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Reactivity of dinuclear arene ruthenium complexes: reactions of the hydrido complex $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ with NaX and HX (X = F, Cl, Br, I)

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Abstract

The dinuclear hydrido complex $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ (**1**) reacts with the sodium halides NaX in methanol to give the halogen analogues $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2X_2(\mu\text{-X})(\mu\text{-H})]$ (**2**: X = F, **3**: X = Br, **4**: X = I). With HX, complex **1** reacts to give the tetrahalo complexes $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2X_2(\mu\text{-X})_2]$ (**5**: X = Cl, **6**: X = Br, **7**: X = I); in the case of X = I, a large excess of HI leads to the formation of the cationic complex $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2(\mu\text{-I})_3]^+$ (**8**). The X-ray structure analysis of **1** shows a dinuclear Ru_2 backbone with two terminal chloro ligands being *trans* with respect to each other as the two *p*-cymene ligands, the two bridging ligands lie in a plane perpendicular to the plane defined by the terminal chloro ligands and the ruthenium atoms.
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Keywords: Rutherium complexes; Hydrido complexes; Arene complexes

1. Introduction

Arene ruthenium complexes have received much attention over the past 25 years because of their unique structures and properties and also due to their inherent catalytic potential [1]. Some of these complexes are reactive towards molecular hydrogen or hydrogen transferring agents, consequently a considerable number of arene ruthenium complexes containing hydride ligands have been synthesised [2–6].

The dinuclear *p*-cymene hydrido complex $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ (**1**) was first obtained by Bennett et al. from the reaction of $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2Cl_2(\mu\text{-Cl})_2]$ with molecular hydrogen, along with the benzene, mesitylene, hexamethylbenzene and 1,2,4,5-tetramethylbenzene derivatives [7]. All these compounds are well characterised, but there is no structural information about these hydrido complexes; furthermore, the other halogen analogues are not known.

We therefore decided to study systematically the reactivity of complex **1** towards NaX and HX in order to complete the series of hydrido complexes $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2X_2(\mu\text{-Cl})(\mu\text{-H})]$ (X = F, Cl, Br, I) and also the series of the tetrahalo complexes $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2Ru_2X_2(\mu\text{-X})_2]$ (X = Cl, Br, I). The single-crystal structure analysis of the starting complex **1** is also reported.

2. Experimental

2.1. General

The following chemicals were purchased from commercial sources and used without further purification: $RuCl_3 \cdot nH_2O$ (Johnson Matthey), (*R*)-(−)-5-isopropyl-2-methyl-1,3-cyclohexadiene (Merck), $KB[CH(CH_3)\text{-}C_2H_5]_3H$ (Aldrich), $MgSO_4$, NaI (Fluka), NaBr (Fluka), HF 47% (Fluka), HCl 48% (Fluka), HBr 37% (Fluka) and HI 47% (Fluka). All reactions and manipulations were carried out under nitrogen using standard Schlenck techniques. The organic solvents were dried over appropriate drying agents, distilled and

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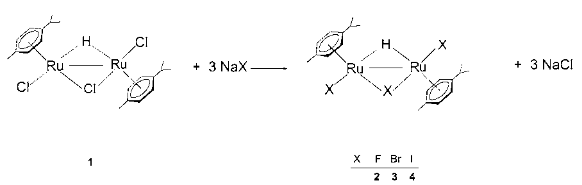
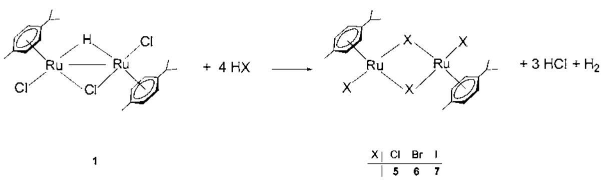
stored under nitrogen prior to use. The complex $[(p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)\text{RuCl}_2(\mu\text{-Cl})(\mu\text{-H})]_2$ (**1**) was synthesized according to published methods [7]. For the preparative thin-layer chromatography, 20 × 20 cm plates coated with silica gel (0.6–0.8 mm) without UV-indicator (Fluka Silica Gel G.) were used. The NMR spectra were recorded on a Varian Gemini 200 BB instrument using a Sun Varian Station for the treatment of the spectra. The mass spectra (FAB) were recorded by Professor Titus A. Jenny, University of Fribourg, using a VG7070E spectrometer. Microanalytical data were obtained from the Mikroelementaranalytisches Laboratorium der ETH Zürich, Switzerland.

2.2. Preparation of $[(p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)_2\text{Ru}_2\text{F}_2(\mu\text{-F})(\mu\text{-H})]$ (2)

A solution of NaF (655.2 mg, 15.6 mmol) in 8 ml of methanol was added dropwise to a solution of **1** (900 mg, 1.56 mmol) in 30 ml of methanol. The mixture was stirred for 48 h at 25°C. After removal of the solvent in vacuo, the orange–red residue was dissolved in 20 ml of dichloromethane and the solution was filtered to remove the excess NaF. The filtrate was concentrated to 5 ml and submitted to thin-layer chromatography using a 4:1:2 mixture of cyclohexane, chloroform and acetone as eluent. After separation of the two bands (orange and red), the plates were dried (1 min) and reintroduced into the same eluent mixture. When the red band (starting material) had separated completely from the orange band, the product was extracted from the main orange band with acetone. Evaporation to dryness gave **2** as an orange–red powder (160.9 mg, 19% yield). MS: *m*/*z* 528.4. Found: C, 45.36; H, 5.31. Calc. for $\text{C}_{20}\text{H}_{29}\text{Ru}_2\text{F}_3$: C, 45.42; H, 5.49%.



Scheme 1.



Scheme 2.

2.3. Preparation of $[(p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)_2\text{Ru}_2\text{Br}_2(\mu\text{-Br})(\mu\text{-H})]$ (3)

A solution of NaBr (178.05 mg, 1.73 mmol) in 5 ml of methanol was added dropwise to a solution of **1** (100 mg, 0.173 mmol) in 30 ml of methanol. The mixture was stirred for 24 h at 25°C. After removal of the solvent, the red residue was dissolved in 20 ml of dichloromethane and the solution was filtered to remove the excess NaBr. The filtrate was concentrated and the residue was submitted to thin-layer chromatography using a 8:1 mixture of cyclohexane and isopropanol as eluent. The dark red main band containing **3** was extracted with acetone, followed by evaporation to dryness to give the product as a dark red powder (196 mg, 63% yield). MS: *m*/*z* 713.8. Found: C, 34.88; H, 4.26. Calc. for $\text{C}_{20}\text{H}_{29}\text{Ru}_2\text{Br}_3\cdot\text{C}_3\text{H}_6\text{O}$: C, 34.86; H, 4.62%.

2.4. Preparation of $[p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)_2\text{Ru}_2\text{Br}_2(\mu\text{-Br})(\mu\text{-H})]$ (4)

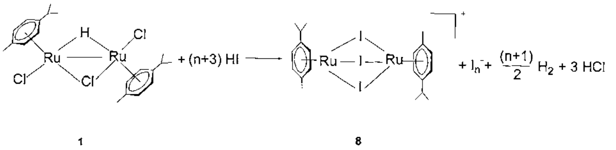
A solution of NaBr (178.5 mg, 1.73 mmol) in 5 ml of methanol was added dropwise to a solution of **1** (110 mg, 0.190 mmol) in 30 ml of methanol. The mixture was stirred for 24 h at 25°C. After removal of the solvent, the red residue was dissolved in the minimum quantity of methanol. The product was separated by thin-layer chromatography using a 8:1 mixture of cyclohexane and isopropanol as eluent. The violet main band containing **4** was extracted with acetone, followed by evaporation to dryness to give the product as a violet powder (190 mg, 85% yield). MS: *m*/*z* 853.61. Found: C, 31.04; H, 3.85. Calc. for $\text{C}_{20}\text{H}_{29}\text{Ru}_2\text{I}_3\cdot\text{C}_3\text{H}_6\text{O}$: C, 31.08; H, 4.04%.

2.5. Preparation of $[(p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)_2\text{RuX}_2(\mu\text{-X})_2]$ (5: X = Cl, 6: X = Br, 7: X = I)

To a solution of compound **1** (150 mg, 0.259 mmol) in 25 ml of THF, 0.692 mmol of concentrated HX (37%, X = Cl; 48%, X = Br; 47%, X = I) was added. The mixture was refluxed for 5 h and then the solution was filtered and evaporated to dryness. The residue was washed with 5 ml of H_2O and then taken up in dichloromethane (20 ml). After drying with MgSO_4, the solution was filtered and evaporated to dryness, which gave the analytically pure product (yield: **5**, 186.48 mg, 85%; **6**, 240.76 mg, 79%; **7**, 342.35 mg, 74%), identified by the NMR data [8,9].

2.6. Preparation of $[(p\text{-Me-C}_6\text{H}_4\text{-Pr}^i)_2\text{Ru}_2(\mu\text{-I})_3]\text{I}$ (cation 8)

An excess of HI (0.275 ml, 2.48 mmol) was added to a solution of compound **1** (120 mg, 0.207 mmol) in 35



Scheme 3.

Table 1
Crystallographic data of compound **1**

Empirical formula	$C_{20}H_{29}Cl_3Ru_2$
M (g mol^{-1})	577.92
Temperature (K)	223(2)
Crystal system	Orthorhombic
Space group	*Pbca*
a (Å)	13.7542(18)
b (Å)	16.0142(18)
c (Å)	19.352(2)
U (Å^3)	4267.7(9)
Z	8
D_{calc} (g cm^{-3})	1.801
F(000)	2304
θ limits (°)	2.10–25.96
hkl ranges	-16 to 16, -19 to 19, -23 to 23
Reflections collected	27768
Independent reflections	4123
Reflections observed [$I = 2\sigma(I)$]	3390
Goodness-of-fit on F^2 [a]	0.976
Final R indices [$I = 2\sigma(I)$] [b]	$R_1 = 0.0284$, $wR_2 = 0.0526$
R indices (all data)	$R_1 = 0.0365$, $wR_2 = 0.0555$

[a] $S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n, number of reflections; p, number of parameters).

[b] $R_1 = \Sigma\|F^o| - |F_c\|/\Sigma|F^o|$. $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F^o)^4]^{1/2}$.

ml of THF. The mixture was refluxed for about 6 h and then filtered. The solvent was removed in vacuo, and the residue was dissolved in benzene. Crystallization at room temperature gave crystals of [(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$(μ-I)$_3$][I$_n$] in 63% yield. The product was identified by the IR and NMR data [11].

2.7. X-ray crystallography

An orange crystal of compound **1** was mounted on a Stoe Imaging Plate Diffractometer System (Stoe and Cie, 1995), equipped with a one-circle φ goniometer and a graphite-monochromator. Data collection was performed at -50°C using Mo–K$_\alpha$ radiation ($\lambda = 0.71073$ Å). In total 170 exposures (3 min per exposure) were obtained at an image plate distance of 70 mm with $0 < \varphi < 170°$ and with the crystal oscillating through 1° in φ. The resolution was $D_{min} - D_{max}$ 12.45–0.81 Å.

The structure was solved by direct methods using the program SHELXS-97 [12], and refined by full-matrix least-squares on F^2 [13] with SHELXL-97 [13]. The positions of the hydride was derived from Fourier difference maps and refined, while the remaining hydrogen atoms of the organic ligand were included in calculated positions and treated as riding atoms using SHELXL-97 default parameters. Crystallographic details are given in Table 1.

3. Results and discussion

The hydrido trichloro complex [(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$Cl$_2$(μ-Cl)(μ-H)] (**1**) reacts in methanol with sodium halides to give, by simple halide exchange, the corresponding hydrido trihalo derivatives [(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$X$_2$(μ-X)(μ-H)] (**2**: X = F, **3**: X = Br, **4**: X = I), unknown hitherto (Scheme 1). All these complexes are air-stable, crystalline solids which are intensely coloured (**2**: orange, **3**: red, **4**: violet) and readily soluble in polar organic solvents and in aromatic hydrocarbons.

The new hydrido complexes were characterised by their ^{1}H-NMR data: apart from the typical signals of the two equivalent p-cymene ligands, there is a singlet in the range of bridging hydrido ligands (Table 2). In the mass spectra (electrospray), all complexes **2**–**4** show the molecular peak with the expected isotope pattern.

The halide exchange reaction of [(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$Cl$_2$(μ-Cl)(μ-H)] (**1**) can also involve the hydrido ligand: if, instead of the sodium halide, the corresponding acid HX is used, the known [8–10] tetrahalo complexes [(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$X$_2$(μ-X)$_2$] (**5**: X = Cl, **6**: X = Br, **7**: X = I) are obtained (Scheme 2). The reaction takes place in refluxing THF (Scheme 3).

Table 2
NMR data of compounds **2**–**4**

Complex	δ (^{1}H) [a]	δ (^{19}F) [a]
[(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$F$_2$(μ-H)(μ-F)] (**2**)	1.47, 1.44 (d, 6H, CH(CH_3)$_2$), 2.20 (s, 3H, C$_6$H$_4$CH_3), 3.01(sp, 1H, CH(CH$_3$)$_2$) 5.67, 5.54 (d, 2H, C$_6$H$_4$), 5.46, 5.39 (d, 2H, C$_6$H$_4$), -9.97 (s, 1H, Ru$_2$$H$)	-127.3, -151.7
[(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$Br$_2$(μ-H)(μ-Br)] (**3**)	1.42, 1.46 (d, 6H, CH(CH_3)$_2$), 2.39 (s, 3H, C$_6$H$_4$CH_3), 3.04 (sp, 1H, CH(CH$_3$)$_2$) 5.00, 5.38 (d, 2H, C$_6$H$_4$), 5.59, 5.69 (d, 2H, C$_6$H$_4$), -11.02 (s, 1H, Ru$_2$$H$)	
[(p-Me-C_6H_4-Pr^i)$_2$Ru$_2$I$_2$(μ-H)(μ-I)] (**4**)	1.08, 1.14 (d, 6H, CH(CH_3)$_2$), 1.99 (s, 3H, C$_6$H$_4$CH_3), 2.81 (sp, 1H, CH(CH$_3$)$_2$) 4.98, 5.15 (d, 2H, C$_6$H$_4$), 5.25, 5.42 (d, 2H, C$_6$H$_4$), -13.16 (s, 1H, Ru$_2$$H$) [b]	

[a] Measured in CDCl$_3$.

[b] Measured in C$_6$D$_6$.

Table 3
NMR data of complexes **5**–**8**

Complex δ	δ (^{1}H) [a]
$[(p\text{-Me-C}_6H_4\text{-Pr}^i)\text{RuCl}(\mu\text{-Cl}]_2$ (**5**)	1.28 (d, 6H, CH(CH_3)$_2$), 2.16 (s, 3H, C$_6$H$_4$CH_3), 2.92 (sp, 1H, CH(CH$_3$)$_2$) 5.33 (d, 2H, C$_6$H$_4$), 5.47 (d, 2H, C$_6$H$_4$)
$[(p\text{-Me-C}_6H_4\text{-Pr}^i)\text{RuBr}(\mu\text{-Br}]_2$ (**6**)	1.29 (d, 6H, CH(CH_3)$_2$), 2.23 (s, 3H, C$_6$H$_4$CH_3), 2.98 (sp, 1H, CH(CH$_3$)$_2$) 5.40 (d, 2H, C$_6$H$_4$), 5.52 (d, 2H, C$_6$H$_4$)
$[(p\text{-Me-C}_6H_4\text{-Pr}^i)\text{RuI}(\mu\text{-I})]_2$ (**7**)	1.25 (d, 6H, CH(CH_3)$_2$), 2.36 (s, 3H, C$_6$H$_4$CH_3), 2.98 (sp, 1H, CH(CH$_3$)$_2$) 5.43 (d, 2H, C$_6$H$_4$), 5.48 (d, 2H, C$_6$H$_4$)
$[(p\text{-MeC}_6H_4\text{-Pr}^i)_2\text{Ru}_2(\mu\text{-I})_3]^+$ (**8**)	1.30 (d, 6H, CH(CH_3)$_2$), 2.34 (s, 3H, C$_6$H$_4$CH_3), 2.90 (sp, 1H, CH(CH$_3$)$_2$) 5.56 (d, 2H, C$_6$H$_4$), 5.66 (d, 2H, C$_6$H$_4$)

[a] Measured in CDCl$_3$.

In the case of HI, the reaction with **1** is sensitive to stoichiometry: while a 1:4 ratio leads to the tetraiodo complex **7**, an excess of HI (1:10) gives rise to the formation of the known [11] cation $[(p\text{-Me-C}_6H_4\text{-Pr}^i)_2\text{Ru}_2(\mu\text{-I})_3]^+$ (**8**), which crystallizes as the polyiodide salt (Table 3).

The molecular structure of the hydrido trichloro complex **1** was solved by a single-crystal X-ray structure analysis using a suitable crystal grown in benzene (Fig. 1). Interatomic distances and angles are given in Table 4. The Ru–Ru distance of 2.9573(4) Å is in accordance with a metal–metal bond, as expected from the electron count (34e). In the isoelectronic hydrido trichloro complexes $[(C_5Me_5)_2Rh_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ [14] and $[(C_5Me_5)_2Ir_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ [15], the metal–metal distances [Rh–Rh 2.906(1) Å, Ir–Ir 2.903(1) Å] are also in the range of bonding interactions. By contrast, in the tetrahalo complexes **5** and **6**, the Ru–Ru distances [3.853(1) and 4.092(1) Å, respectively] [10] are too long for bonding interactions, in accordance with the electron count of 36e.

The angle Ru1–Cl1–Ru2 is found to be 75.616(19)° and compares well with the corresponding angles of 72.65(8) and 73.20(6)° in $[(C_5Me_5)_2Rh_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ [14] and $[(C_5Me_5)_2Ir_2Cl_2(\mu\text{-Cl})(\mu\text{-H})]$ [15], which is in line with a description of the bonding situation in terms of a closed RuHRu three-center two-electron interaction in which there is substantial metal–metal bonding. The angles Cl2–Ru1–Ru2 [88.98(2)°] and Cl3–Ru2–Ru1 [87.48(2)°] are almost right angles, the torsion angle

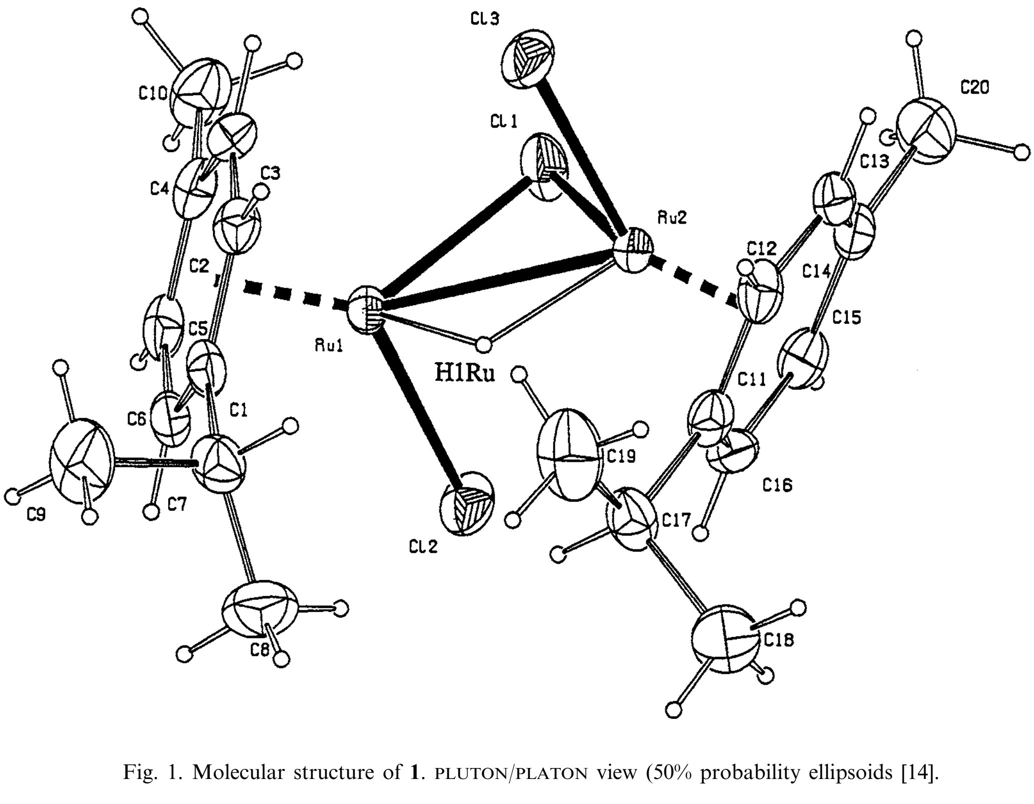


Fig. 1. Molecular structure of **1**. PLUTON/PLATON view (50% probability ellipsoids [14].

Table 4
Selected bond lengths (Å) and bond angles (°) for **1**

H(1Ru)–Ru(1)–Ru(2)	52.211(17)	H(1Ru)–Ru(2)–Ru(1)	0.0000(0)
Bond lengths (Å)			
Ru(1)–H(1Ru)	1.75(2)	Ru(2)–H(1Ru)	1.76(3)
Ru(1)–C(2)	2.151(2)	Ru(2)–C(12)	2.154(3)
Ru(1)–C(3)	2.171(3)	Ru(2)–C(16)	2.170(3)
Ru(1)–C(1)	2.178(2)	Ru(2)–C(15)	2.177(3)
Ru(1)–C(6)	2.179(3)	Ru(2)–C(11)	2.178(3)
Ru(1)–C(5)	2.207(3)	Ru(2)–C(13)	2.198(3)
Ru(1)–C(4)	2.211(3)	Ru(2)–C(14)	2.216(3)
Ru(1)–Cl(2)	2.4053(8)	Ru(2)–Cl(3)	2.4122(9)
Ru(1)–Cl(1)	2.4115(7)	Ru(2)–Cl(1)	2.4127(7)
Ru(1)–Ru(2)	2.9573(4)		
Bond angles (°)			
H(1Ru)–Ru(1)–Cl(2)	85.9(9)	H(1Ru)–Ru(2)–Cl(3)	90.6(9)
C(3)–Ru(1)–Cl(2)	160.53(7)	C(12)–Ru(2)–Cl(3)	90.81(8)
H(1Ru)–Ru(1)–Cl(1)	84.6(8)	H(1Ru)–Ru(2)–Cl(1)	84.5(7)
C(3)–Ru(1)–Cl(1)	93.56(8)	C(12)–Ru(2)–Cl(1)	164.69(8)
H(1Ru)–Ru(1)–Ru(2)	32.5(8)	H(1Ru)–Ru(2)–Ru(1)	32.5(7)
C(5)–Ru(1)–Ru(2)	172.77(9)	Cl(3)–Ru(2)–Ru(1)	87.48(2)
Cl(2)–Ru(1)–Ru(2)	88.98(2)	Cl(1)–Ru(2)–Ru(1)	52.173(18)
Cl(1)–Ru(1)–Ru(2)	52.211(17)	Ru(1)–Cl(1)–Ru(2)	75.616(19)
Cl(3)–Ru(2)–Cl(1)	89.77(3)	Cl(2)–Ru(1)–Cl(1)	88.82(3)

Cl3–Ru2–Ru1–Cl2 being −179.04(3)°, so that the two terminal chloro ligands are almost perfectly opposite to each other and lie in the same plane with the Ru–Ru backbone. The angles Cl3–Ru2–Cl1 and Cl2–Ru1–Cl1 are found to be 89.77(3) and 88.82(3)°, respectively; therefore the plane defined by the chloro bridge and the Ru_2 backbone is almost perpendicular to that defined by the termine chloro ligands and the two ruthenium atoms. The bridging hydride ligand is almost in the same plane as the chloro bridge, the torsion angle H–Ru1–Cl1–Ru2 being −3.7(9)°.

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