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Ruthenium(III) cyclometallates: Regioselective metallation of 1-pyrenyl in 1-pyrenaldehyde 4-*R*-benzoylhydrazones

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ABSTRACT

Reactions of $[Ru(PPh_3)_3Cl_2]$ with 1-pyrenaldehyde 4-*R*-benzoylhydrazones (H_2pnbh*R*, where *R* = H, Me, OMe, Cl and NO_2) in presence of NaOAc afford *ortho*-metallated ruthenium(III) complexes of formula *trans*-[Ru(pnbh*R*)$(PPh_3)_2$Cl] (**1**–**5**). The complexes have been characterized by elemental analysis, magnetic susceptibility, spectroscopic (IR, UV–vis and EPR) and cyclic voltammetric measurements. Molecular structure of **2** (*R* = Me) determined by single crystal X-ray crystallography shows a distorted octahedral $CNOClP_2$ coordination sphere around the trivalent metal centre assembled by the 1-pyrenyl *ortho*-C, azomethine-N and amidate-O donor pnbhMe^{2-}, two mutually *trans* PPh_3 and the chloride. Electronic spectra of **1**–**5** in dichloromethane display several strong bands within 555–276 nm due to ligand to metal charge transfer and ligand centred transitions. The complexes are one-electron paramagnetic (μ_{eff} = 1.90–1.99 μ_B) and display rhombic EPR spectra in frozen (130 K) dichloromethane–toluene (1:1). Cyclic voltammograms of the complexes in dimethylformamide display a ligand substituent sensitive metal centred one electron reduction couple in the $E_{1/2}$ range −0.24 to −0.31 V (vs. Ag/AgCl).

1. Introduction

Compared to the vast literature on ruthenium(II) complexes having M–C bond [1–7], ruthenium(III) complexes of that kind are a handful [8–27]. For the past few years we have been working on cyclometallated palladium and ruthenium complexes with aroylhydrazones of various monocyclic and polycyclic aromatic aldehydes [26–32]. It has been found that this pincer like [32–35] aryl-C, azomethine-N and amidate-O donor ligand system is very efficient to stabilize ruthenium not only in the trivalent state but also makes its tetravalent state accessible [26,27]. The aroylhydrazones of benzaldehyde and acetophenone can afford only *ortho*-metallated complexes [26–29]. On the other hand, aroylhydrazones of polycyclic aromatic aldehydes provide both *ortho* and *peri* positions of the polycyclic aryl moiety as the potential metallation site depending upon the position of the azomethine functionality on it [30–32]. Recently we have reported some cyclopalladated complexes with aroylhydrazones of indole-3-aldehyde and 4-*R*-1-naphthaldehydes [30,32]. In each type of complexes, the tridentate ligand forms 6,5-membered fused chelate rings due to regioselective *peri* palladation of its polycyclic aryl (indole-3 and 1-naphthalenyl) fragment. In the present work, we have reported the results in our investigation on the ruthenium coordination chemistry with 1-pyrenaldehyde 4-*R*-benzoylhydrazones (H_2pnbh*R*). The objectives were to explore the possibility of C–H activation and formation of cyclometallated species, stabilization of ruthenium(III) and regioselective metallation of the 1-pyrenyl moiety if any. In this effort, we have isolated a series of *ortho*-metallated ruthenium(III) complexes of formula *trans*-[Ru(pnbh*R*)$(PPh_3)_2$Cl] (Chart 1). In the following account, we have described synthesis, characterization and physical properties of these complexes. X-ray crystal structure of one representative complex has been also reported.

2. Experimental

2.1. Materials

$[Ru(PPh_3)_3Cl_2]$ was prepared by following a reported procedure [36]. All other chemicals were of analytical grade available commercially and were used as supplied without further purification. The solvents used were purified by following standard methods [37].

2.2. Physical measurements

Microanalyses (C, H, N) were performed by using a Thermo Finnigan Flash EA1112 series elemental analyzer. Magnetic susceptibilities were measured with the help of a Sherwood scientific

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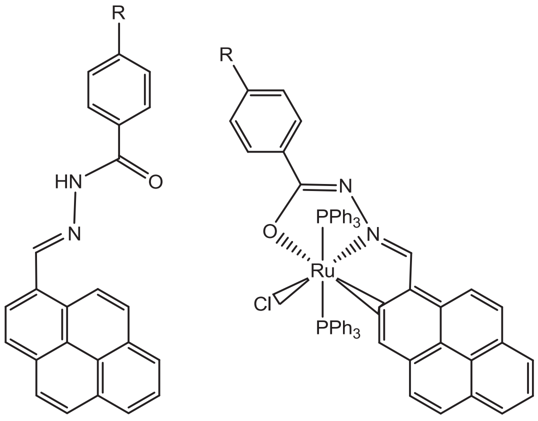

H_2pnbhR *trans*-$[Ru(pnbhR)(PPh_3)_2Cl]$

Chart 1. The chemical structure diagrams of the Schiff base system 1-pyrenaldehyde 4-*R*-benzoylhydrazone (H_2pnbhR, where $R = H$, Me, OMe, Cl and NO_2) and the complex with it (*trans*-$[Ru(pnbhR)(PPh_3)_2Cl]$).

balance. Diamagnetic corrections calculated from Pascal's constants [38] were used to obtain the molar susceptibilities. A Digisun DI-909 conductivity meter was used to measure the solution electrical conductivities. The infrared spectra were obtained by using KBr pellets on a Jasco-5300 FT-IR spectrophotometer. A Shimadzu LCMS 2010 liquid chromatograph mass spectrometer was used for the purity verification of the Schiff bases. The electronic spectra were recorded on a Cary 100 Conc UV/vis spectrophotometer. A Jeol JES-FA200 spectrometer was used to record the X-band EPR spectra. The NMR spectra were obtained with the help of a Bruker 400 MHz NMR spectrometer. A CH-Instruments model 620A electrochemical analyzer was used for cyclic voltammetric experiments with dimethylformamide solutions of the complexes containing tetrabutylammonium perchlorate (TBAP) as the supporting electrolyte. The three electrode measurements were carried out at 298 K under dinitrogen atmosphere with a platinum disk working electrode, a platinum wire auxiliary electrode and a Ag/AgCl reference electrode. Under identical condition the $E_{1/2}$ value of the Fc^+/Fc couple was observed at 0.42 V.

2.3. Synthesis of H_2pnbhH

Benzoylhydrazine (177 mg, 1.3 mmol) was added to an ethanol solution (20 ml) of 1-pyrenaldehyde (300 mg, 1.3 mmol). The mixture was boiled under reflux for 3 h and then cooled to room temperature. The pale yellow solid separated was collected by filtration, washed with ethanol and finally dried in air. The yield was 420 mg (92%).

The same procedure described above was used for the synthesis of each of the four remaining Schiff bases ($H_2pnbhMe$, $H_2pnbhOMe$, $H_2pnbhCl$ and $H_2pnbhNO_2$) in ~90% yield from 1 mol equiv each of 1-pyrenaldehyde and the corresponding 4-substituted benzoylhydrazine.

2.4. Synthesis of $[Ru(pnbhH)(PPh_3)_2Cl]$ (**1**)

Solid $[Ru(PPh_3)_3Cl_2]$ (200 mg, 0.21 mmol) was added to a methanol solution (30 ml) of H_2pnbhH (73 mg, 0.21 mmol) and NaOAc (35 mg, 0.43 mmol). The mixture was refluxed for 30 min and then cooled to room temperature. The red solid deposited was collected by filtration and dried in air. This material was dissolved in minimum amount of dichloromethane and transferred to a silica gel column packed with dichloromethane. The first moving yellow band eluted with dichloromethane/*n*-hexane (2:3) was discarded. The following red band containing the complex (**1**) was eluted using the same dichloromethane/*n*-hexane mixture but in the ratio 3:2. The red solution thus obtained was evaporated to dryness and the complex was collected as a dark red solid. The yield was 140 mg (62%).

The other four complexes **2**–**5** having the general formula $[Ru(pnbhR)(PPh_3)_2Cl]$ (**2** ($R = Me$), **3** ($R = OMe$), **4** ($R = Cl$) and **5** ($R = NO_2$)) were synthesized in 56–64% yield using $[Ru(PPh_3)_3Cl_2]$, NaOAc and the corresponding Schiff bases (in 1:2:1 mole ratio) by following procedures very similar to that described above for **1** ($R = H$).

2.5. X-ray crystallography

Single crystals of $[Ru(pnbhMe)(PPh_3)_2Cl]$ (**2**) were grown by slow evaporation of its dichloromethane–acetonitrile (1:1) solution. Unit cell parameters and intensity data at 298 K were obtained on an Oxford Diffraction Xcalibur Gemini single crystal X-ray diffractometer fitted with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The CrysAlisPro software [39] was used for data collection, reduction and absorption correction. The structure was solved by direct method and refined on F^2 by full-matrix least-squares procedures. All non-hydrogen atoms were refined with anisotropic thermal parameters. A riding model was used to include the hydrogen atoms in the structure factor calculations. The SHELX-97 programs [40] provided in the WinGX package [41] were used for structure solution and refinement. The Platon [42] and the Mercury [43] packages were used for molecular graphics. Selected crystallographic data are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and characterization

The Schiff bases H_2pnbhR ($R = H$, Me, OMe, Cl and NO_2) were obtained in very good yields (~90%) in the condensation reactions of 1-pyrenaldehyde and the corresponding 4-*R*-benzoylhydrazines in 1:1 mole ratio in ethanol. The identity and structures of the Schiff bases were established by elemental analysis and spectroscopic (infrared, electronic, mass and ^1H NMR) measurements. These characterization data are given in Tables S1 and S2 (Supplementary material). Reactions of $[Ru(PPh_3)_3Cl_2]$, the corresponding H_2pnbhR and NaOAc in 1:1:2 mole ratio in methanol under aerobic

Table 1
Selected crystallographic data for *trans*-$[Ru(pnbhMe)(PPh_3)_2Cl]$ (**2**).

Empirical formula	$RuC_{61}H_{46}N_2OP_2Cl$
Formula weight	1021.46
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	15.0653(4)
b (Å)	14.8940(4)
c (Å)	22.3055(5)
β (°)	94.404(2)
V (Å^3)	4990.2(2)
Z	4
ρ (g cm^{-3})	1.360
μ (mm^{-1})	0.476
Reflections collected	18,056
Reflections unique	8773
Reflections ($I \geq 2\sigma(I)$)	6451
Parameters	614
$R1$, $wR2$ ($I \geq 2\sigma(I)$)	0.0434, 0.0926
$R1$, $wR2$ (all data)	0.0688, 0.1046
GOF (F^2)	1.032
Largest diff. peak and hole (e Å^{-3})	0.594 and −0.356

Table 2
Elemental analysis, electronic spectroscopic[a] and effective magnetic moments.[b]

Complex	Found (calc) (%)			λ_{max} (nm) ($10^{-4} \times \varepsilon$ (M^{-1} cm^{-1}))	μ_{eff} (μ_B)
	C	H	N		
1	71.38 (71.51)	4.51 (4.40)	2.71 (2.78)	535[c] (0.51), 501 (0.57), 447 (1.47), 421 (1.22), 369 (1.77), 276 (2.78)	1.99
2	71.56 (71.71)	4.61 (4.54)	2.65 (2.74)	536[c] (0.50), 500 (0.56), 446 (1.44), 420 (1.19), 369 (1.73), 313[c] (1.65), 279 (2.32)	1.98
3	70.81 (70.61)	4.41 (4.47)	2.65 (2.70)	537[c] (0.47), 497 (0.57), 445 (1.37), 419 (1.15), 370 (1.65), 312[c] (1.65), 279 (2.37)	1.90
4	68.95 (69.15)	4.23 (4.16)	2.61 (2.69)	537[c] (0.50), 500 (0.58), 449 (1.44), 423 (1.21), 370 (1.68), 312[c] (1.66), 279 (2.18)	1.93
5	68.57 (68.45)	4.18 (4.12)	3.86 (3.99)	555[c] (0.51), 518 (0.55), 452 (1.05), 409[c] (1.15), 371 (1.41), 280 (2.30)	1.97

[a] In dichloromethane.
[b] In powder phase at 298 K.
[c] Shoulder.

conditions produce red coloured complexes of formula *trans*-[Ru(pnbh*R*)(PPh_3)$_2$Cl] (**1**–**5**) in 56–64% yield. The elemental analysis data (Table 2) of the complexes are in good agreement with the above general molecular formula. All of **1**–**5** are one-electron paramagnetic. The room temperature (298 K) magnetic moments are within 1.90–1.99 μ_B. These values suggest the low-spin trivalent oxidation state of ruthenium in each of **1**–**5**. In all probability the oxygen in air oxidizes the metal centre during the synthesis of the complexes from [Ru(PPh_3)$_3$$Cl_2$]. All the complexes are highly soluble in dichloromethane, chloroform, dimethylsulfoxide and dimethylformamide and provide red solutions. In solution, they behave as non-electrolyte.

3.2. Spectroscopic properties

Infrared spectra of the Schiff bases and the complexes (**1**–**5**) were recorded using KBr pellets. The Schiff bases display the N–H and C=O stretches of the amide functionality in the ranges 3222–3162 and 1663–1633 cm^{-1}, respectively. A medium to strong band observed within 1605–1592 cm^{-1} is assigned to the C=N stretching vibration of the Schiff bases. Complexes **1**–**5** do not display any band associated with the amide N–H and C=O groups. Thus the amide functionality of the tridentate ligand (pnbhR^{2-}) is deprotonated in the complexes. A medium intensity band observed in the range 1582–1574 cm^{-1} for **1**–**5** is attributed to the metal coordinated C=N stretching vibration of pnbhR^{2-}. Each of **1**–**5** shows three strong bands at ~743, ~693 and ~17 cm^{-1} typical of metal bound PPh_3 [20–22,25–27].

Electronic spectral profiles of **1**–**5** obtained using the corresponding dichloromethane solutions are very similar. The spectral data are listed in Table 2. A representative spectrum is shown in Fig. 1. The complexes display several strong to very strong absorptions in the wavelength range 555–276 nm. The electronic spectra of all the Schiff bases (H_2pnbh*R*) in dichloromethane were also recorded for comparison. The spectra of H_2pnbh*R* are also very similar and display two groups of intense absorptions centred at ~370 and ~285 nm (Table S1). The spectrum of H_2pnbh*H* is shown in Fig. 1. Below 400 nm the spectral profiles of the complexes are somewhat similar to those of the Schiff bases. Thus the absorptions observed above 400 nm for **1**–**5** are assigned to ligand-to-metal charge transfer transitions and the absorptions observed below 400 nm are primarily due to ligand centred transitions [20–22,25–27].

The one-electron paramagnetic complexes are EPR active. All the complexes in dichloromethane–toluene (1:1) solution display an isotropic spectrum with $g \approx 2.12$ at room temperature (298 K). However, the spectra of **1**–**5** in frozen (130 K) solution show three distinct EPR signals. The spectrum of **3** is illustrated in Fig. 2 and the *g*-values for all five complexes are listed in Table 3. Such EPR spectra are typical of rhombically distorted octahedral low-spin ruthenium(III) species [11,14,18,20–23,26,27]. Considering the type of ligands in each of these quaternary complexes (**1**–**5**) and hence the stereochemistry of the molecule, rhombic distortion of the coordination sphere around the metal centre from ideal octahedral geometry is quite anticipated. The molecular structure of *trans*-[Ru(pnbh*Me*)(PPh_3)$_2$Cl] (**2**) determined by X-ray crystallography (*vide infra*) corroborates the observed EPR characteristics.

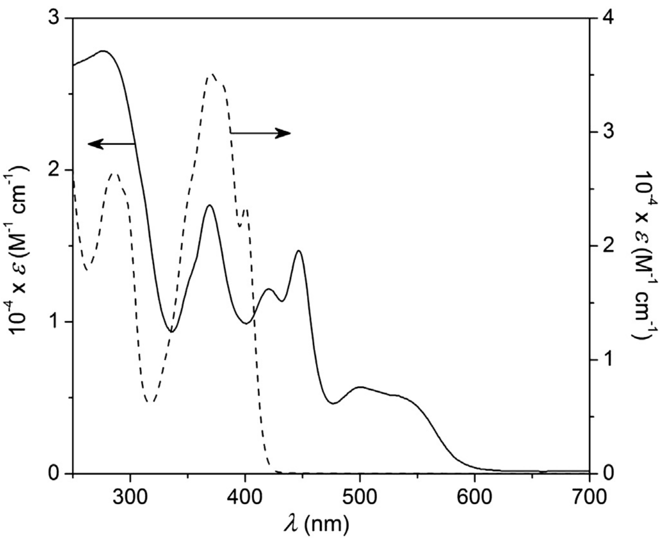

Fig. 1. Electronic spectra of H_2pnbh*H* (---) and *trans*-[Ru(pnbh*H*)(PPh_3)$_2$Cl] (**1**) (—) in dichloromethane.

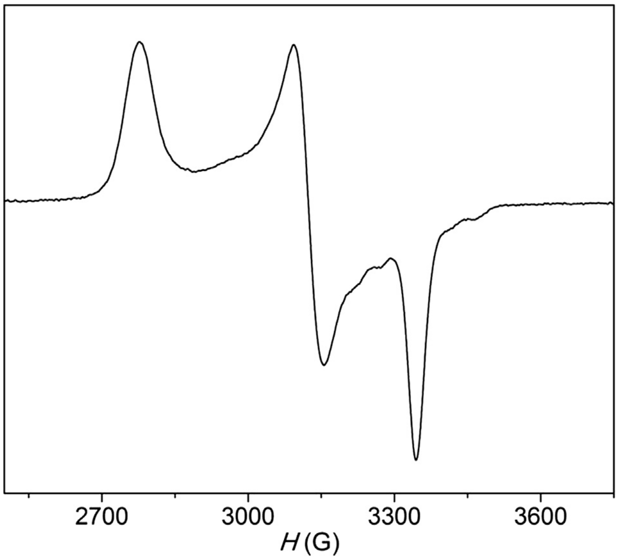

Fig. 2. X-band EPR spectrum of *trans*-[Ru(pnbh*OMe*)(PPh_3)$_2$Cl] (**3**) in frozen (130 K) dichloromethane–toluene (1:1) solution.

Table 3
EPR[a] and cyclic voltammetric[b] data.

Complex	g_1	g_2	g_3	$E_{1/2}$ (V) (ΔE_p (mV))[c]
1	2.36	2.09	1.95	−0.28 (90)
2	2.36	2.09	1.96	−0.29 (100)
3	2.35	2.09	1.96	−0.31 (90)
4	2.36	2.10	1.95	−0.27 (90)
5	2.36	2.09	1.95	−0.24 (130)

[a] In frozen (130 K) dichloromethane–toluene (1:1).

[b] In dimethylformamide.

[c] $E_{1/2} = (E_{pa} + E_{pc})/2$ and $\Delta E_p = E_{pa} - E_{pc}$, where E_{pa} and E_{pc} are anodic and cathodic peak potentials, respectively.

Table 4
Selected bond lengths (Å) and angles (°) for *trans*-[Ru(pnbh*Me*)(PPh$_3$)$_2$Cl] (**2**).

Ru–C(1)	2.048(3)	Ru–N(1)	2.025(3)
Ru–O	2.132(2)	Ru–Cl	2.3651(8)
Ru–P(1)	2.4030(9)	Ru–P(2)	2.3941(9)
C(1)–Ru–N(1)	77.38(11)	C(1)–Ru–O	150.65(11)
C(1)–Ru–Cl	106.58(9)	C(1)–Ru–P(1)	88.89(9)
C(1)–Ru–P(2)	92.39(9)	N(1)–Ru–O	74.34(9)
N(1)–Ru–Cl	173.84(8)	N(1)–Ru–P(1)	91.30(7)
N(1)–Ru–P(2)	87.49(7)	O–Ru–Cl	102.27(6)
O–Ru–P(1)	84.10(6)	O–Ru–P(2)	94.04(6)
Cl–Ru–P(1)	93.47(3)	Cl–Ru–P(2)	87.63(3)
P(1)–Ru–P(2)	178.01(3)		

3.3. X-ray structure of *trans*-[Ru(pnbh*Me*)(PPh$_3$)$_2$Cl] (**2**)

The X-ray molecular structure of *trans*-[Ru(pnbh*Me*)(PPh$_3$)$_2$Cl] (**2**) is depicted in Fig. 3. All the bond lengths and angles involving the metal centre and the coordinating atoms are listed in Table 4. These bond parameters indicate a distorted octahedral CNOP$_2$Cl coordination sphere around the ruthenium atom. The meridionally spanning pnbh*Me*$^{2-}$ coordinates the metal centre through the 1-pyrenyl *ortho*-C, the azomethine-N and the amidate-O atoms and forms 5,5-fused chelate rings. The chlorine atom completes a CNOCl square-plane around the metal centre and the two PPh$_3$ molecules occupy the two axial positions. Both bite angles (77.38(11)° and 74.34(9)°) in the 5,5-fused chelate rings of **2** are comparable with those reported for trivalent ruthenium complexes containing similar fused chelate rings formed by C,N,O-donor pincer like ligands [11,22,26,27]. The remaining *cis* angles are in the range 84.10(6)°–106.58(9)°. The P(1)–Ru–P(2) angle (178.01(3)°) formed by the two mutually *trans* PPh$_3$ molecules is very close to the ideal value, while *trans* N(1)–Ru–Cl angle (173.84(8)°) is somewhat shorter than that. In contrast, due to steric requirement the *trans* C(1)–Ru–O angle (150.65(11)°) formed by the two ends of the C,N,O-donor pnbh*Me*$^{2-}$ is significantly shorter than 180°. The two very similar *trans* oriented Ru–P bond lengths are somewhat longer than the Ru–Cl bond length but significantly longer than the bond lengths between the metal centre and the coordinating atoms from pnbh*Me*$^{2-}$ (Table 4). On the whole, the Ru to the aryl-C, the azomethine-N, the amidate-O and the Cl-atom bond lengths and the two Ru–P bond lengths are within the ranges observed for ruthenium(III) complexes having similar coordinating atoms [11,22,23,26,27].

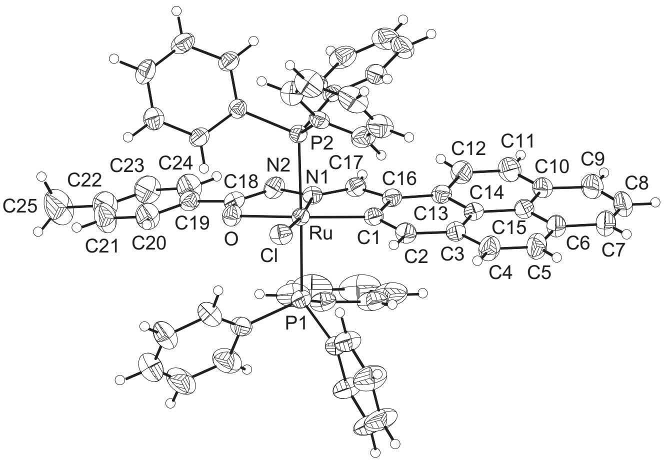

Fig. 3. X-ray molecular structure of *trans*-[Ru(pnbh*Me*)(PPh$_3$)$_2$Cl] (**2**) with the atom labelling schemes. Carbon atoms of the PPh$_3$ molecules are not labelled for clarity. All non-hydrogen atoms are represented by their 40% probability thermal ellipsoids.

3.4. Electron transfer properties

Electron transfer behaviour of **1**–**5** was studied using cyclic voltammetry. All the complexes in dimethylformamide display a reduction response on the cathodic side of Ag/AgCl reference electrode. The $E_{1/2}$ values are within −0.24 to −0.31 V and the ΔE_p values span the range 90–130 mV (Table 3). The cyclic voltammogram of **4** is illustrated in Fig. 4. The one electron nature of this reduction response has been inferred by comparing the current heights with known one electron transfer processes under identical condition [26,27]. The trend in the $E_{1/2}$ values of this reduction reflects the effect of the electronic nature of the substituent (*R*) on the aroyl fragment of pnbh*R*$^{2-}$. There is a satisfactory linear relationship between the $E_{1/2}$ values and the Hammett constants [44] of the substituents (Fig. 4). The free Schiff bases do not display any such response in this potential range under identical condition. Thus the electron transfer process observed for **1**–**5** is assigned to the ruthenium(III)–ruthenium(II) reduction couple. Our previously reported cyclometallated complexes of *trans*-{Ru(PPh$_3$)$_2$Cl}$^{2+}$ with aroylhydrazones of benzaldehyde display only a metal centred oxidation couple within 0.35–0.58 V [26], while analogous complexes with aroylhydrazones of acetophenone display both reduction (−0.66 to −0.70 V) and oxidation (0.75–0.80 V) of the metal centre [27]. In these complexes,

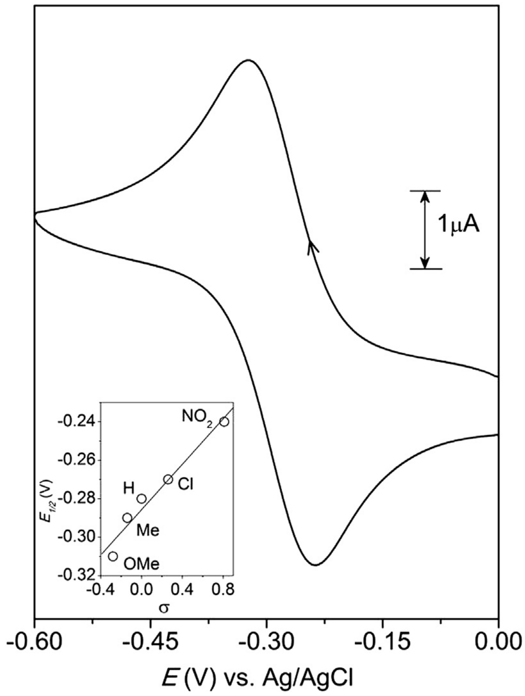

Fig. 4. Cyclic voltammogram of *trans*-[Ru(pnbh*Cl*)(PPh$_3$)$_2$Cl] (**4**) in dimethylformamide (0.1 M TBAP) 298 K. Inset: linear correlation between the $E_{1/2}$ and the Hammett substituent constant (σ).

the tridentate ligand is the phenyl *ortho*-C, the azomethine-N and the amidate-O donor, while pnbhR^{2-} uses the *ortho*-C of 1-pyrenyl together with the azomethine-N and the amidate-O to bind the metal centre in **1**–**5**. Thus it appears that the change in the aromatic part of the *ortho*-metallated fragment is primarily responsible for the observed differences in the electron transfer behaviours of these three very similar series of complexes. Perhaps the aromatic part plays a major role in tuning the distortion of the coordination geometry from octahedral symmetry and hence the electronic energy levels of the metal centre [27].

4. Conclusion

A series of cycloruthenated complexes, *trans*-[Ru(pnbh*R*)$(PPh_3)_2Cl$] (H_2pnbh*R* = 1-pyrenaldehyde 4-*R*-benzoylhydrazone (*R* = H, Me, OMe, Cl and NO_2)) is reported. The magnetic moments and the EPR spectra are consistent for trivalent state of the metal ion in these complexes. X-ray structure of *trans*-[Ru(pnbh*Me*)$(PPh_3)_2Cl$] reveals an unsymmetrical pincer like coordination mode of pnbhMe^{2-} through the 1-pyrenyl *ortho*-C, the azomethine-N and the amidate-O. Very similar spectroscopic and electron transfer properties suggest the same coordination mode of pnbhR^{2-} in all the complexes. Thus regioselective *ortho*-metallation over *peri*-metallation of the 1-pyrenyl fragment of pnbhR^{2-} and hence formation of 5,5- instead of 6,5-fused chelate rings at the trivalent ruthenium is preferred in the present series of cycloruthenates.

Acknowledgements

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Appendix A. Supplementary material

CCDC 924431 (for *trans*-[Ru(pnbh*Me*)$(PPh_3)_2Cl$] (**2**)) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary data (Tables S1 and S2) associated with this article can be found in the online version, at http://dx.doi.org/10.1016/j.jorganchem.2013.03.040.

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