5 (bpy), 126.3 (bpy), 126.3 (bpy), 123.9 (bpy), 123.6 (bpy), 122.0 (imid), 116.5 (imid), 106.7 (3,5-py), 58.6 (*C*Me$_3$), 53.0 (O*Me*), 31.2 (C*Me*$_3$). ESI-MS (*m/z*): 656.4 $[M–PF_6]^+$. Elemental analysis (%) calcd for $C_{31}H_{36}N_7O_3RuPF_6$: C 46.50, H 4.53, N 12.25; Found: C 46.05, H 4.38, N 11.77.

Preparation of [(CNC)Ru(bpy)(NCMe)](PF$_6$)$_2$ (4): To a solution of [(CNC)Ru(bpy)Cl]PF_6 (**1**) (38.1 mg, 0.050 mmol) in MeCN (10 mL) was added $AgPF_6$ (15.2 mg, 0.060 mmol). After refluxing for 3 h, the mixture was filtrated and evaporated to dryness. The crude product was crystallized by slow diffusion of Et_2O into a CH_3CN solution to give [(CNC)Ru(bpy)(NCMe)](PF$_6$)$_2$ (**4**) as orange crystals (44.8 mg, 98%).

4: IR (KBr, pellet): ν(PF) 845 (s) cm^{-1}. ^1H NMR (CD_3CN): δ 9.49 (d, J = 5.7 Hz, 1H, bpy), 8.47 (d, J = 7.9 Hz, 1H, bpy), 8.32–8.28 (m, 2H, 4-py + bpy), 8.14 (ddd, J = 8.2, 7.7, 1.4 Hz, 1H, bpy), 8.08 (d, J = 2.4 Hz, 2H, imid), 7.90 (d, J = 8.2 Hz, 2H, 3,5-py), 7.83 (ddd, J = 7.4, 5.9, 1.5 Hz, 1H, bpy), 7.76 (ddd, J = 8.2, 7.5, 1.5 Hz, 1H, bpy), 7.40 (d, J = 2.4 Hz, 2H, imid), 7.13 (d, J = 5.8 Hz, 1H, bpy), 7.06 (ddd, J = 7.4, 5.9, 1.4 Hz, 1H, bpy), 2.08 (s, 3H, NC*Me*), 1.08 (s, 18H, ^{t}Bu). $^{13}C\{^1H\}$ NMR (CD_3CN): 186.7 (Ru-C_{NHC}), 158.0 (bpy), 157.2 (bpy), 155.5 (bpy), 154.9 (2,6-py), 151.6 (bpy), 142.2 (4-py), 137.6 (bpy), 137.2 (bpy), 127.2 (bpy), 127.2 (bpy), 124.8 (bpy), 124.2 (bpy), 123.0 (imid), 117.5 (imid), 108.4 (3,5-py), 59.2 (*C*Me$_3$), 31.3 (N*C*Me), 31.1 (C*Me*$_3$), 4.6 (NC*Me*). ESI-MS (*m/z*): 767.3 $[M–PF_6]^+$, 311.2 $[M–(PF_6)_2]^{2+}$. Elemental analysis (%) calcd for $C_{31}H_{36}N_8RuP_2F_{12}$: C 40.84; H 3.98, N 12.29; Found: C 40.97; H 4.01, N 12.09.

Treatments of [(CNC)Ru(bpy){OC(O)OMe}]PF$_6$ (3) with methylating reagents:

MeI: A J. Young NMR tube was charged with [(CNC)Ru(bpy){OC(O)OMe}]PF_6 (**3**) (8.0 mg, 0.010 mmol) and ferrocene (*ca*. 0.2 mg) as an internal standard in CD_3CN (0.6 mL), and MeI (12.5 μL, 0.20 mmol) was added. The reaction was completed at room temperature in 15 days. The ^1H NMR revealed the formation of dimethylcarbonate (15%) and a CD_3CN coordinated complex

$[(CNC)Ru(bpy)(NCCD_3)]^{2+}$, along with a trace amount of the iodide analogue $[(CNC)Ru(bpy)I]^{+}$.

MeOTf: A J. Young NMR tube was charged with $[(CNC)Ru(bpy)\{OC(O)OMe\}]PF_6$ (**3**) (8.1 mg, 0.010 mmol) and hexamethylbenzene as an internal standard in CD_3CN (0.6 mL), and MeOTf (5.5 μL, 0.050 mmol) was added. The reaction was immediately completed. The ^1H NMR revealed the formation of dimethylcarbonate (82%) and the CD_3CN coordinated complex $[(CNC)Ru(bpy)(NCCD_3)]^{2+}$.

X-ray Crystal Structure Determinations: Crystallographic data are summarized in Table S1. X-ray quality single crystals were obtained from MeOH/ether (for **1** and $[(CNC)_2Ru](PF_6)_2 \cdot Et_2O$), acetone/ether (for **2**·MeCOMe·0.5Et_2O), and CH_3CN/ether (for **3**·Et_2O and **4**·MeCN), respectively. Diffraction data were collected at -180 ºC under a stream of cold dinitrogen gas on a Rigaku RA-Micro7 HFM instrument equipped with a Rigaku Saturn724+ CCD detector by using graphite-monochromated Mo Kα radiation. The intensity images were obtained at the exposure of 8.0 s/º. The frame data were integrated using a Rigaku CrystalClear program package, and the data sets were corrected for absorption using a REQAB program.

The calculations were performed with a CrystalStructure software package. The structures were solved by direct methods, and refined on F^2 by the full-matrix least squares methods. For $[(CNC)_2Ru](PF_6)_2 \cdot Et_2O$ and **4**·MeCN, there are two ruthenium complexes, four PF_6^- anion, and two crystal solvents in the asymmetrical unit, respectively. The ether crystal solvent in **2**·MeCOMe·0.5Et_2O is disordered over two positions with an occupancy factor of 0.5 / 0.5. For **3**·Et_2O, restraints were applied to ether atoms of the crystal solvent. Anisotropic refinement for all structures was applied to all non-hydrogen atoms with the exception of the ether crystal solvents of **2**·MeCOMe·0.5Et_2O and **3**·Et_2O. Hydrogen atoms of all structures were put at calculated positions, and the ether crystal solvent molecules of **2**·MeCOMe·0.5Et_2O and **3**·Et_2O were not included in the calculations.

Table S1. Crystallographic data for [(CNC)Ru(bpy)Cl]PF_6 (**1**), [$(CNC)_2$Ru]$(PF_6)_2$·Et_2O, [(CNC)Ru(bpy)(OMe)]PF_6 (**2**)·MeCOMe·0.5Et_2O, [(CNC)Ru(bpy){OC(O)OMe}]PF_6 (**3**)·Et_2O, and [(CNC)Ru(bpy)($NCCH_3$)]$(PF_6)_2$ (**4**)·MeCN

	1	[$(CNC)_2$Ru]$(PF_6)_2$·Et_2O	**2**·MeCOMe·0.5Et_2O
formula	$C_{29}H_{33}N_7ClF_6PRu$	$C_{42}H_{60}N_{10}F_{12}OP_2Ru$	$C_{35}H_{42}N_7F_6O_{2.5}PRu$
fw	761.11	1112.00	846.80
cryst system	triclinic	triclinic	triclinic
space group	*P* -1 (No. 2)	*P* -1 (No. 2)	*P* -1 (No. 2)
color of crystal	red	orange	red
crystal size (mm)	0.19 x 0.16 x 0.08	0.17 x 0.16 x 0.10	0.15 x 0.13 x 0.08
a (Å)	9.601(2)	12.674(2)	10.495(3)
b (Å)	12.173(2)	19.858(3)	12.179(4)
c (Å)	13.646(2)	20.062(3)	15.393(5)
α (deg)	80.999(5)	83.598(5)	83.468(9)
β (deg)	83.265(5)	80.720(4)	85.291(11)
γ (deg)	83.885(5)	81.683(4)	79.745(10)
V (Å^3)	1558.0(5)	4910.5(11)	1919.4(9)
Z	2	4	2
ρ_{calc} (g cm^{-3})	1.622	1.504	1.465
μ (cm^{-1})	7.097	4.747	5.217
$2\theta_{max}$ (deg)	55.0	55.0	54.9
no. of all reflns collected	13019	41253	23930
no. of unique reflns	7047	22227	8724
R_{int}	0.0197	0.0423	0.0431
no. of obsd reflns [a]	5901	16199	7331
no. of parameters	412	1253	474
R_1 [a, b]	0.0270	0.0752	0.0600
Rw (all data)[c]	0.0668	0.1800	0.1463
GOF (all data)[d]	1.043	1.088	1.060

[a] $I > 2\sigma(I)$. [b] $R_1 = \Sigma\, ||Fo| - |Fc||/\Sigma\, |Fo|$. [c] $Rw = \{\Sigma w\, (Fo^2 - Fc^2)^2/\Sigma w(Fo^2)^2\}^{1/2}$.

[d] GOF = $[\{\Sigma w(Fo^2 - Fc^2)^2\}/(No - Np)]^{1/2}$, where *No* and *Np* denote the number of data and parameters.

Table S1. (Continued)

	3·Et_2O	**4**·MeCN
formula	$C_{35}H_{36}N_7F_6O_4PRu$	$C_{33}H_{39}F_{12}N_9P_2Ru$
fw	864.75	952.73
cryst system	monoclinic	monoclinic
space group	$P2_1/n$ (No. 14)	$P2_1/c$ (No. 14)
color of crystal	red	yellow
crystal size (mm)	0.15 x 0.13 x 0.07	0.12 x 0.10 x 0.05
a (Å)	14.470(3)	24.816(5)
b (Å)	16.057(3)	17.489(3)
c (Å)	16.622(3)	19.357(4)
α (deg)	90	90
β (deg)	97.887(3)	110.388(2)
γ (deg)	90	90
V (Å^3)	3825.5(12)	7875(3)
Z	4	8
ρ_{calc} (g cm^{-3})	1.501	1.607
μ (cm^{-1})	5.282	5.749
$2\theta_{max}$ (deg)	55.0	55.0
no. of all reflns collected	31195	65122
no. of unique reflns	8737	18012
R_{int}	0.0483	0.0704
no. of obsd reflns [a]	7078	14312
no. of parameters	469	1043
R_1 [a, b]	0.0797	0.0746
Rw (all data)[c]	0.2162	0.1826
GOF (all data)[d]	1.087	1.114

[a] $I > 2\sigma(I)$. [b] $R_1 = \Sigma \, ||Fo| - |Fc|| / \Sigma \, |Fo|$. [c] $Rw = \{\Sigma w \, (Fo^2 - Fc^2)^2 / \Sigma w (Fo^2)^2\}^{1/2}$.

[d] GOF = $[\{\Sigma w(Fo^2 - Fc^2)^2\}/(No - Np)]^{1/2}$, where No and Np denote the number of data and parameters.

Figure S1. X-ray crystal structure of the cation part of [(CNC)Ru(bpy)Cl]PF_6 (**1**). Hydrogen atoms have been omitted for clarity. Selected bond distances [Å] and angle [°]: Ru-Cl 2.4310(6), Ru-N1 2.0046(17), Ru-N6 2.0402(16), Ru-N7 2.0640(17), Ru-C1 2.1190(19), Ru-C2 2.1201(18); C1-Ru-C2 155.00(8).

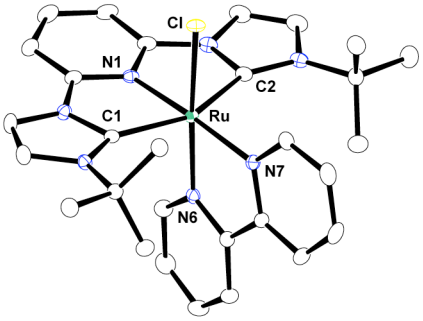


Figure S2. One of the two independent molecules [$(CNC)_2$Ru]$(PF_6)_2$ (the cation part) in the unit cell. Hydrogen atoms have been omitted for clarity. Selected bond distances [Å] and angle [°]: Ru-N1 2.028(4), Ru-N6 2.017(4), Ru-C1 2.134(5), Ru-C2 2.124(4), Ru-C20 2.123(5), Ru-C21 2.135(6); C1-Ru-C2 153.80(17), C20-Ru-C21 154.02(18).

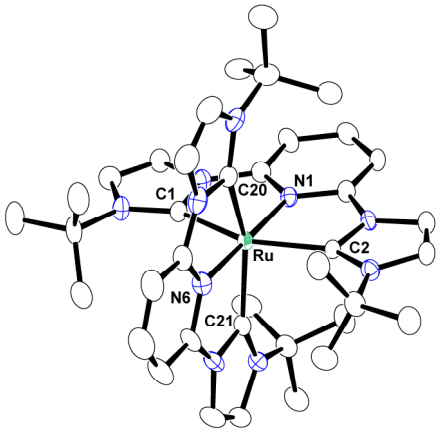


Figure S3. X-ray crystal structure of the cation part of [(CNC)Ru(bpy)(OMe)]PF_6 (**2**). Hydrogen atoms except for the methyl group have been omitted for clarity. Selected bond distances [Å] and angle [°]: Ru-O1 2.121(3), Ru-N1 2.006(3), Ru-N6 2.056(4), Ru-N7 2.066(3), Ru-C1 2.142(4), Ru-C2 2.116(4), O1-C30 1.313(7); C1-Ru-C2 155.15(15).

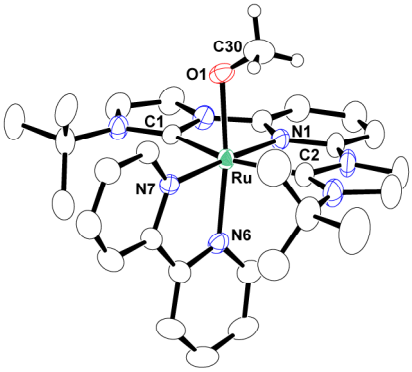
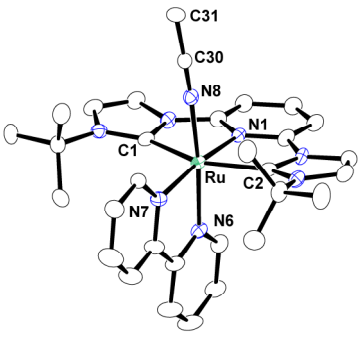


Figure S4. One of the two independent molecules [(CNC)Ru(bpy)($NCCH_3$)]$(PF_6)_2$ (**4**) (the cation part) in the unit cell. Hydrogen atoms have been omitted for clarity. Selected bond distances [Å] and angle [°]: Ru-N1 2.019(5), Ru-N6 2.057(4), Ru-N7 2.081(5), Ru-N8 2.027(4), Ru-C1 2.128(5), Ru-C2 2.134(5), N8-C30 1.140(6), C30-C31 1.464(6); C1-Ru-C2 154.9(2).



References

1. R. A. Krause, *Inorg. Chim. Acta*, 1977, **22**, 209-213.
2. D. J. Nielsen, K. J. Cavell, B. W. Skelton and A. H. White, *Inorg. Chim. Acta*, 2006, **359**, 1855-1869.
3. L.-H. Chung, K.-S. Cho, J. England, S.-C. Chan, K. Wieghardt and C.-Y. Wong, *Inorg. Chem.*, 2013, **52**, 9885-9896.