Electronic supplementary materials *Mendeleev Commun.*, 2019, **29**, 38–40

Monothiolate ruthenium alkylidene complexes with tricyclic fluorinated N-heterocyclic carbene ligands

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Table of Contents

Experimental Section

Materials and metods. All solvents were freshly distilled over appropriate drying agents before use. All other reagents were recrystallized or distilled as necessary. Syntheses of ruthenium complexes were performed under an argon atmosphere using a standard Schlenk technique. Analytical TLC was performed using Merck silica gel 60 F254 plates. Visualization was accomplished by UV light (254 and 366 nm), spraying by $Ce(SO_4)_2$ solution in 5% H_2SO_4 or $KMnO_4$ solution in water. Column chromatography was carried out using Merck silica gel 60 (230−400 mesh ASTM) and ethyl acetate/petroleum ether mixture as eluent. NMR spectra were recorded at room temperature unless otherwise stated on Bruker AV-400 and AV-500 spectrometers operating at 400 MHz for ^{1}H; 101 and 126 MHz for ^{13}C; and 376 MHz for ^{19}F ($CFCl_3$ as reference). The chemical shifts are frequency referenced relative to the solvent residual peaks. Purification of allylbenzene[S1] and synthesis of dichloro(2-isopropoxybenzylidene)[2-mesityl-7,9-dimethyl-5,5-bis(trifluoromethyl)-3a,5-dihydro-3*H*-benzo[*d*]imidazo[5,1-*b*][1,3]oxazin-1-ylidene]ruthenium(II) (**2a**), dichloro(2-isopropoxybenzylidene)[2-mesityl-7-methyl-5,5-bis(trifluoromethyl)-3a,5-dihydro-3*H*-benzo[*d*]imidazo[5,1-*b*][1,3]oxazin-1-

[S1] J. Engel, W. Smit, M. Foscato, G. Occhipinti, K. W. Törnroos and V. R. Jensen, *J. Am. Chem. Soc.*, 2017, **139**, 16609.

ylidene]ruthenium(II) (**2b**)[S2], potassium 2,4,6-triphenylthiophenolate,[S3] and *N*,*N*-diallyl-4-methylbenzenesulfonamide[S4] were carried out according to literature procedures. Crystals suitable for X-ray diffraction analisys were grown by slow diffusion of hexane in CH_2Cl_2 solution.

General procedure for the synthesis of arylthio-complexes 3a and 3b. In a flame-dried Schlenk flask, corresponding dichloro complex **2** (0.12 mmol) was dissolved in anhydrous THF (12 ml) under argon atmosphere. Then solid potassium 2,4,6-triphenylthiophenolate (54 mg, 0.14 mmol) was added, the resulting mixture was degassed three times and stirred for 3 h at r.t. Once it was complete (TLC-control), the solvents were removed from the reaction mixture under reduced pressure, and the resulting substance was purified by column chromatography using petroleum ether/ethyl acetate (6 : 1) as eluent under an argon atmosphere. The resulting solid was recrystallized from MeOH to yield ruthenium arylthio-complex as a brown solid.

Chloro(2-isopropoxybenzylidene)[2-mesityl-7,9-dimethyl-5,5-bis(trifluoromethyl)-3a,5-dihydro-3*H*-benzo[*d*]imidazo[5,1-*b*][1,3]oxazin-1-ylidene](2,4,6-triphenylphenylthio)-ruthenium(II) (3a). Yield: 32%. Mixture of diastereomers 85 : 15, the signals of major product are given. ^{1}H NMR (400 MHz, C_6D_6), δ: 14.96 (s, 1H, C*H*Ar), 8.01 (s, 2H, H_{Ar}), 7.49–7.44 (m, 4H, H_{Ar}), 7.37–7.28 (m, 5H, H_{Ar}), 7.24–7.09 (m, 3H, H_{Ar}), 7.03–6.94 (m, 3H, H_{Ar}), 6.91–6.84 (m, 2H, H_{Ar}), 6.76 (s, 1H, H_{Ar}), 6.59–6.51 (m, 4H, H_{Ar}), 6.23 (d, $J_{H,H}$ 8.2 Hz, 1H, H_{Ar}), 4.79 (d, $^3J_{H,H}$ 5.7 Hz, 1H, NC*H*O), 4.26 (hept, $^3J_{H,H}$ 5.8 Hz, 1H, C*H*Me$_2$), 3.42 (dd, $J_{H,H}$ 13.0, 6.1 Hz, 1H, CH_2), 2.77 (d, $J_{H,H}$ 12.8 Hz, 1H, CH_2), 2.76 (s, 3H, CH_3), 2.70 (s, 3H, CH_3), 2.28 (s, 3H, CH_3), 2.24 (s, 3H, CH_3), 1.88 (s, 3H, CH_3), 1.46 [d, $^3J_{H,H}$ 6.0 Hz, 3H, C(CH_3)$_2$], 0.58 ppm [d, $^3J_{H,H}$ 6.0 Hz, 3H, C(CH_3)$_2$]. ^{13}C NMR (126 MHz, C_6D_6), δ: 273.0 (Ru=*C*H), 215.8(N*C*N), 154.2 (C_{Ar}), 149.0 (C_{Ar}), 147.5 (C_{Ar}), 145.4 (C_{Ar}), 145.1 (C_{Ar}), 141.7 (C_{Ar}), 141.1 (C_{Ar}), 139.0 (C_{Ar}), 138.6 (C_{Ar}), 138.1 (C_{Ar}), 138.0 (C_{Ar}), 137.8 (C_{Ar}), 136.4 (C_{Ar}), 133.8 (C_{Ar}), 131.9 (C_{Ar}), 131.2 (C_{Ar}), 130.9 (C_{Ar}), 130.6 (C_{Ar}), 130.4 (C_{Ar}), 129.0 (C_{Ar}), 128.5 (C_{Ar}), 127.4 (C_{Ar}), 127.1 (C_{Ar}), 126.9 (C_{Ar}), 125.0 (C_{Ar}), 125.0 (C_{Ar}), 123.7 (q, $^1J_{C,F}$ 288 Hz, *C*F$_3$), 123.1 (C_{Ar}), 122.6 (q, $^1J_{C,F}$ 291 Hz, *C*F$_3$), 122.2 (C_{Ar}), 121.5 (C_{Ar}), 121.4 (C_{Ar}), 118.6 (C_{Ar}), 114.6 (C_{Ar}), 113.6 (C_{Ar}), 113.0 (C_{Ar}), 84.5 (N*C*O), 79.2–78.1 [m, *C*(CF$_3$)$_2$], 75.6 (O*C*Me$_2$), 59.2 (*C*H$_2$), 23.7 (*C*H$_3$), 23.1 (*C*H$_3$), 21.1 (*C*H$_3$), 20.9 (*C*H$_3$), 20.5 (*C*H$_3$), 20.1 [C(*C*H$_3$)$_2$], 18.3 [C(*C*H$_3$)$_2$]. ^{19}F NMR (376 MHz, C_6D_6), δ: –72.03 (s, 3F, CF$_3$), –72.60 (s, 3F, CF$_3$). Elemental analysis, calcd. for $C_{57}H_{51}ClF_6N_2O_2RuS$ (%): C, 63.47; H, 4.77; N, 2.60; found C, 63.63; H, 4.91; N, 2.48.

Chloro(2-isopropoxybenzylidene)[2-mesityl-7-methyl-5,5-bis(trifluoromethyl)-3a,5-dihydro-3*H*-benzo[*d*]imidazo[5,1-*b*][1,3]oxazin-1-ylidene](2,4,6-triphenylphenylthio)-ruthenium(II) (3b). Yield: 64%. Mixture of diastereomers 88 : 12, the signals of major product are given. ^{1}H NMR (400 MHz, C_6D_6), δ: 13.65 (s, 1H, C*H*Ar), 8.96 (d, $^3J_{H,H}$ 7.2 Hz, 1H, H_{Ar}), 7.65–7.60 (m, 2H, H_{Ar}), 7.54–7.44 (s, 1H, H_{Ar}), 7.38–7.21 (m, 4H, H_{Ar}), 7.15–6.99 (m, 7H, H_{Ar}), 6.98–6.86 (m, 3H, H_{Ar}), 6.85–6.61 (m, 6H, H_{Ar}), 6.51 (d, $J_{H,H}$ 7.4 Hz, 1H, H_{Ar}), 6.36 (d, $J_{H,H}$ 8.3 Hz, 1H, H_{Ar}), 4.63 (s, 1H, NC*H*O), 4.40 (s, 1H, C*H*Me$_2$), 3.29 (s, 2H, CH_2), 2.40 (s, 3H, CH_3),

[S2] S. M. Masoud, T. R. Akmalov, K. A. Palagin, F. M. Dolgushin, S. E. Nefedov and S. N. Osipov, *Eur. J. Org. Chem.*, 2018, doi: 10.1002/ejoc.201801116.

[S3] G. Occhipinti, F. R. Hansen, K. W. Törnroos and V. R. Jensen, *J. Am. Chem. Soc.*, 2013, **135**, 3331.

[S4] C. R. Larsen and D. B. Grotjahn, *J. Am. Chem. Soc.*, 2012, **134**, 10357.

2.28 (s, 3H, CH_3), 2.12 (s, 3H, CH_3), 2.09 (s, 3H, CH_3), 1.67 [d, $^3J_{H,H}$ 6.1 Hz, 3H, C(CH_3)$_2$], 0.62 [d, $^3J_{H,H}$ 6.1 Hz, 3H, C(CH_3)$_2$]. ^{13}C NMR (101 MHz, C_6D_6), δ: 274.1 (Ru=*C*H), 215.1 (N*C*N), 153.6 (C_{Ar}), 148.9 (C_{Ar}), 147.5 (C_{Ar}), 145.4 (C_{Ar}), 143.8 (C_{Ar}), 142.2 (C_{Ar}), 141.3 (C_{Ar}), 141.1 (C_{Ar}), 138.6 (C_{Ar}), 138.1 (C_{Ar}), 137.9 (C_{Ar}), 137.6 (C_{Ar}), 137.4 (C_{Ar}), 137.1 (C_{Ar}), 135.1 (C_{Ar}), 131.3 (C_{Ar}), 131.1 (C_{Ar}), 130.8 (C_{Ar}), 129.7 (C_{Ar}), 129.2 (C_{Ar}), 128.9 (C_{Ar}), 128.8 (C_{Ar}), 128.6 (C_{Ar}), 127.4 (C_{Ar}), 127.2 (C_{Ar}), 127.2 (C_{Ar}), 127.0 (C_{Ar}), 126.77 (C_{Ar}), 125.8 (C_{Ar}), 125.3 (C_{Ar}), 123.5 (q, $^1J_{C,F}$ 291 Hz, *C*F$_3$), 122.8 (C_{Ar}), 122.7 (q, $^1J_{C,F}$ 289 Hz, *C*F$_3$), 121.6 (C_{Ar}), 115.7 (C_{Ar}), 113.0 (C_{Ar}), 83.8 (N*C*O), 79.7–78.5 [m, *C*(CF$_3$)$_2$], 75.0 (O*C*Me$_2$), 57.7 (*C*H$_2$), 23.0 (*C*H$_3$), 22.1 (*C*H$_3$), 21.2 (*C*H$_3$), 20.3 (*C*H$_3$), 18.4 [C(*C*H$_3$)$_2$], 18.0 [C(*C*H$_3$)$_2$]. ^{19}F NMR (376 MHz, C_6D_6), δ: –71.04 (s, 3F, CF$_3$), –74.43 ppm (s, 3F, CF$_3$). Elemental analysis, calcd. for $C_{56}H_{49}ClF_6N_2O_2RuS$ (%): C, 63.18; H, 4.64; N, 2.63; found C, 63.27; H, 4.79; N, 2.57. CCDC 1866377 contains the supplementary crystallographic data for this complex. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

Ring-closing metathesis of *N,N*-diallyltosylamide.

In a flame-dried Schlenk flask charged with *N,N*-diallyl-4-methylbenzenesulfonamide (201 mg, 0.8 mmol), a catalyst solution in toluene (1 mM, 8.0 ml, 8 μmol) was added *via* syringe under argon atmosphere. The reaction mixture was stirred at 30°C unless otherwise stated. Data points were collected over an appropriate period of time by taking the samples (~0.5 ml) from the reaction mixture. The reaction was quenched using an excess of ethyl vinyl ether (0.5 ml). The conversion into CM product was determined by ^{1}H NMR comparing the ratio of integrals of the methylene protons in starting material (δ = 3.80 ppm, d, $^3J_{H,H}$ 6.3 Hz, 4H) with those in the product (δ = 4.11 ppm, s, 4H).

Self-metathesis of allylbenzene.

In a flame-dried Schlenk flask charged with allylbenzene (355 mg, 3.0 mmol), a catalyst solution in toluene (50 mM, 0.6 ml, 30 μmol) was added *via* syringe under argon atmosphere. The reaction mixture was stirred at 30°C unless otherwise stated. Data points were collected over an appropriate period of time by taking the samples (~0.1 ml) from the reaction mixture. The reaction was quenched using an excess of ethyl vinyl ether (0.5 ml). The conversion into CM product was determined by ^{1}H NMR.

Allylbenzene. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 7.35–7.29 (m, 2H, H_{Ph}), 7.25–7.20 (m, 3H, H_{Ph}), 6.00 (ddt, $J_{H,H}$ 16.8, 10.1, 6.8 Hz, 1H, BnC*H*), 5.14–5.07 (m, 2H, C=CH_2), 3.42 (d, $^3J_{H,H}$ 6.6 Hz, 2H, PhCH_2).

Other compounds. ^{1}H NMR spectral data of (*E*)-1,4-diphenylbut-2-ene [3.41 ppm (bd, $J_{H,H}$ 4.5 Hz, 4H)],[S5] (*Z*)-1,4-diphenylbut-2-ene [3.54 ppm (d, $J_{H,H}$ 5.5 Hz, 4H)],[S6] (*E*)-prop-1-enylbenzene [1.85 ppm (dd, 3H)], (*Z*)-prop-1-enylbenzene [1.83 ppm (dd, $J_{H,H}$ 7.2, 1.8 Hz, 3H)],[S7] (*E*)-but-1-ene-1,4-diyldibenzene [2.94 (t, $J_{H,H}$ 8.0 Hz, 2H), 2.73–2.62 (m, 2H)],[S8] and (*Z*)-but-1-ene-1,4-diyldibenzene [2.82–2.75 (m, 2H), 2.69–2.65 ppm (m, 2H)][S9] were found in literature.

[S5] P. R. Blakemore and M. S. Burge, *J. Am. Chem. Soc.*, 2007, **129**, 3068.
[S6] E. Mai and C. Schneider, *Chem. Eur. J.*, 2007, **13**, 2729.
[S7] C. Belger, N. M. Neisius and B. Plietker, *Chem. Eur. J.*, 2010, **16**, 12214.
[S8] D. Habrant, B. Stengel, S. Meunier and C. Mioskowski, *Chem. Eur. J.*, 2007, **13**, 5433.
[S9] S. Hajra, B. Maji and D. Mal, *Adv. Synth. Catal.*, 2009, **351**, 859.

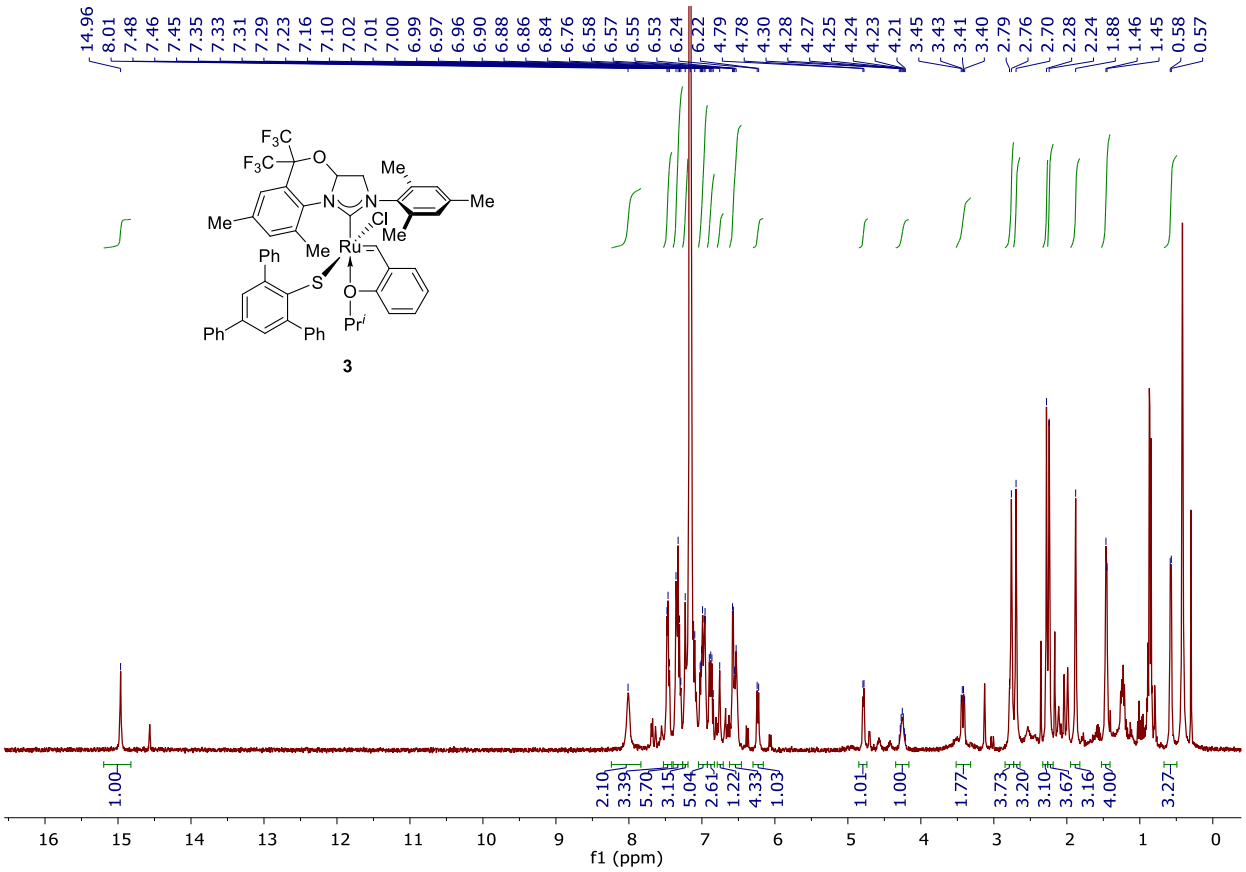

Figure S1 ^{1}H NMR spectrum of complex **3a** in C_6D_6.

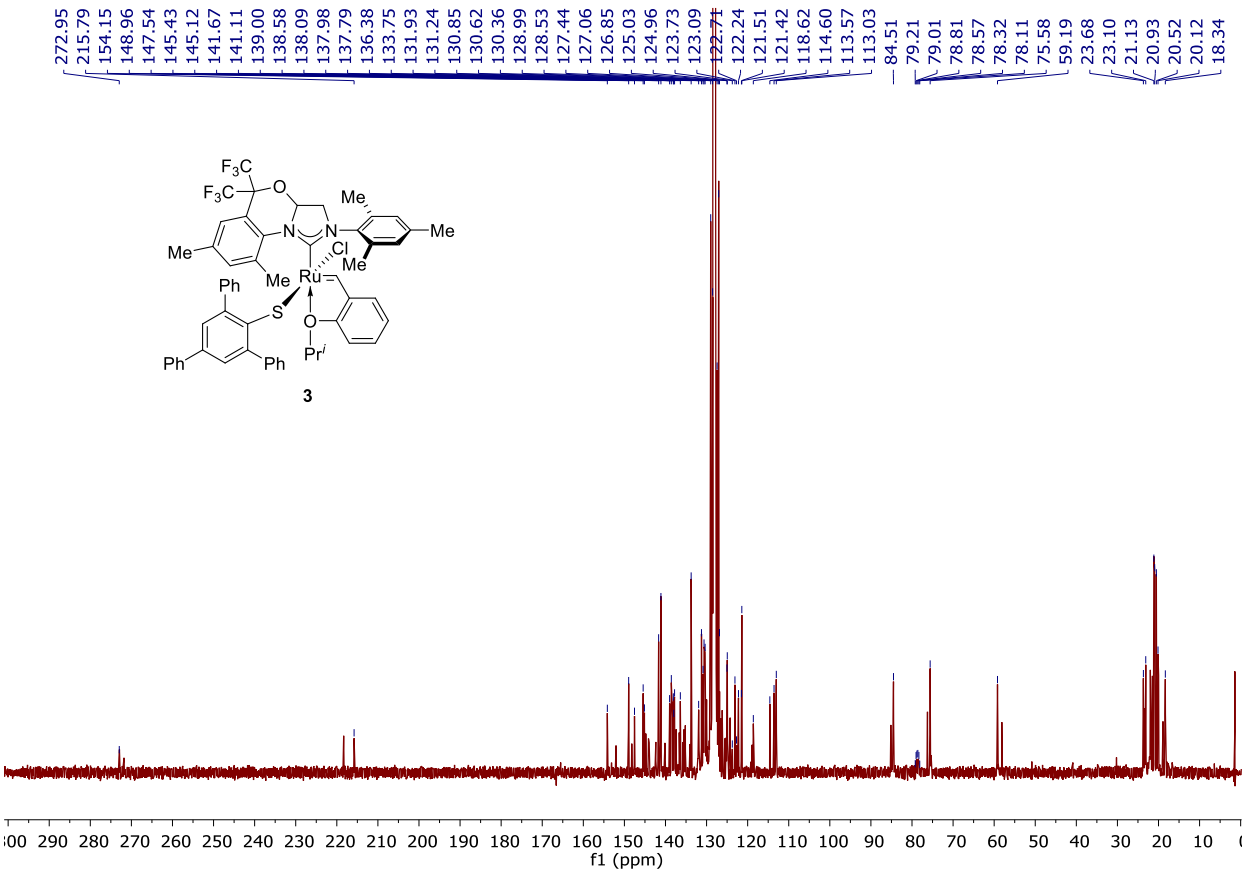

Figure S2 ^{13}C NMR spectrum of complex **3a** in C_6D_6.

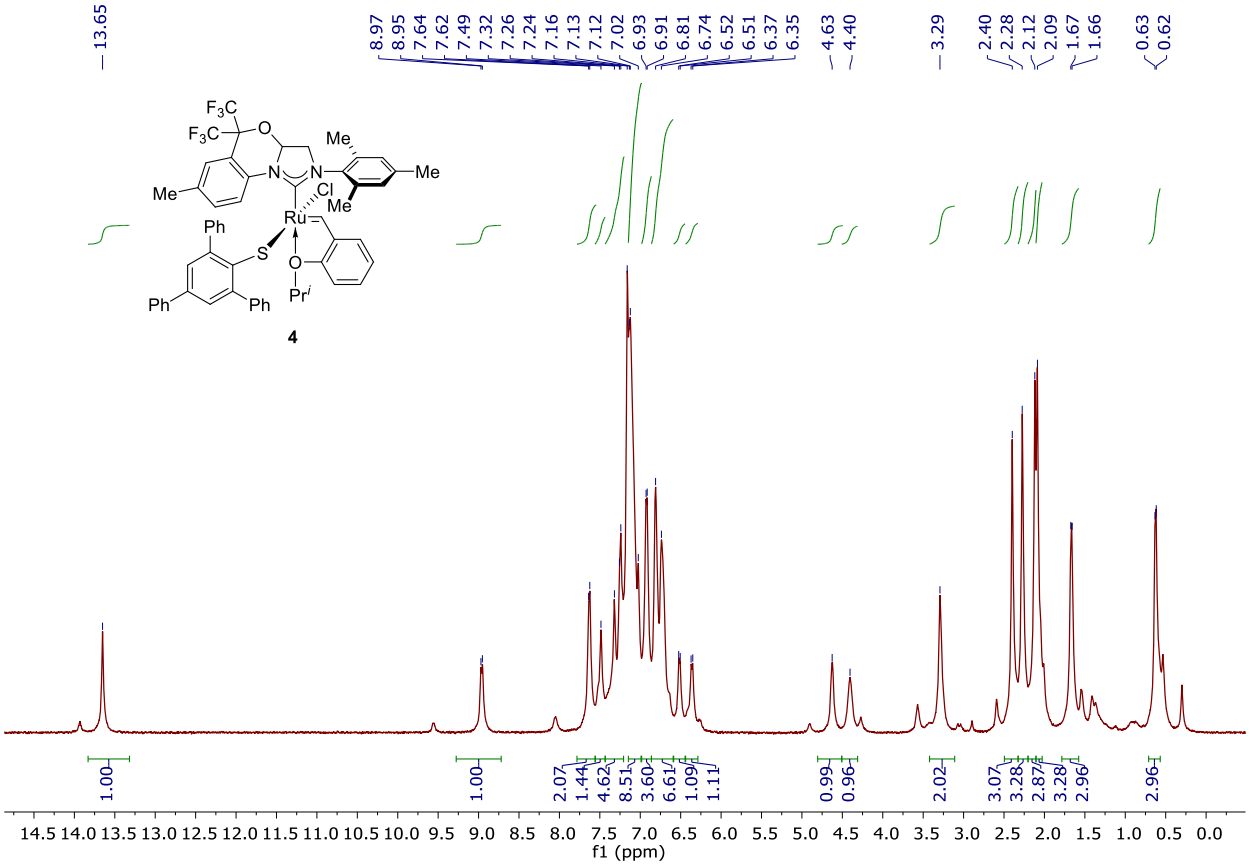

Figure S3 ^{1}H NMR spectrum of complex **3b** in C_6D_6.

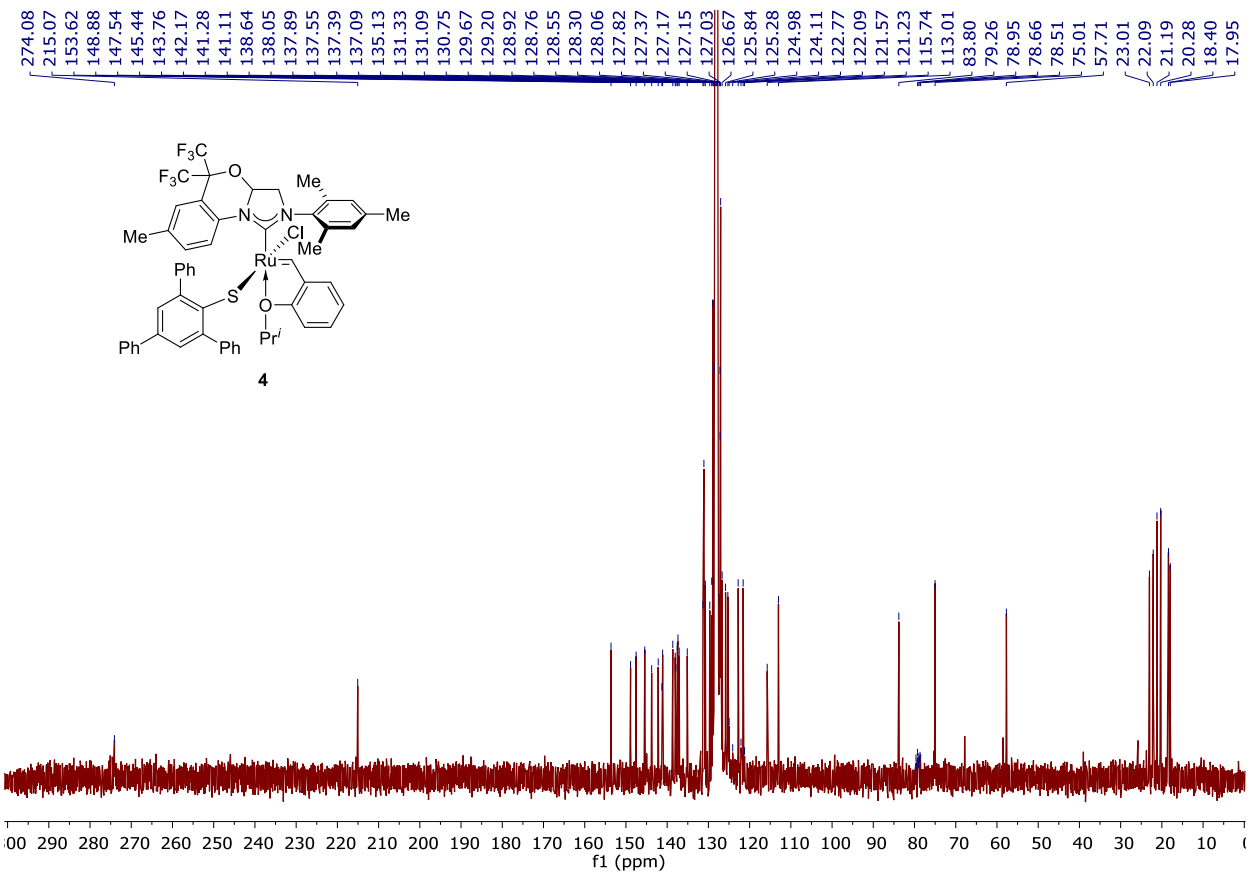

Figure S4 ^{13}C NMR spectrum of complex **3b** in C_6D_6.

X-ray supporting information

X-ray structure analysis of **3b** was made on a Bruker AXS SMART 1000, CCD-detector λ(MoK*a*) = 0.71073 Å, graphite monochromator, ω-scanning, $2\theta_{max}$= 60°, $C_{56}H_{50}Cl_2F_6N_2O_2RuS$, M = 1107.01, triclinic, space group P-1, a = 11.6280(13) Å, b = 21.281(2) Å, and c = 22.280(3) Å; α = 113.133(2)°, β = 92.415(3)°, and γ = 102.219(3)°; V = 4906.7(10) Å^3 (120 K), Z = 4, d_{calc} = 1.499 g cm^{-3}, 28598 measured reflections, 19669 independent reflections with $F^2 > 2\sigma(I)$, μ = 0.540 mm^{-1}, R_1 = 0.0506, wR_2 = 0.0806. Corrections for absorption were made by SADABS[S10]. The structure was solved by direct method and refined by full-matrix least squares method for F^2 with anisotropic parameters for all non-hydrogen atoms. All calculations were performed with the use of the SAINT[S11] and SHELXTL-97[S12] program packages.

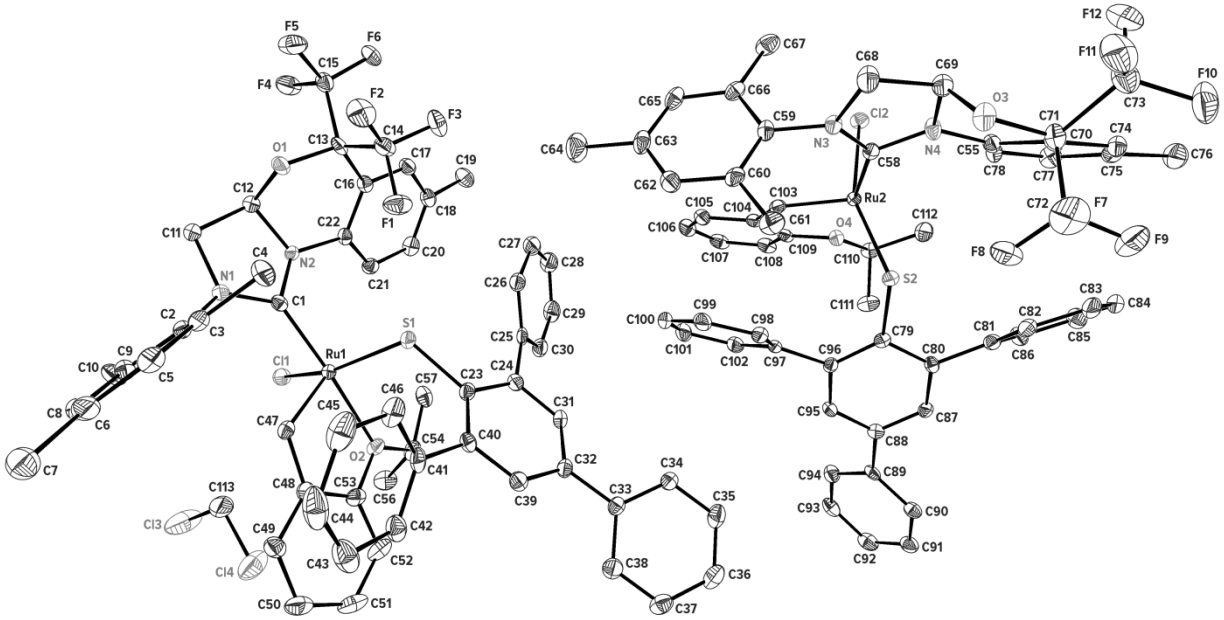

Figure S5 Structure of complex **3b**. Thermal ellipsoids are drawn at 50% probability level.

Table S1 Bond lengths (Å) and angles (°) for **3b**.

Ru1 C47 1.837(3)	Ru1 C1 1.970(3)
Ru1 O2 2.246(2)	Ru1 S1 2.2922(8)
Ru1 Cl1 2.4036(8)	Ru2 C103 1.842(3)
Ru2 C58 1.974(3)	Ru2 O4 2.2567(19)
Ru2 S2 2.3009(8)	Ru2 Cl2 2.3915(7)
Cl3 C113 1.765(4)	Cl4 C113 1.749(4)
S1 C23 1.793(3)	S2 C79 1.793(3)
F1 C14 1.328(3)	F2 C14 1.337(4)
F3 C14 1.332(3)	F4 C15 1.352(3)
F5 C15 1.330(3)	F6 C15 1.326(3)
F7 C72 1.335(4)	F8 C72 1.333(4)
F9 C72 1.337(4)	F10 C73 1.346(4)
F11 C73 1.333(4)	F12 C73 1.326(4)
N1 C1 1.358(4)	N1 C2 1.442(3)
N1 C11 1.467(3)	N2 C1 1.390(3)
N2 C22 1.423(3)	N2 C12 1.448(4)
N3 C58 1.348(3)	N3 C59 1.441(4)
N3 C68 1.467(4)	N4 C58 1.391(4)

[S10] G. M. Sheldrick, *SADABS*, Bruker AXS Inc., Madison, WI, 1997.
[S11] *SMART, v. 5.051* and *SAINT, v.5.00, Area Detector Control and Integration Software*, Bruker AXS Inc., Madison, WI, 1998.
[S12] G. M. Sheldrick, *SHELXTL, v. 5.10*, Bruker AXS Inc., Madison, WI, 1997.

N4 C55 1.411(4)	N4 C69 1.444(4)
O1 C13 1.428(3)	O1 C12 1.441(3)
O2 C53 1.388(3)	O2 C54 1.478(3)
O3 C69 1.426(4)	O3 C71 1.429(3)
O4 C109 1.386(3)	O4 C110 1.468(3)
C2 C3 1.392(4)	C2 C9 1.405(4)
C3 C5 1.390(4)	C3 C4 1.512(4)
C5 C6 1.401(4)	C6 C8 1.395(4)
C6 C7 1.509(4)	C8 C9 1.388(4)
C9 C10 1.509(4)	C11 C12 1.501(4)
C13 C16 1.523(4)	C13 C15 1.540(4)
C13 C14 1.548(4)	C16 C17 1.394(4)
C16 C22 1.407(4)	C17 C18 1.392(4)
C18 C20 1.394(4)	C18 C19 1.508(4)
C20 C21 1.389(4)	C21 C22 1.381(4)
C23 C40 1.423(4)	C23 C24 1.423(4)
C24 C31 1.397(4)	C24 C25 1.489(4)
C25 C26 1.398(4)	C25 C30 1.406(4)
C26 C27 1.396(4)	C27 C28 1.383(5)
C28 C29 1.383(5)	C29 C30 1.385(4)
C31 C32 1.384(4)	C32 C39 1.393(4)
C32 C33 1.488(4)	C33 C38 1.392(4)
C33 C34 1.402(4)	C34 C35 1.393(4)
C35 C36 1.380(5)	C36 C37 1.392(5)
C37 C38 1.388(4)	C39 C40 1.401(4)
C40 C41 1.493(4)	C41 C42 1.398(4)
C41 C46 1.399(4)	C42 C43 1.402(4)
C43 C44 1.380(5)	C44 C45 1.389(5)
C45 C46 1.395(4)	C47 C48 1.449(4)
C48 C53 1.392(4)	C48 C49 1.402(4)
C49 C50 1.376(4)	C50 C51 1.384(5)
C51 C52 1.396(5)	C52 C53 1.382(4)
C54 C57 1.512(4)	C54 C56 1.524(4)
C55 C78 1.396(4)	C55 C70 1.402(4)
C59 C66 1.397(4)	C59 C60 1.399(4)
C60 C62 1.398(4)	C60 C61 1.499(4)
C62 C63 1.391(5)	C63 C65 1.382(5)
C63 C64 1.515(4)	C65 C66 1.387(4)
C66 C67 1.508(4)	C68 C69 1.512(4)
C70 C74 1.394(4)	C70 C71 1.525(4)
C71 C72 1.546(4)	C71 C73 1.560(4)
C74 C75 1.383(4)	C75 C77 1.395(4)
C75 C76 1.513(4)	C77 C78 1.391(4)
C79 C96 1.415(4)	C79 C80 1.426(4)
C80 C87 1.401(4)	C80 C81 1.489(4)
C81 C82 1.397(4)	C81 C86 1.401(4)
C82 C83 1.392(4)	C83 C84 1.391(5)
C84 C85 1.381(4)	C85 C86 1.386(4)
C87 C88 1.392(4)	C88 C95 1.397(4)
C88 C89 1.484(4)	C89 C90 1.400(4)
C89 C94 1.409(4)	C90 C91 1.385(4)
C91 C92 1.391(5)	C92 C93 1.390(5)
C93 C94 1.381(4)	C95 C96 1.402(4)
C96 C97 1.498(4)	C97 C98 1.393(4)
C97 C102 1.395(4)	C98 C99 1.392(4)
C99 C100 1.388(5)	C100 C101 1.385(5)
C101 C102 1.390(4)	C103 C104 1.446(4)
C104 C109 1.400(4)	C104 C105 1.403(4)
C105 C106 1.388(4)	C106 C107 1.384(5)
C107 C108 1.392(4)	C108 C109 1.389(4)

C110 C112 1.508(4)	C110 C111 1.510(4)

C47 Ru1 C1 99.91(12) .	C47 Ru1 O2 79.85(10) .
C1 Ru1 O2 174.95(9) . .	C47 Ru1 S1 104.32(9) .
C1 Ru1 S1 91.79(8) . .	O2 Ru1 S1 93.15(6) . .
C47 Ru1 Cl1 102.89(9) .	C1 Ru1 Cl1 88.31(8) . .
O2 Ru1 Cl1 86.83(5) . .	S1 Ru1 Cl1 152.35(3) .
C103 Ru2 C58 100.64(12)	C103 Ru2 O4 79.02(10) .
C58 Ru2 O4 174.93(9) .	C103 Ru2 S2 105.84(9) .
C58 Ru2 S2 88.15(8) . .	O4 Ru2 S2 96.82(5) . .
C103 Ru2 Cl2 98.47(9) .	C58 Ru2 Cl2 90.71(8) .
O4 Ru2 Cl2 84.35(5) . .	S2 Ru2 Cl2 155.45(3) .
C23 S1 Ru1 114.34(10) .	C79 S2 Ru2 117.13(9) .
C1 N1 C2 129.4(2) . . ?	C1 N1 C11 113.5(2) . .
C2 N1 C11 116.5(2) . .	C1 N2 C22 129.4(2) . .
C1 N2 C12 112.6(2) . .	C22 N2 C12 112.3(2) . .
C58 N3 C59 127.5(2) . .	C58 N3 C68 114.7(2) . .
C59 N3 C68 117.8(2) . .	C58 N4 C55 131.6(2) . .
C58 N4 C69 114.8(2) . .	C55 N4 C69 113.0(2) . .
C13 O1 C12 113.9(2) . .	C53 O2 C54 119.0(2) . .
C53 O2 Ru1 109.65(17) .	C54 O2 Ru1 130.94(17) .
C69 O3 C71 113.6(2) . .	C109 O4 C110 118.4(2) .
C109 O4 Ru2 109.43(16)	C110 O4 Ru2 130.39(16)
N1 C1 N2 104.8(2) . . ?	N1 C1 Ru1 132.8(2) . .
N2 C1 Ru1 121.7(2) . .	C3 C2 C9 122.4(3) . . ?
C3 C2 N1 119.7(3) . . ?	C9 C2 N1 117.6(2) . . ?
C5 C3 C2 118.0(3) . . ?	C5 C3 C4 121.2(3) . . ?
C2 C3 C4 120.9(3) . . ?	C3 C5 C6 121.6(3) . . ?
C8 C6 C5 118.4(3) . . ?	C8 C6 C7 119.8(3) . . ?
C5 C6 C7 121.8(3) . . ?	C9 C8 C6 122.0(3) . . ?
C8 C9 C2 117.6(3) . . ?	C8 C9 C10 121.2(3) . .
C2 C9 C10 121.2(3) . .	N1 C11 C12 102.1(2) . .
O1 C12 N2 109.3(2) . .	O1 C12 C11 107.7(2) . .
N2 C12 C11 102.5(2) . .	O1 C13 C16 114.0(2) . .
O1 C13 C15 104.8(2) . .	C16 C13 C15 113.7(2) .
O1 C13 C14 103.9(2) . .	C16 C13 C14 109.3(2) .
C15 C13 C14 110.6(2) .	F1 C14 F3 108.1(3) . .
F1 C14 F2 107.2(2) . .	F3 C14 F2 107.4(2) . .
F1 C14 C13 110.1(2) . .	F3 C14 C13 111.4(2) . .
F2 C14 C13 112.4(3) . .	F6 C15 F5 107.1(2) . .
F6 C15 F4 106.7(2) . .	F5 C15 F4 106.8(2) . .
F6 C15 C13 115.2(2) . .	F5 C15 C13 111.3(2) . .
F4 C15 C13 109.2(2) . .	C17 C16 C22 119.1(3) .
C17 C16 C13 120.7(3) .	C22 C16 C13 120.1(2) .
C18 C17 C16 121.4(3) .	C17 C18 C20 118.0(3) .
C17 C18 C19 119.8(3) .	C20 C18 C19 122.2(3) .
C21 C20 C18 121.5(3) .	C22 C21 C20 119.9(3) .
C21 C22 C16 119.9(3) .	C21 C22 N2 122.8(3) . .
C16 C22 N2 117.1(2) . .	C40 C23 C24 117.8(3) .
C40 C23 S1 122.2(2) . .	C24 C23 S1 119.8(2) . .
C31 C24 C23 119.7(3) .	C31 C24 C25 114.7(3) .
C23 C24 C25 125.6(3) .	C26 C25 C30 118.0(3) .
C26 C25 C24 122.8(3) .	C30 C25 C24 118.5(3) .
C27 C26 C25 120.3(3) .	C28 C27 C26 120.6(3) .
C27 C28 C29 119.8(3) .	C28 C29 C30 120.0(3) .
C29 C30 C25 121.2(3) .	C32 C31 C24 123.0(3) .
C31 C32 C39 117.2(3) .	C31 C32 C33 121.4(3) .
C39 C32 C33 121.3(3) .	C38 C33 C34 118.2(3) .
C38 C33 C32 121.0(3) .	C34 C33 C32 120.8(3) .

C35 C34 C33 120.5(3) .	C36 C35 C34 120.6(3) .
C35 C36 C37 119.4(3) .	C38 C37 C36 120.2(3) .
C37 C38 C33 121.0(3) .	C32 C39 C40 122.6(3) .
C39 C40 C23 119.7(3) .	C39 C40 C41 114.4(3) .
C23 C40 C41 125.7(3) .	C42 C41 C46 118.3(3) .
C42 C41 C40 119.4(3) .	C46 C41 C40 121.8(3) .
C41 C42 C43 120.9(3) .	C44 C43 C42 119.8(3) .
C43 C44 C45 120.1(3) .	C44 C45 C46 120.1(4) .
C45 C46 C41 120.7(3) .	C48 C47 Ru1 118.3(2) .
C53 C48 C49 118.2(3) .	C53 C48 C47 118.5(3) .
C49 C48 C47 123.2(3) .	C50 C49 C48 120.8(3) .
C49 C50 C51 119.6(3) .	C50 C51 C52 121.2(3) .
C53 C52 C51 118.1(3) .	C52 C53 O2 124.6(3) . .
C52 C53 C48 122.0(3) .	O2 C53 C48 113.4(3) . .
O2 C54 C57 106.2(2) . .	O2 C54 C56 110.7(3) . .
C57 C54 C56 110.4(3) .	C78 C55 C70 119.7(3) .
C78 C55 N4 122.4(3) . .	C70 C55 N4 117.4(3) . .
N3 C58 N4 104.5(2) . .	N3 C58 Ru2 132.7(2) . .
N4 C58 Ru2 122.6(2) . .	C66 C59 C60 122.7(3) .
C66 C59 N3 118.9(3) . .	C60 C59 N3 118.4(3) . .
C62 C60 C59 117.2(3) .	C62 C60 C61 121.7(3) .
C59 C60 C61 121.1(3) .	C63 C62 C60 121.4(3) .
C65 C63 C62 119.3(3) .	C65 C63 C64 120.4(3) .
C62 C63 C64 120.2(3) .	C63 C65 C66 121.7(3) .
C65 C66 C59 117.6(3) .	C65 C66 C67 121.3(3) .
C59 C66 C67 121.1(3) .	N3 C68 C69 103.2(2) . .
O3 C69 N4 110.6(2) . .	O3 C69 C68 109.0(2) . .
N4 C69 C68 102.7(2) . .	C74 C70 C55 119.4(3) .
C74 C70 C71 121.1(3) .	C55 C70 C71 119.4(3) .
O3 C71 C70 114.8(2) . .	O3 C71 C72 103.1(2) . .
C70 C71 C72 110.0(2) .	O3 C71 C73 106.1(2) . .
C70 C71 C73 112.3(2) .	C72 C71 C73 110.0(3) .
F8 C72 F7 107.5(3) . .	F8 C72 F9 107.8(3) . .
F7 C72 F9 107.2(3) . .	F8 C72 C71 109.9(3) . .
F7 C72 C71 112.0(3) . .	F9 C72 C71 112.2(3) . .
F12 C73 F11 108.2(3) .	F12 C73 F10 107.3(3) .
F11 C73 F10 106.7(3) .	F12 C73 C71 110.0(3) .
F11 C73 C71 111.8(3) .	F10 C73 C71 112.6(3) .
C75 C74 C70 121.4(3) .	C74 C75 C77 118.6(3) .
C74 C75 C76 120.4(3) .	C77 C75 C76 120.9(3) .
C78 C77 C75 121.1(3) .	C77 C78 C55 119.7(3) .
C96 C79 C80 117.8(2) .	C96 C79 S2 123.4(2) . .
C80 C79 S2 118.7(2) . .	C87 C80 C79 119.5(3) .
C87 C80 C81 115.7(2) .	C79 C80 C81 124.7(3) .
C82 C81 C86 118.0(3) .	C82 C81 C80 123.2(3) .
C86 C81 C80 118.7(3) .	C83 C82 C81 120.5(3) .
C84 C83 C82 120.4(3) .	C85 C84 C83 119.6(3) .
C84 C85 C86 120.0(3) .	C85 C86 C81 121.4(3) .
C88 C87 C80 123.3(3) .	C87 C88 C95 116.5(3) .
C87 C88 C89 121.9(3) .	C95 C88 C89 121.7(3) .
C90 C89 C94 117.6(3) .	C90 C89 C88 121.4(3) .
C94 C89 C88 121.0(3) .	C91 C90 C89 121.3(3) .
C90 C91 C92 120.2(3) .	C93 C92 C91 119.4(3) .
C94 C93 C92 120.5(3) .	C93 C94 C89 121.0(3) .
C88 C95 C96 122.7(3) .	C95 C96 C79 120.2(3) .
C95 C96 C97 114.6(2) .	C79 C96 C97 125.2(3) .
C98 C97 C102 117.8(3) .	C98 C97 C96 122.1(3) .
C102 C97 C96 119.8(3) .	C99 C98 C97 120.7(3) .
C100 C99 C98 120.7(3) .	C101 C100 C99 119.3(3)
C100 C101 C102 119.8(3)	C101 C102 C97 121.7(3)

C104 C103 Ru2 117.9(2)	C109 C104 C105 118.7(3)
C109 C104 C103 118.5(3)	C105 C104 C103 122.8(3)
C106 C105 C104 120.3(3)	C107 C106 C105 119.8(3)
C106 C107 C108 121.3(3)	C109 C108 C107 118.6(3)
O4 C109 C108 125.9(3) .	O4 C109 C104 112.8(2) .
C108 C109 C104 121.3(3)	O4 C110 C112 107.7(2) .
O4 C110 C111 109.6(2) .	C112 C110 C111 112.1(3)
Cl4 C113 Cl3 110.3(2) .	