

Synthesis and substitution reactions on dichloro[2-(N-(2-hydroxyphenyl)-carbamoyl)pyridine]bis(dimethylsulphoxide)-ruthenium(II)

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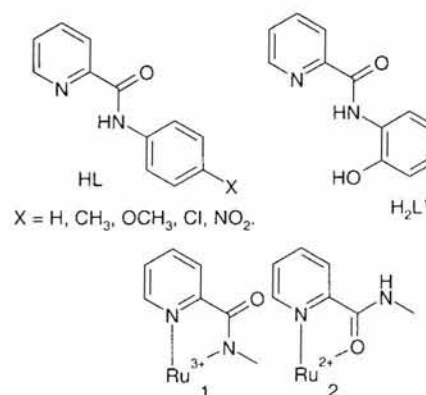
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The amide ligand 2-(N-(2-hydroxyphenyl)carbamoyl)pyridine (H_2L^1) has been prepared by condensing pyridine-2-carboxylic acid with 2-aminophenol. This amide ligand has been characterised by elemental analysis, infrared, ^1H NMR and mass spectral studies. The complex $[Ru(H_2L^1)(dms)_2Cl_2]$ **I**, has been synthesised by the reaction of $[Ru(dms)_4Cl_2]$ with H_2L^1 . Further reaction of **I** with π -acidic diimines, 2,2'-bipyridine and 1,10-phenanthroline, lead to the substitution of the coordinated dms molecules in **I** forming heteroleptic ruthenium complexes $[Ru(bpy)(HL^1)Cl_2]$, **II** and $[Ru(phen)(HL^1)Cl_2]$, **III** (bpy = 2,2'-bipyridine and $phen$ = 1,10-phenanthroline), respectively. The complex **I** is diamagnetic while the complexes **II** and **III** are paramagnetic due to one electron and show rhombic EPR spectra at 77K. All the complexes show intense absorptions due to metal to ligand charge transfer (MLCT) transitions in the electronic spectra. The IR and ^1H NMR spectral studies indicate that in **I**, the amide ligands coordinate to the ruthenium (II) ion as a bidentate ligand, coordinating from pyridyl nitrogen and the carbonyl oxygen of the amide linkage, leaving the other potential coordinating phenolic functional group intact. The IR spectral studies indicate that in **II** and **III**, the amide ligand coordinates from the pyridyl nitrogen and the amidato nitrogen, leaving the other potential coordinating phenolic functional group intact. The complex **I** undergoes a reversible ruthenium(II)/ruthenium(III) oxidation at -0.17 V in acetonitrile solution, while the complexes **II** and **III** undergo irreversible ruthenium(III)/ruthenium(IV) oxidation at *ca.* 0.37 V.

It was shown in our earlier reports¹ that the ability of the amide linkage of the 2-carbamoylpyridyl moiety to coordinate through the oxygen atom or through nitrogen atom leads to the formation of the linkage isomeric chelate rings 1 and 2. For the chelate ring 1, the coordinating atoms are pyridyl nitrogen and amidato nitrogen, while for the chelate ring 2, the coordinating atoms are pyridyl nitrogen and the carbonyl oxygen atom of the amide linkage. However, the type of coordination from the amide linkage depends upon the nature of the coligands bound to the metal centre. The substitution of the coordinated dms molecules of the complexes $[Ru(HL)(dms)_2Cl_2]$ ^{1a} by 2,2'-bipyridine leads to the change of the coordination site from amide oxygen to amidato nitrogen in the complexes $[Ru(L)(bpy)Cl_2]$, together with a change in the oxidation state from (+2) to (+3)^{1d}.

To explore the effect of potential coordinating group at the adjacent position on the phenyl ring in N-phenyl-2-carbamoylpyridyl moiety, a potentially tridentate amide ligand, H_2L^1 has been synthesised by the condensation of pyridine-2-carboxylic acid and 2-aminophenol. Reaction of H_2L^1 with $[Ru(dms)_4Cl_2]$ yielded the complex $[Ru(dms)_2(H_2L^1)Cl_2]$ and further substitution of the coordinated dms molecules in this complex by π -acidic diimine ligands, bpy and



phen, yielded heteroleptic ruthenium complexes $[Ru(bpy)(HL^1)Cl_2]$ and $[Ru(phen)(HL^1)Cl_2]$. Characterisation and cyclic-voltammetric properties of the complexes are reported in this paper.

Materials and Methods

Micro-analyses (C, H, N) were performed using a Perkin-Elmer 240C elemental analyser. IR spectra were obtained on a Perkin-Elmer 783 spectrophotometer. Electronic spectra were recorded on a Shimadzu 240-UV-visible spectrophotometer. Magnetic susceptibilities were measured with the help of a PAR 155 vibrating sample magnetometer. The ^1H NMR spectra were obtained with a Varian Fourier-

transform spectrometer, using TMS as an internal standard. The electrospray mass spectra were recorded on a MICROMASS QUATTRO II triple quadrupole mass spectrometer (assignments are based on the ^{102}Ru isotope). Electrochemical measurements were made using the 174A polarographic analyser, a universal programmer, an X-Y recorder, a platinum working electrode, a platinum wire auxiliary electrode and Ag/AgNO₃ reference electrode. All electrochemical measurements were performed under dinitrogen atmosphere. Ferrocene was used as an internal standard; all the potentials being quoted *vs.* the ferrocene-ferrocenium couple. Supporting electrolyte used was Tetraethylammonium perchlorate (TEAP). The EPR measurements were made using a Varian model 109C E-line X-band spectrometer filled with a quartz dewar for measurements at 77 K (liquid nitrogen). The spectra were calibrated using tetracyanoethylene ($g = 2.0023$). RuCl₃· x H₂O (Loba, India) was converted to RuCl₃·3H₂O by repeated evaporation to dryness with concentrated hydrochloric acid. Pyridine-2-carboxylic acid (E-merk, Germany), 2-aminophenol, 2,2'-bipyridine and 1,10-phenanthroline (Loba, India) were used as received. Acetonitrile was distilled over CaH₂ before performing electrochemical experiments. Tetraethylammonium perchlorate (TEAP) was prepared using a reported procedure². Methanol was dried over fused CaO and [Ru(dmsO)₄Cl₂] (dmsO = dimethylsulfoxide) was prepared, as reported before³.

Synthesis of the ligand

2-(*N*-(2-hydroxyphenyl)carbamoyl)pyridine (H₂L¹)

In to a solution of pyridine-2-carboxylic acid (3.08 g., 25 mmol) in 10 mL pyridine, 2-aminophenol (2.73 g., 25 mmol) was added and was warmed under stirring condition for 15 min. Into the resulting solution, 6.6 mL (25 mmol) triphenylphosphite was added and the mixture was stirred at 110°C for 4h. The cold reaction mixture was washed with 100 mL distilled water and the resulting white paste was taken in 40 mL dichloromethane and extracted with 150 mL 1:1 (v/v) aq. hydrochloric acid. The acidic aq. extract was neutralised by saturated aq. sodium bicarbonate solution. The resulting white solid was filtered, washed thoroughly with distilled water and crystallised from aq. methanol as a colourless crystalline solid. Yield 2.84 g. (53%), mp 208°C. [Found C, 66.98; H, 4.35; N, 12.93; Calc. for C₁₂H₁₀N₂O₂, C, 67.29; H, 4.67; N, 13.08 %].

Syntheses of the complexes

Dichloro [2-(*N*-(2-hydroxyphenyl)carbamoyl)pyridine]bis(dimethylsulfoxide)ruthenium-(II), [Ru(H₂L¹)(dmsO)₂Cl₂] **I**: A suspension of [Ru(dmsO)₄Cl₂] (0.484 g., 1 mmol) and 2-(*N*-(2-hydroxyphenyl)carbamoyl)pyridine (H₂L¹) (0.214 g., 1 mmol) in 10 mL dry methanol was refluxed for 1h and 8 mL solvent was distilled out from the reaction mixture. On cooling, an orange coloured microcrystalline product separated. The solid was isolated by filtration, washed with minimum volume of dry methanol and then by (10 × 3) mL diethyl ether, and dried over fused calcium chloride, yield 0.326 g. (60 %); [Found C, 35.16; H, 4.02; N, 5.27; Calc. for RuC₁₆H₂₂N₂O₄Cl₂S₂; C, 36.42; H, 4.06; N, 5.17 %].

Dichloro (2,2'-bipyridine) [2-(*N*-(2-hydroxyphenyl)carbamoyl)pyridine]ruthenium(III), [Ru(bpy)(HL¹)Cl₂] **II**: A solid mixture of **I** 0.15 g (0.28 mmol) and 2,2'-bipyridine 43.2 mg. (0.28 mmol) was refluxed in dimethylformamide (6 mL) for 4h. The solvent was evaporated from the deep red coloured reaction mixture. The resulting solid was dissolved in a minimum volume of dichloromethane and was purified by column chromatography using silica gel (60-120 mesh). The desired product was isolated by eluting the column by a 4:1 v/v benzene-acetonitrile mixture. Evaporation of the solvent from the eluate yielded a dark-red coloured crystalline solid. Yield 0.065 g (43%). [Found C, 48.37; H, 3.05; N, 10.13; Calc. for RuC₂₂H₁₇N₄O₂Cl₂; C, 48.79; H, 3.14; N, 10.35 %].

Dichloro (1, 10-phenanthroline)[2-(*N*-(2-hydroxyphenyl)carbamoyl)-pyridine]ruthenium(III), [Ru(phen)(HL¹)Cl₂] **III**: The complex was synthesised by a method similar to that described for **II**. Yield 0.072 g (46 %). [Found C, 50.73; H, 3.17; N, 9.67; Calc. for RuC₂₄H₁₇N₄O₂Cl₂; C, 50.97; H, 3.01; N, 9.91 %].

Result and Discussion

The synthetic methodology for the ligands has been adapted from Barnes *et al.*⁴ with a modification in the isolation step. The reaction between pyridine-2-carboxylic acid and 2-aminophenol in presence of triphenylphosphite proceeds smoothly in pyridine medium, resulting in a brown oil. The product was isolated by acidifying the reaction mixture by 1:1 (v/v) aqueous hydrochloric acid, followed by neutralisation by saturated aq. sodium bicarbonate solution and finally was crystallised from aqueous methanol.

Microanalytical data (Experimental Section) agree well with the empirical formula of H_2L^1 . In the FAB mass spectrum the maximum peak is observed at m/z 214, which corresponds to the molecular ion (calculated molecular weight 214). The ^1H NMR spectrum was recorded in $dms\text{-}d_6$ solvent. Six resonances in the aromatic region of ^1H NMR spectrum of H_2L^1 are found to integrate to eight protons; three doublets due to three protons, two triplets due to two protons and a multiplet due to three protons. Two singlets, which disappear on D_2O exchange, at δ 10.55 and 9.73, have been assigned to NH proton of the amide linkage and the phenolic proton of the phenyl ring, respectively. In the IR spectrum of H_2L^1 , the bands due to the NH stretching and CO stretching appear at 2997 and 1651 cm^{-1} , respectively. The band due to the OH stretching of the phenolic hydroxyl group appears at 3308 cm^{-1} .

The complex $[Ru(H_2L^1)(dms\text{-}O)_2Cl_2]$ **I** has been synthesised by substituting two coordinated dmsO molecules from the starting complex $[Ru(dms\text{-}O)_4Cl_2]$ by the amide ligand, H_2L^1 . The reaction of $[Ru(dms\text{-}O)_4Cl_2]$ with an equimolar quantity of H_2L^1 proceeds smoothly, in dry methanol, to precipitate the microcrystalline complex, **I** in good yield. Further reactions of **I** with π -acidic chelating diimine ligands, bpy and phen in refluxing dimethylformamide lead to the substitution of the coordinated dmsO molecules forming the substituted complexes **II** and **III**. The complexes were isolated from the reaction mixture by column chromatography.

The elemental analytical data correspond to the expected composition of these complexes. Magnetic susceptibility measurements show that complex **I** is diamagnetic, as expected for the complexes of ruthenium(II) (low spin d^6 , $S = 0$), while the complexes **II** and **III** are paramagnetic, due to one unpaired electron, as expected for the complexes of ruthenium(III) (low spin d^5 , $S = 1/2$).

The infrared spectrum of **I** contains many sharp bands of different intensities due to the vibrations arising from the coordinated dmsO, H_2L^1 and Cl^- ligands, and are, therefore, complex in nature. No attempt has been made to assign the individual bands. However, comparison of the IR spectrum of **I** with that of the free ligand shows a negative shift in ν_{CO} (1651 cm^{-1} in H_2L^1 and 1627 cm^{-1} in **I**) of the amide linkage in the complex, indicating⁵ that oxygen atom of the amide linkage is coordinated to the ruthenium(II) centre. The sharp band at 1082 cm^{-1} has been assigned to ν_{SO} . The same band in free dmsO appears

at 1055 cm^{-1} .⁶ The positive shift in ν_{SO} indicates that sulphur atom of the dmsO molecule coordinates to the ruthenium(II) centre⁶. The bands at 3449 and 3397 cm^{-1} have been assigned to the stretching vibration of NH bond of the amide linkage and OH bond of the phenolic hydroxyl group of H_2L^1 , respectively. The appearance of NH stretching band, in the IR spectrum of the complex **I**, indicate that ruthenium(II) centre is not coordinated by the amidato nitrogen of H_2L^1 and indirectly supports that the coordination is from the oxygen atom of the amide linkage of H_2L^1 . The sharp bands at 307 cm^{-1} (medium intensity) and 270 cm^{-1} are assigned to ν_{Ru-Cl} ⁷ and $\nu_{Ru-N(pyridine)}$ ⁸ stretching modes. Thus in complex **I**, the amide ligand H_2L^1 coordinates from the pyridyl nitrogen and oxygen atom of the amide linkage, forming the chelate ring 2.

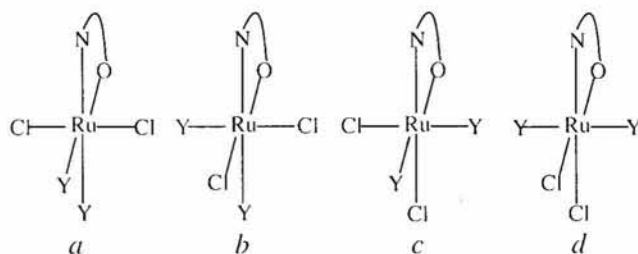
Infrared spectra of **II** and **III** are very similar; each spectrum shows many vibrations of different intensity below 1650 cm^{-1} due to the presence of coordinated amide ligand and the chelating diimines. No attempt has been made for the individual band assignment. The ν_{CO} of the coordinated amide ligand is found to appear at *ca.* 1631 cm^{-1} . A broad band, centred at 3344 cm^{-1} in case of **II** and at 3340 cm^{-1} in case of **III**, in the IR spectra of the corresponding complexes, has been assigned to the ν_{OH} of H_2L^1 . The appearance of ν_{OH} indicates the presence of noncoordinated phenolic moiety of H_2L^1 , while the disappearance of ν_{NH} band indicates the amidato nitrogen atom coordinating to the ruthenium(III) centre in these substituted complexes. Thus in **II** and **III**, H_2L^1 coordinates to ruthenium(III) centre from the amidato nitrogen and pyridyl nitrogen atoms, forming the chelate ring 1. This may be because the π -acidic bpy ligand reduces the electron density at the ruthenium centres, forcing the amide ligand to coordinate from the σ -donor amidato nitrogen atom.

The ^1H NMR spectrum of **I** was recorded in $dms\text{-}d_6$ solvent to confirm the ratio of one bidentate N,O-donor ligand to two coordinated dmsO ligands. In the ^1H NMR spectrum of **I** all the signals due to the ligand protons are retained but are found to be shifted from their positions compared to the same in the free ligand. Two singlets, which disappear on D_2O exchange, at δ 11.57 and 10.31 have been assigned to NH proton of the amide linkage and the phenolic hydroxyl group of H_2L^1 in the complex **I**. Two singlets at δ 3.38 and 3.17, each of which integrates to six protons, have been assigned to the two coordinated dmsO molecules. The appearance of two different sig-

nals for the two coordinated dmso molecules indicate that they are magnetically nonequivalent. Seven resonances in the aromatic region of the ^1H NMR spectrum of **I** are found to integrate to eight protons; three doublets due to three protons, three triplets due to three protons and a multiplet due to two protons.

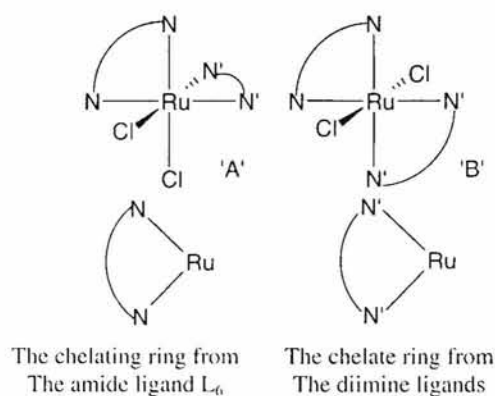
Geometry around ruthenium centre:

The ruthenium(II) center in **I** may, in principle, exist in the following four geometrical isomeric forms:



In the IR spectra, a single $\nu_{\text{Ru-Cl}}$ band is expected for a linear grouping of the *trans*- RuCl_2 moiety (as in *a*), whereas, two $\nu_{\text{Ru-Cl}}$ bands are expected for a *cis*- RuCl_2 moiety (as in *b*, *c* and *d*)⁹. Appearance of a single $\nu_{\text{Ru-Cl}}$ band excludes the isomers *b*, *c* and *d*, hence the arrangement of the donor atoms around the ruthenium(II) centre is as depicted in *a* ($\text{Y} = \text{dmso}$). This is further supported by the appearance of two different signals for the methyl groups of the coordinated dmso molecules in the ^1H NMR spectrum of **I**, indicating that they are magnetically non-equivalent.

The complexes **II** and **III** may, in principle, exist in the following two geometrical isomeric forms 'A' and 'B'.



The structure of an analogous bis-chelated complex, dichloro(2,2'-bipyridine)[2-(N-(4-nitrophenyl)carbamoyl)pyridine]ruthenium(III) involving the amide ligand 2-(N-(4-nitrophenyl)carbamoyl)pyridine and the chelating diimine bpy, has been determined

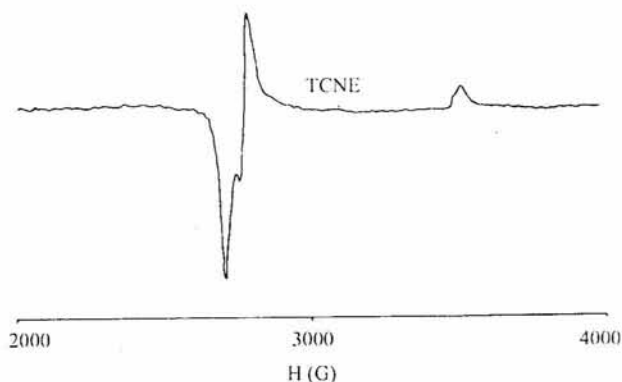


Fig. 1 — EPR spectrum of **II** in 1:1 (v/v) dichloromethane/toluene solution at 77 K

by X-ray crystallographic analysis^(1d). The geometry around the ruthenium(III) centre in this complex is as described in 'A' of the above figure. By analogy with the structure of this complex the geometry around the ruthenium(III) centre in **II** and **III** is assumed to be as described in 'A'.

The EPR spectra of complexes **II** and **III** were recorded in 1:1 (v/v) dichloromethane-toluene solution at 77 K. A representative spectrum is shown in Fig. 1. Each complex shows rhombic EPR spectrum with three distinct g values (Table 1). However, both the spectra may be regarded as pseudo-axial, consisting of two very closely spaced signals (g_1 and g_2 , rhombic component of g -perpendicular) and a rather isolated signal (g_3 , g -parallel in the axial case). Thus the axial distortion, that splits the t_2 level into 'a' and 'e' components, is expected to be much larger than the rhombic distortion, which splits 'e'⁽¹⁰⁾.

The complex **I** is insoluble in chloroform, dichloromethane and methanol but is partially soluble in acetonitrile and is totally soluble in DMSO. The complex is soluble in 4:1 (v/v) dichloromethane-methanol, dichloromethane-acetonitrile, chloroform-methanol and chloroform-acetonitrile mixtures. The electronic spectrum of this complex has been recorded in mixed solvent, dichloromethane-methanol 4:1 (v/v) mixture and the spectral data are collected in Table 1. The complex shows two intense bands at 318 and 436 nm. On the basis of the values of the extinction coefficients, these bands are assigned to the metal to ligand charge transfer (MLCT) transitions.

The complexes **II** and **III** are readily soluble in polar organic solvents such as methanol, ethanol, acetonitrile, dichloromethane and chloroform, but are insoluble in non-polar solvents, such as benzene, toluene and *n*-hexane. The electronic spectra of these

Table I—Characterisation data of the complexes

Complex	Electronic spectra ^a λ_{max} , nm ($\epsilon \cdot \text{M}^{-1} \text{cm}^{-1}$)	Magnetic moment $\mu(\text{BM})$	Cyclic voltammetric data ^b E_{298}^0 , V (ΔE_p , mV)		g values ^d		
			Metal centred	Ligand centred	g_1	g_2	g_3
I	318(7480.2), 436(2187.5)	—	—0.17(60)	—	—	—	—
II	295(12016.6), 375(3482.1), 447(1502.1), 595(409.7)	1.77	+0.38 ^c	−1.26(60)	1.68	1.72	2.17
III	291(20240.7), 377(9755.2), 457(3464.2), 593(912.2)	1.78	+0.39 ^c	−1.29(60)	1.69	1.72	2.16

^ain 4:1 (v/v) dichloromethane-methanol solution for **I** and in dichloromethane solution for **II** and **III**.

^bSolvent, acetonitrile; supporting electrolyte, TEAP; scan rate 100 mVs^{-1} ; $E_{298}^0 = 0.5 (E_{\text{pa}} + E_{\text{pc}})$, where E_{pa} and E_{pc} are anodic and cathodic peak potentials respectively. $\Delta E = E_{\text{pa}} - E_{\text{pc}}$.

^c E_{pa} , the anodic peak potential.

^din 1:1 (v/v) dichloromethane-toluene solution, at 77 K.

complexes have been recorded in dichloromethane solution and the spectral data are collected in Table I. Four bands appear in the electronic spectra of these complexes. The bands at *ca.* 375, 450 and 595 nm have been assigned to the MLCT transitions, on the basis of high extinction coefficient values of these bands. Another intense band (at 295 nm for **II** and at 291 nm for **III**) appears in the UV region of the electronic spectra of these complexes. In the electronic spectra of free bpy and phen, an intense band appears at 290 and 289 nm, respectively. Thus the band at *ca.* 290 nm, in the electronic spectra of the complexes **II** and **III**, has been assigned to the intraligand transition in the coordinated π -acidic diimine ligands. An intense band is found to appear in the electronic spectrum of the free amide ligand H_2L^1 at 279 nm. This band due to the intraligand transition does not appear in the electronic spectrum of any of these three complexes.

The electrospray mass spectra of all the complexes were recorded. The peaks observed at m/z 543.1 for **I**, m/z 542 for **II** and at m/z 566 for **III**, correspond to the molecular ions of the corresponding complexes. (Calc. mol. Wt. : 542.57 for **I**, 541.47 for **II** and 565.49 for **III**.)

Thus the mass spectral data, along with micro-analytical, magnetic moment, ^1H NMR, ESR and IR spectral results, collectively establish the suggested

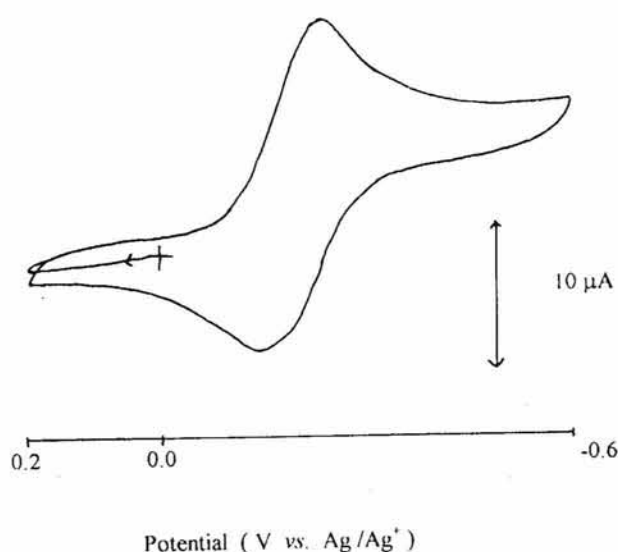
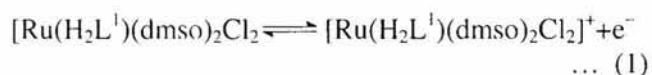


Fig. 2 — Cyclic voltammogram of **I** in acetonitrile solution (0.1 M TEAP) at a scan rate of 200 mV s^{-1}

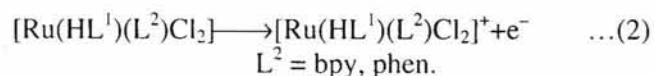
composition and the stereochemistry of the complexes.

The electrochemical properties of the complexes were studied in acetonitrile (0.1 M in TEAP) by cyclic voltammetric technique. All the potentials are quoted vs. the ferrocene-ferrocenium couple. The voltammetric data are presented in Table I and a representative voltammogram is shown in Fig. 2. The complex **I** shows a reversible response, due to ruthenium(II)/ruthenium(III) couple (Eq. 1) at -0.17 V .



The ΔE_p values of this couple is 60 mV, which does not change with change in the scan rate and the i_{pa}/i_{pc} (i_{pa} = anodic peak current and i_{pc} = cathodic peak current) ratio is close to 1.0, as expected for reversible couples. This potential of the ruthenium(II)/ruthenium(III) couple can be compared with that for the complexes $[\text{Ru}(\text{HL})(\text{dmsO})_2\text{Cl}_2]$, which bear *p*-substituted *N*-phenyl-2-carbamoylpyridine as the ligand (HL). For these complexes the potential of the ruthenium(II)/ruthenium(III) couple lies in the potential range 0.54 to 0.60 V vs the ferrocene–ferrocenium couple^{1a}. The presence of a phenolic hydroxyl group at the *o*-position of the phenyl ring of the ligand H_2L^1 , in the complex **I**, increases the electron density at the ruthenium(II) centre making it more prone to undergo oxidation. As a consequence, the ruthenium(II)/ruthenium(III) oxidation potential in the complex **I** is much lower compared to the same for the complexes $[\text{Ru}(\text{HL})(\text{dmsO})_2\text{Cl}_2]$.

In the cyclic voltammogram of the complexes **II** and **III**, an irreversible response appears at a potential *ca.* +0.38 V, due to the irreversible oxidation of the ruthenium(III) centres (Eq. 2)



The irreversibility of the ruthenium(III)/ruthenium(IV) couple for these complexes may be due to the instability of the oxidised species within the cyclic voltammetric time scale. In the cathodic side of the cyclic voltammograms of these complexes a reversible response appears at *ca.* -1.25 V. This reversible response has been attributed to the reduction of the coordinated diimine ligands (bpy, phen). It is well known^{12,13}, that bpy or phen can

successively accept two electrons in the lowest unoccupied molecular orbital. In the complexes **II** and **III** only the first reduction has been observed experimentally. The second reduction, which usually takes place at a more negative potential¹⁴, is not observed due to solvent cut off⁷.

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