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Syntheses and characterization of arene ruthenium (II) complexes containing *N*,*N′*-donor Schiff base ligands. Crystal and molecular structure of [(η^6-C_6Me_6)Ru(C_5H_4N-2-CH=N–C_6H_4-*p*-NO_2)]PF_6

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Abstract

Reaction of [(η^6-C_6Me_6)$RuCl_2$]$_2$ **(1a)** with *N*,*N′*-donor Schiff bases viz., para-substituted *N*-(2-pyridinylmethylene)phenylamines yielded complexes of the formulation [(η^6-C_6Me_6)Ru(C_5H_4N-2-CH=N–C_6H_4-*p*-X)Cl]$^+$ **2a**–**e** where C_6Me_6 = hexamethylbenzene, X = H (**2a**), CH_3 (**2b**), OCH_3 (**2c**), Cl (**2d**) and NO_2 (**2e**). Reaction of **1a** and [(η^6-*p*-cymene)$RuCl_2$]$_2$ **(1b)** where *p*-cymene = ($CH_3C_6H_4CH(CH_3)_2$) with *N*-(2-pyridinylmethylene)cyclohexylamine (**2-PC**) yielded complexes of the type [(η^6-arene)Ru(**2-PC**)Cl]$^+$ (arene = C_6Me_6 (**3a**); *p*-cymene, (**3b**)) respectively. All these complexes were isolated as their PF_6 salt. These complexes were characterized by spectroscopic and analytical data. The molecular structure of [(η^6-C_6Me_6)Ru(C_5H_4N-2-CH=N–C_6H_4-*p*-NO_2)Cl]PF_6 **2e** has been determined by X-ray crystallography.

Keywords: *p*-Cymene; Hexamethylbenzene; *N*-(2-pyridinylmethylene)phenylamines; *N*-(2-pyridinylmethylene)cyclohexylamine; Schiff base; Ruthenium

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1. Introduction

Recent interest in half sandwich arene ruthenium complexes arises from their use as catalysts [1] in organic syntheses and also recent studies have shown that they are found to inhibit cancer cell growth [2]. Moreover, the photophysics, photochemistry and redox properties of α,α′-diimines ruthenium [3] complexes have been prolific areas of research over the past two decades. There are few reports of arene ruthenium complexes with α,α′-diimines [4] available in literature. Herein, we would like to report the synthesis and characterization of new cationic half sandwich ruthenium complexes of *N*,*N′*-Schiff base ligands. The molecular structure of the complex [(η^6-C_6Me_6)Ru(C_5H_4N-2-CH=N–C_6H_4-*p*-NO_2)Cl]PF_6 is also reported.

2. Experimental

All reactions were carried out in distilled and dried solvents. Pyridine 2-carboxaldehyde (Fluka) was used as received. All liquid aromatic amines were reagent grade and were distilled prior to use and solid aromatic amines were used as such. ^{1}H NMR spectra were recorded in CD_3CN, $CDCl_3$ and $(CD_3)_2CO$ solvents with tetramethylsilane as an internal standard and recorded on a Brucker ACF-300 MHz spectrometer. Infrared spectra were taken on a Perkin-Elmer model 983 spectrophotometer using KBr pellets. Elemental analysis was performed in Perkin-Elmer-2400 CHNS/O analyser. Conductivity measurement was performed in Wayne Kerr Automatic Precision Bridge B-905. The ligands C_5H_4N-2-CH=N–C_6H_4-*p*-X (where X = H, CH_3, OCH_3, Cl, NO_2) and **2-PC** were prepared according to

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the literature methods [5]. The precursor complexes [(arene)RuCl$_2$]$_2$, arene = C$_6$Me$_6$ **1a,** *p*-cymene **1b** were prepared by known methods [6].

2.1. Preparation of [(η^6-C$_6$Me$_6$)Ru(C$_5$H$_4$N-2-CH=N–C$_6$H$_4$-p-X)Cl]PF$_6$ (2a–e) (X = H, CH$_3$, OCH$_3$, Cl, NO$_2$)

These complexes were prepared using a general procedure in which the mixture of [(η^6-C$_6$Me$_6$)RuCl$_2$]$_2$ (0.15 mmol) and the ligand (0.30 mmol) in methanol (20 ml) were stirred at room temperature for 1 h. The resulting solution was filtered and NH$_4$PF$_6$ (0.32 mmol) was added to the clear solution. Then stirred at 0 °C whereby orange compound precipitated for complexes with X = CH$_3$, Cl, NO$_2$ which was collected and washed with water, methanol and finally with diethylether and dried in vacuum.

For the complexes with X = H and OMe, the dark brown solution was stirred for 2 h and the solvent was removed under reduced pressure. The residue was extracted with dichloromethane and filtered through short silica column. The resulting solution was reduced in volume to about 2 ml and hexane (15 ml) was added to give red brown compound, which was recrystallized from the mixture of acetone and hexane to give orange crystals. Yield ranges from 70–80%.

2.2. Preparation of [(η^6-arene)Ru(2-PC)Cl]PF$_6$; arene = C$_6$Me$_6$ (3a), p-cymene (3b)

The complexes were prepared in similar manner to that of the preparation of **2** (X = H, OMe) where the oily product obtained was washed several times with hexane to gave dark brown solid in 65% yield.

2.3. Crystallographic analysis

X-ray quality crystals of complex **2e** as PF$_6$ salt, were obtained by a diffusion technique in which diethylether was carefully layered into acetonitrile solution of the complex. An orange–red crystal was mounted on a glass fiber with epoxy cement. X-ray intensity data were collected on a Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo Kα radiation (λ = 0.71069 Å) at a temperature of 293 K. The intensity data were corrected for Lorentz and polarization effects and for absorption using REQAB [7]. The structure was solved by direct methods (SIR92) [8]. Refinement was by full-matrix least squares based on F^2 using SHELXL-93 [9]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w = 1/(\sigma^2(F_o^2) + 0.0693P^2 + 2.5252P)$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a 'riding' model. Refinement converged to $R_1 = 0.0534$ and $wR_2 = 0.1332$ for 5471 reflections for which $F > 4\sigma(F)$ and $R_1 = 0.0592$, $wR_2 = 0.1378$ and GOF = 1.138 for all 5971 unique, non-zero reflections and 350 variables[1].

Table 1 shows cell information, data collection parameters, and refinement data. Table 2 lists bond distances and bond angles. Fig. 1 is an ORTEP [10] representation of the molecule with 30% probability thermal ellipsoids displayed.

3. Results and discussion

The reaction of [(η^6-arene)RuCl$_2$]$_2$ with neutral *N*,*N′*-Schiff base ligands yielded complexes of the type **2** and **3** in fairly good yield. The orange–red colored complexes of **2** are air stable while the dark brown complexes of **3** are slightly air sensitive. These complexes are insoluble in water, chlorinated, alcohol and non-polar solvents but soluble in other polar solvents. These complexes exhibited 1:1 electrolyte system [11] in acetonitrile solution (~130 ohm^{-1} cm^2 mol^{-1}).

Table 1
Summary of structure determination of compound [(η^6-C$_6$Me$_6$)Ru(C$_5$H$_4$N-2-CH=N–C$_6$H$_4$-*p*-NO$_2$)]PF$_6$ (**2e**)

Formula	RuC$_{24}$H$_{27}$N$_3$PO$_2$F$_6$Cl
Formula weight	670.98
Crystal class	monoclinic
Space group	$P2_1/n$ (#14)
Z	4
Cell constants	
a (Å)	10.65120(10)
b (Å)	11.71340(10)
c (Å)	21.7134(3)
β (°)	98.1180(10)
V (Å^3)	2681.86(5)
μ (cm^{-1})	8.13
Crystal size (mm)	0.42 × 0.11 × 0.06
D_{calc} (g cm^{-3})	1.662
F(000)	1352
Radiation	Mo Kα (λ = 0.71069Å)
$2\theta \leq 28$ range (°)	5.14–54.96
hkl collected	$-13 \leq h \leq 13$; $-14 \leq k \leq 15$; $-28 \leq l$
No. reflections measured	26 725
No. unique reflections	5971 ($R_{int} = 0.0312$)
No. observed reflections	5471 ($F > 4\sigma$)
No. reflections used in refinement	5971
No. parameters	350
R indices ($F > 4\sigma$)	$R_1 = 0.0534$, $wR_2 = 0.1332$
R indices (all data)	$R_1 = 0.0592$, $wR_2 = 0.1378$
GOF	1.138
Final difference peaks (e Å^{-3})	+0.659, −0.638

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[1] $R_1 = \Sigma||F_o| - |F_c||/\Sigma F_o|$, $wR_2 = \{\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2\}^{1/2}$, GOF = $\{\Sigma w(F_o^2 - F_c^2)^2/(n-p)\}^{1/2}$, where n = the number of reflections and p = the number of parameters refined.

Table 2
Selected bond lengths (Å) and angles (°) for $[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N\text{-}C_6H_4\text{-}p\text{-}NO_2)]PF_6$ (**2e**)

Bond lengths				Bond angle	
Ru–N1	2.099(3)	Ru–C13	2.204(3)	N1–Ru–N2	75.90(11)
Ru–N2	2.106(3)	C13–C14	1.435(5)	N1–Ru–C18	96.13(13)
Ru–C14	2.250(3)	C14–C15	1.395(6)	N2–Ru–C13	94.42(12)
Ru–C15	2.227(3)	C15–C16	1.448(6)	C7–N2-Ru	126.5(2)
Ru–C16	2.225(4)	C16–C17	1.410(6)	C6–N2–Ru	116.3(2)
Ru–C17	2.251(4)	C17–C18	1.422(6)	C5–N1-Ru	116.2(2)
Ru–C18	2.190(3)	N2–C6	1.288(4)	C15–Ru–Cl	90.79(11)
N3–O1	1.184(8)	N2–C7	1.431(4)	C16–Ru–Cl	92.52(12)
N3–O2	1.207(8)			N1–Ru-–Cl	83.92((9)
				N2–Ru–C	85.84(8)
				O1–N3–O2	123.7(6)
				O1–N3–C10	118.9(6)
				O2–N3–C10	117.4(6)

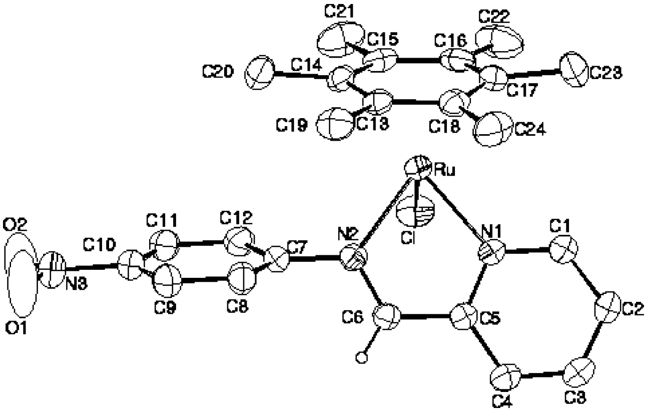

Fig. 1. ORTEP view of the complex $[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N\text{-}C_6H_4\text{-}p\text{-}NO_2)]$ **2e** showing 30% probability thermal ellipsoids with atom numbering scheme. PF_6 omitted for clarity.

$$[(\eta^6\text{-}C_6Me_6)RuCl_2]_2 + 2(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}X)$$

$$\rightarrow [(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}X)Cl]PF_6$$

$(X{=}H,\ CH_3,\ OCH_3,\ Cl,\ NO_2$ (**2a-e**))

$$[(\eta^6\text{-arene})RuCl_2]_2 + 2(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_{11})$$

$$\rightarrow [(\eta^6\text{-arene})Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_{11})Cl]PF_6$$

arene = $(\eta^6\text{-}C_6Me_6)$ (**3a**); *p*-cymene (**3b**)

These complexes are characterized by elemental analysis, IR and ^{1}H NMR spectral data (Table 3). All these complexes gave satisfactory analytical data. IR spectra of the complexes showed medium intensity bands in the range of 1600–1400, 770–557 cm^{-1} for the benzene ring. Complex **2e** has showed characteristic IR bands for ν_{NO_2} at 1526–1488 cm^{-1} similar to the range observed for ν_{NO_2} of other reported compounds. ^{1}H NMR data of complexes **2** suggests a 1:1 ratio of the hexamethylbenzene and Schiff base ligands. ^{1}H NMR spectra of the complexes **2** and **3a** display sharp singlet for methyl protons of hexamethylbenzene at around δ 1.8 and 2.1, respectively and the peaks for the ligand moiety appeared in the range of δ 7–9. The methine proton of **2** and **3** appear at around δ 8.4 and 8.8, respectively. This proton resonated in the same range of corresponding *p*-cymene compounds [4b]. The α-proton of the pyridine ring of complex **3** showed a up field shift of about δ 0.3 in comparison to complex **2** due to the cyclohexyl group. ^{1}H NMR spectrum of complex **3b** suggests the presence of η^6-*p*-cymene and **2-PC** in 1:1 ratio. The protons of isopropyl group of the η^6-*p*-cymene ligand appear as two sets of doublets at around δ 1.16 due to the loss of planarity of the benzene ring because of steric nature of the ligand (Fig. 2) [12]. Septet at δ 2.70 was observed for single proton of isopropyl group. The benzene ring protons of *p*-cymene appeared as two doublets and a triplet in the range of δ 5.92–6.23, respectively due to long range coupling with isopropyl-proton. ^{1}H NMR spectrum of complex **3b** is shown in Fig. 2.

Electronic spectra of these complexes in acetonitrile exhibited bands in the range of 340–400 nm (Table 4). These low-energy absorption were assigned to Ru (dπ) to ligand (π*) metal to ligand charge transfer (MLCT) transition.

The complex $[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N\text{-}C_6H_4\text{-}p\text{-}NO_2)]PF_6$ (**2e**) has been structurally characterized by X-ray crystallography. An ORTEP view of the complex is shown in Fig. 1. The complex **2e** exist as half sandwich complex with the arene ring occupying three facial sites. The ruthenium atom is π bonded to the hexamethylbenzene ligand with an average Ru–C distance 2.22 Å similar to that observed in related hexamethylbenzene ruthenium complexes [13]. The distance between ruthenium and the chloride ligand is 2.39 Å. In addition to being bonded to the arene and the chloride ligands, the ruthenium atom is also directly coordinated to two nitrogen atoms of the Schiff base ligand with an average distance of 2.10 Å. The bite angle

Table 3
IR and ^{1}H NMR data of the complexes **2a–e** and **3a–b**

Complex	IR bands (KBr pellets, cm^{-1})	^{1}H NMR, δ (ppm) (Schiff base)	H NMR, δ (ppm) (Arene)
2a	1590 ($\nu_{C–N}$) 844 ($\nu_{P–F}$)	9.16 (d, 1H, 3), 8.80 (s, 1H), 8.34 (m, 2H), 7.96 (m, 1H), 7.85 (d, 2H), 7.63 (m, 2H)	1.87 (s, 18 H)
b	1586 ($\nu_{C–N}$) 844 ($\nu_{P–F}$)	8.90 (d, 1H, 3), 8.41 (s, 1H), 8.11 (m, 2H), 7.77 (t, 1H), 7.59 (d, 2H), 7.50 (d, 2H), 2.44 (s, 3H)	1.87 (s, 18 H)
c	1589 ($\nu_{C–N}$) 839 ($\nu_{P–F}$)	8.89 (d, 1H, 3), 8.39 (s, 1H), 8.10 (m, 2H), 7.75 (t, 1H), 7.62 (d, 1H), 7.13 (d, 1H), 3.98 (s, 3H)	1.88 (s, 18H)
d	1589 ($\nu_{C–N}$) 843 ($\nu_{P–F}$)	8.92 (m, 1H, 3), 8.46 (s, 1H), 8.12 (m, 2H), 7.79 (t, 1H), 7.69-7.58 (m, 4H)	1.86 (s, 18H)
e	1584 ($\nu_{C–N}$) 836 ($\nu_{P–F}$)	8.96 (m, 1H), 8.53 (s, 1H), 8.45 (m, 2H), 8.17 (m, 2H), 7.92-7.83 (m, 4H)	1.93 (s, 18H)
3a[a]	1597 ($\nu_{C–N}$) 840 ($\nu_{P–F}$)	9.43 (d, 1H, 6), 8.74 (s, 1H), 8.42 (d, 1H), 7.99 (m, 2H), 7.65 (m, 1H), 2.00-1.24 (m)	2.14 (s, 18H)
3b[a]	1598 ($\nu_{C–N}$) 838 ($\nu_{P–F}$)	9.54 (d, 1H, 6), 8.86 (s, 1H), 8.29-8.24 (m, 2H), 7.81 (t, 1H, 6), 1.95-1.98 (m)	6.23 (dd, 2H, 6), 5.92 (t, 2H, 6), 2.70 (sp, 1H), 2.28 (s, 3H), 1.14 (dd, 6H, 6)

^{1}H NMR of complexes taken in CD_3CN.
[a] In $(CD_3)_2CO$, s, singlet; d, doublet; t, triplet; m, multiplet.

Table 4
MLCT absorption at room temperature

Complex[a]	λ_{max}, nm
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}H)Cl]PF_6$	374
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}CH_3)Cl]PF_6$	366
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}OCH_3)Cl]PF_6$	360
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}Cl)Cl]PF_6$	374
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_4\text{-}p\text{-}NO_2)Cl]PF_6$	400
$[(\eta^6\text{-}C_6Me_6)Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_{11})Cl]PF_6$	342
$[(\eta^6\text{-}p\text{-Cymene})Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_{11})Cl]PF_6$	340

of the chelating ligand is 75.90(11)° not very different from other related complexes [4b]. Notably, the phenyl and pyridyl ring of the Schiff base are not coplanar.

The average C–C bond lengths in the hexamethylbenzene ring is 1.421 Å with alternate short and long bond lengths where C(13)–C(18), C(16)–C(17) and C(14)–C(15) bonds are shorter than the C(13)–C(14), C(15)–C(16) and C(17)–C(18) which could be due to the loss of planarity of the hexamethylbenzene ring. The alternate bond lengths are indicative of a contribution from the cyclohexatriene resonance structure to the overall resonance hybrid [14].

4. Conclusions

Reactions of $[(arene)RuCl_2]_2$ (arene = hexamethylbenzene, *p*-cymene) with Schiff bases viz. para-substituted *N*-(2-pyridinylmethylene)phenylamines and *N*-(2-

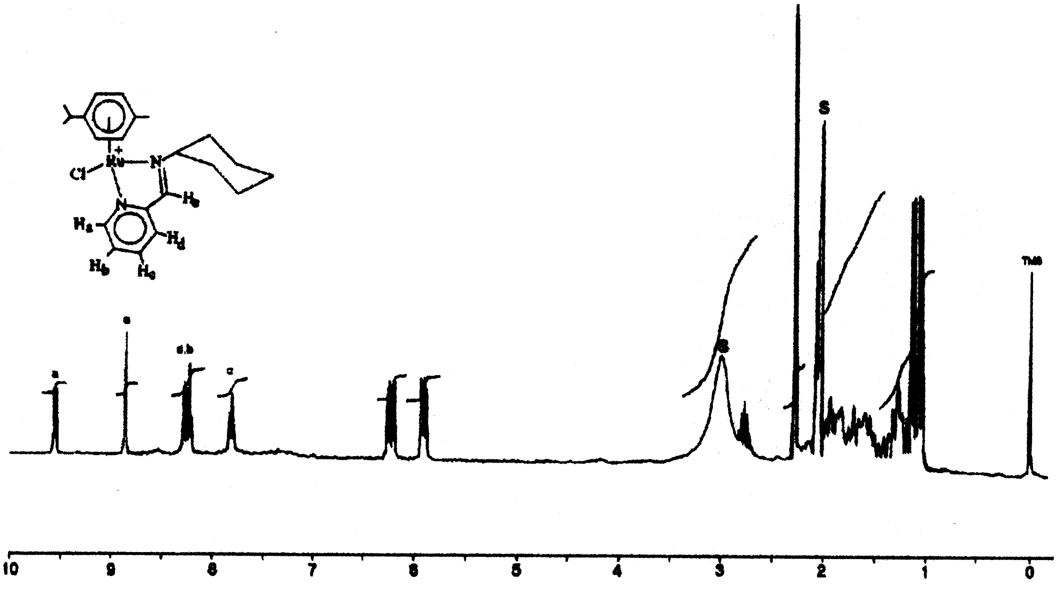

Fig. 2. ^{1}H NMR spectrum of complex $[(\eta^6\text{-}p\text{-cymene})Ru(C_5H_4N\text{-}2\text{-}CH{=}N{-}C_6H_{11})]PF_6$ (**3b**).

pyridinylmethylene)cyclohexylamine yielded mononuclear cationic complexes. The molecular structure of the representative complex **2e** has been determined by single crystal X-ray diffraction analysis.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre (CCDC), CCDC No. 193369 for complex **2e**. Copies of the information may be obtained free of charge from the director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk.

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