

Spectral, redox and catalytic studies of triphenylphosphine/triphenylarsine complexes of Ru(III) with N, O donor ligands derived from 2-hydroxy-1-naphthaldehyde and primary amines

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Abstract

A series of new mixed ligand hexacoordinated ruthenium(III) Schiff base complexes of the type $[\text{RuX}_2(\text{EPh}_3)_2(\text{LL}')] (X = \text{Cl}, E = \text{P}; X = \text{Cl or Br}, E = \text{As and LL}' = \text{anion of the Schiff bases derived from the condensation of 2-hydroxy-1-naphthaldehyde with aniline, 4-chloroaniline, 2-methyl aniline and 4-methoxy aniline})$ are reported. All the complexes have been characterized by analytical and spectral (IR, electronic and EPR) data. The redox behavior of the complexes has also been studied. The complexes exhibit catalytic activity in the oxidation of benzyl alcohol to benzaldehyde in the presence of *N*-methyl morpholine-*N*-oxide (NMO). An octahedral structure has been proposed for all of the complexes. © 2005 Elsevier B.V. All rights reserved.

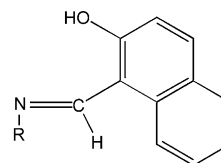
Keywords: Ruthenium(III); Spectroscopic characterization; Cyclic voltammetry; Catalytic oxidation

1. Introduction

The oxidation of primary and secondary alcohols into their corresponding aldehydes and ketones, respectively, plays a central role in organic synthesis [1]. Traditionally, such transformations have been performed with stoichiometric quantities of inorganic oxidants, such as chromium (VI) compounds [2]. The quest for effective catalytic systems that use clean, inexpensive primary oxidants, such as *N*-methyl morpholine-*N*-oxide, molecular oxygen or hydrogen peroxide, i.e. a ‘green method’ for converting alcohols to carbonyl compounds on an industrial scale remains an important challenge [3]. Ruthenium(III) complexes containing triphenylphosphine or triphenyl arsine have been extensively investigated as catalyst [4] for alcohol oxidations using a variety of primary oxidants, e.g. *N*-methyl morpholine-*N*-oxide [5], *tert*-butyl hydroperoxide [6], iodosobenzene [7], hypochlorite [8], bromate [9] or a combination of oxygen and an aldehyde [10].

The synthesis and characterization of several hexacoordinated ruthenium(III) complexes containing Schiff bases derived from salicylaldehyde and primary amines have been reported by our research group earlier [11]. As part of our systematic investi-

gations on the reactions of ruthenium(III) complexes with Schiff base ligands, we intended to study the effect of introducing an additional phenyl ring in the aldehyde part of the Schiff base in terms of catalytic activity and electrochemistry. Thus, the present paper deals with the synthesis and spectral characterization of some new ruthenium(III) Schiff base complexes obtained from the reactions of $[\text{RuCl}_3(\text{PPh}_3)_3]$, $[\text{RuCl}_3(\text{AsPh}_3)_3]$ and $[\text{RuBr}_3(\text{AsPh}_3)_3]$ with Schiff bases derived from 2-hydroxy-1-naphthaldehyde and primary amines such as aniline, 4-chloroaniline, 2-methylaniline and 4-methoxyaniline. The general structure of the Schiff base ligands used in this study is depicted below.



R	Abbreviation
C ₆ H ₅	HNA
4-ClC ₆ H ₄	HNCA
2-CH ₃ C ₆ H ₄	HNMA
4-OCH ₃ C ₆ H ₄	HNMOA

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2. Experimental

2.1. Reagents and materials

All the chemicals used are of analytical reagent grade or chemically pure grade. $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ purchased from Himedia was used without further purification. Solvents were purified following standard procedures. The starting complexes $[\text{RuCl}_3(\text{PPh}_3)_3]$ [12], $[\text{RuCl}_3(\text{AsPh}_3)_3]$ [13] and $[\text{RuBr}_3(\text{AsPh}_3)_3]$ [14] and the Schiff bases [15,16] were prepared according to literature procedures. The supporting electrolyte, *tertiary*-butyl ammonium perchlorate (TBAP) was dried in vacuum before use.

2.2. Physical measurements

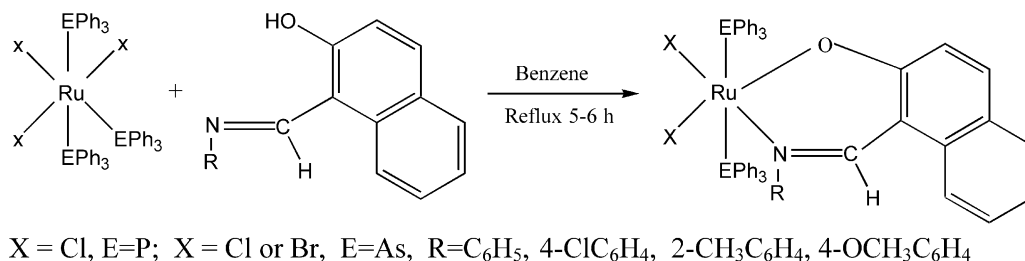
Infrared spectra were recorded as KBr discs on a Nicolet Avatar Model FT-IR spectrophotometer in the $4000\text{--}400\text{ cm}^{-1}$ range at room temperature. Electronic spectra of the complexes were recorded in CH_2Cl_2 solution with a Hitachi U-3210 spectrophotometer in the $800\text{--}200\text{ nm}$ range. Room temperature EPR spectra of powdered samples were recorded on a E-112 Varian model instrument in X-band frequencies using DPPH

2.3.2. Catalytic oxidation [17]

To a solution of benzyl alcohol (1 mmol) in CH_2Cl_2 (20 cm^3), *N*-methyl-morpholine-*N*-oxide (3 mmol) and ruthenium complex (0.01 mmol) were added. The solution was stirred at room temperature for 3 h. The mixture was evaporated to dryness and extracted with petroleum ether (bp $60\text{--}80^\circ\text{C}$) ($3 \times 10\text{ cm}^3$). The combined petroleum ether mixture was filtered and evaporated to give benzaldehyde, which was then quantified as its 2,4-dinitrophenyl hydrazone derivative.

3. Results and discussion

Reactions of the various Schiff bases with $[\text{RuX}_3(\text{EPh}_3)_3]$ ($\text{X}=\text{Cl}$, $\text{E}=\text{P}$; $\text{X}=\text{Cl}$ or Br , $\text{E}=\text{As}$) yielded complexes of the general formula $[\text{RuX}_2(\text{EPh}_3)_2(\text{LL}')]$ ($\text{X}=\text{Cl}$, $\text{E}=\text{P}$; $\text{X}=\text{Cl}$ or Br , $\text{E}=\text{As}$ and LL' = anion of the Schiff bases derived from the condensation of 2-hydroxy-1-naphthaldehyde with aniline, 4-chloroaniline, 2-methylaniline and 4-methoxyaniline). The complexes are green in colour, non-hygroscopic and are soluble in common organic solvents such as CH_2Cl_2 , DMSO and DMF. In all the reactions, it has been found that the Schiff bases behave as uninegative bidentate ligands replacing a triphenylphosphine/arsine and a halide ligand from the starting complexes.



as internal standard. Magnetic moments were measured on a EG & G-PARC vibrating sample magnetometer at 25°C . Cyclic voltammograms were recorded on a BAS CV-50 electrochemical analyzer in CH_3CN using a glassy-carbon working electrode and the potentials were referenced to a standard calomel electrode. All microanalyses (C, H and N) were performed using a Vario EL III Elementar analyzer. Melting points were recorded with a Raaga apparatus and are uncorrected.

2.3. Recommended procedures

2.3.1. Synthesis of new ruthenium(III) complexes

All the new complexes were prepared by the following general procedure. The Schiff base (0.020–0.028 g, 0.079–0.100 mmol) was added to a solution of $[\text{RuX}_3(\text{EPh}_3)_3]$ ($\text{X}=\text{Cl}$, $\text{E}=\text{P}$; $\text{X}=\text{Cl}$ or Br , $\text{E}=\text{As}$) (0.1 g, 0.079–0.100 mmol) in benzene (20 cm^3). The metal to ligand ratio was 1:1. The mixture was heated under reflux for 5–6 h. A green colour solution was obtained, which was concentrated to about 3 cm^3 . The complex was separated by the addition of 5 cm^3 of petroleum ether (bp $60\text{--}80^\circ\text{C}$). It was then recrystallised from CH_2Cl_2 /petroleum ether (1:5) and dried in vacuum (yield 60–75%).

The analytical data obtained for the new ruthenium(III) complexes are given in Table 1 and agree very well with the proposed molecular formula. Magnetic measurements show that all the complexes are paramagnetic and confirm that ruthenium is in +3 oxidation state.

3.1. Spectroscopic studies

The characteristic IR frequencies of the new ruthenium(III) complexes and their assignments are given in Table 2. In the IR spectra of the free Schiff bases, a band of medium intensity appears around $3400\text{--}3300\text{ cm}^{-1}$ due to $\nu(\text{O-H})$. This band disappears on complexation with the metal ion, which indicates the deprotonation of the ligands prior to coordination through oxygen atom [18]. A strong band around $1628\text{--}1618\text{ cm}^{-1}$ due to azomethine group of the ligands undergoes a shift to lower frequency after complexation indicating the coordination of azomethine nitrogen to ruthenium [19]. A high intensity band at $1303\text{--}1296\text{ cm}^{-1}$ in the free ligands may be assigned to the phenolic C–O stretching [20]. In the complexes, C–O stretching vibrations are shifted to higher frequency by $20\text{--}50\text{ cm}^{-1}$. This shift to higher frequency indicates the bonding of phenolic oxygen to ruthenium [21]. Further, a medium intensity band which appeared around $530\text{--}470\text{ cm}^{-1}$ assignable for $\nu(\text{Ru-O})$

Table 1
Analytical data and mp s of new Ru(III) complexes

	Complex ^a	mp (°C)	Calculated (found) %		
			C	H	N
(1)	[RuCl ₂ (PPh ₃) ₂ (NA)]	158	67.52 (67.50)	4.49 (4.59)	1.49 (1.41)
(2)	[RuCl ₂ (PPh ₃) ₂ (NCA)]	153	65.14 (65.21)	4.23 (4.19)	1.43 (1.54)
(3)	[RuCl ₂ (PPh ₃) ₂ (NMA)]	130	67.78 (67.72)	4.24 (4.21)	1.46 (1.43)
(4)	[RuCl ₂ (PPh ₃) ₂ (NMOA)]	159	66.67 (66.63)	4.56 (4.48)	1.44 (1.43)
(5)	[RuCl ₂ (AsPh ₃) ₂ (NA)]	173	61.76 (61.72)	4.11 (4.11)	1.36 (1.31)
(6)	[RuCl ₂ (AsPh ₃) ₂ (NCA)]	144	59.76 (59.62)	3.88 (3.64)	1.31 (1.22)
(7)	[RuCl ₂ (AsPh ₃) ₂ (NMA)]	142	62.08 (62.03)	4.25 (4.23)	1.34 (1.31)
(8)	[RuCl ₂ (AsPh ₃) ₂ (NMOA)]	147	61.14 (61.06)	4.18 (4.04)	1.32 (1.28)
(9)	[RuBr ₂ (AsPh ₃) ₂ (NA)]	135	56.86 (56.83)	3.78 (3.76)	1.25 (1.22)
(10)	[RuBr ₂ (AsPh ₃) ₂ (NCA)]	143	55.16 (54.43)	3.58 (4.08)	1.21 (1.63)
(11)	[RuBr ₂ (AsPh ₃) ₂ (NMA)]	141	57.21 (57.18)	3.91 (3.87)	1.24 (1.22)
(12)	[RuBr ₂ (AsPh ₃) ₂ (NMOA)]	125	56.42 (56.22)	3.86 (3.42)	1.22 (1.18)

^a Green.

Table 2
IR and electronic spectra of the Ru(III) complexes

Complex	$\nu(\text{C}=\text{N}), \nu(\text{C}-\text{O})$ (Phenolic) (cm^{-1})	PPh ₃ or AsPh ₃ bands (cm^{-1})	λ_{max} (nm)
(1)	1614, 1354	1426, 1092, 694	305, 386, 506
(2)	1604, 1320	1439, 1072, 692	300, 360, 506
(3)	1616, 1376	1434, 1092, 693	380, 530
(4)	1597, 1338	1437, 1094, 694	400, 530
(5)	1616, 1316	1435, 1094, 692	305, 340, 403, 495
(6)	1597, 1330	1435, 1095, 692	305, 344, 381, 485
(7)	1614, 1330	1435, 1072, 692	305, 338, 390, 488
(8)	1615, 1336	1435, 1095, 692	270, 305, 486
(9)	1614, 1335	1435, 1075, 691	306, 330, 440, 525
(10)	1623, 1336	1435, 1087, 692	302, 337, 440, 528
(11)	1614, 1335	1435, 1083, 691	302, 340, 416, 456, 524
(12)	1597, 1335	1436, 1085, 691	304, 344, 388, 480, 525

also supports oxygen coordination [22]. These facts prove the uninegative bidentate nature of the Schiff base ligands. The other characteristic bands due to triphenylphosphine or triphenylarsine (around 1440, 1100 and 700 cm^{-1}) are also present in the spectra of all the complexes [23].

The UV–vis spectra of all the complexes in CH_2Cl_2 show two to five bands in the region 270–530 nm and their assignments are given in Table 2. The ground state of ruthenium(III) (t^5_2g configuration) is $^2T_{2g}$, while the first excited doublet levels in the order of increasing energy are $^2A_{2g}$ and $^2T_{1g}$, which arise from $t^5_2g e_g^1$ configuration [24]. In most of the ruthenium(III) complexes, the electronic spectra show only charge transfer bands [11,25]. Since in a d^5 system, and especially in ruthenium(III) that has relatively high oxidising properties, the charge transfer bands of the type $L\pi_y \rightarrow t_{2g}$ are prominent in the low energy region obscuring the weaker bands due to d–d transition. Therefore, it becomes difficult to assign conclusively the bands of ruthenium(III) complexes appearing in the visible region. Hence, all the bands that appear in this region have been assigned to charge transfer transitions, which are in conformity with the assignments made for similar ruthenium(III) complexes [26–28].

The effective magnetic moments (μ_{eff}) of some of the complexes were measured at room temperature using vibrating sample magnetometer. These values range from 1.92 to 2.02 BM

corresponding to single unpaired electron in a low-spin $4d^5$ configuration and confirms that ruthenium is in +3 oxidation state in all the complexes [29,30].

The solid state EPR spectra of the powdered samples were recorded at room temperature in X-band frequencies using DPPH as internal standard. The spectra showed no indication of any hyperfine splitting. All the complexes exhibit spectra with g_{\perp} in the range 2.52–2.59 and g_{\parallel} in the range 2.38–2.47 (Table 3;

Table 3
EPR spectra data of the Ru(III) complexes

Complex	g_x	g_y	g_z	$\langle g \rangle^*$
(1)	2.53	2.53	2.42	2.49
(2)	2.52	2.52	2.41	2.48
(3)	2.53	2.53	2.42	2.49
(4)	2.52	2.52	2.43	2.49
(5)	2.56	2.56	2.47	2.53
(6)	2.56	2.56	2.40	2.50
(7)	2.56	2.56	2.47	2.53
(8)	2.56	2.56	2.38	2.50
(9)	2.54	2.54	2.48	2.52
(10)	2.57	2.57	2.47	2.53
(11)	2.55	2.55	2.47	2.52
(12)	2.59	2.59	2.41	2.53

$$\langle g \rangle^* = [1/3 g_x^2 + 1/3 g_y^2 + 1/3 g_z^2]^{1/2}.$$

Table 4
Cyclic voltammetric data^a for some Ru(III) complexes

Complex	Ru(IV)–Ru(III)				Ru(III)–Ru(II)			
	E_{pa} (V)	E_{pc} (V)	E_f (V)	ΔE_p (mV)	E_{pa} (V)	E_{pc} (V)	E_f (V)	ΔE_p (mV)
(2)	1.06	–	–	–	–0.97	–0.73	–0.85	240
(3)	1.13	–	–	–	–0.89	–0.67	–0.78	220
(6)	1.46	1.26	1.36	200	–0.97	–0.74	–0.85	230
(7)	1.23	–	–	–	–0.96	–0.74	–0.85	220
(8)	0.78	–	–	–	–1.00	–0.74	–0.87	260
(9)	1.30	0.92	1.11	380	–0.93	–0.63	–0.78	300

^a Supporting electrolyte: [NBu₄]ClO₄ (0.05 M); concentration of the complex: 0.001 M; scan rate: 100 mV s^{–1}; all the potentials were referenced to s.c.e.

Fig. 1). The presence of two different ‘g’ values indicates a tetragonal distortion in an octahedral field, where $g_x = g_y \neq g_z$ [31,32].

3.2. Redox properties

The redox behavior of some of the complexes was studied by cyclic voltammetry in acetonitrile containing 0.05 M [NBu₄]ClO₄. The electrochemical data are compiled in Table 4. Since the ligands used in this study are not reversibly oxidized or reduced in the applied potential range, it is assumed that all redox processes are metal centered only. The cyclic voltammograms (Fig. 2) of the complexes exhibit a reversible oxidation peak at a scan rate of 100 mV s^{–1}. The oxidation and reduction of each complex are characterized by well-defined waves with E_f values in the range +1.11 to +1.36 V and –0.78 to –0.87 V, respectively. The peak to peak separation values (ΔE_p) for the redox processes [Ru(IV)–Ru(III)/Ru(III)–Ru(II)] range from 200 to 380 mV, suggestive of a quasi-reversible single step one electron transfer process [33,27]. The complexes [RuCl₂(PPh₃)₂(NCA)], [RuCl₂(PPh₃)₂(NMA)], [RuCl₂(AsPh₃)(NMA)] and [RuCl₂(AsPh₃)₂(NMOA)] showed an irreversible oxidation peak, which may be ascribed as due to the short-lived oxidized state of the metal ion. Further, a close

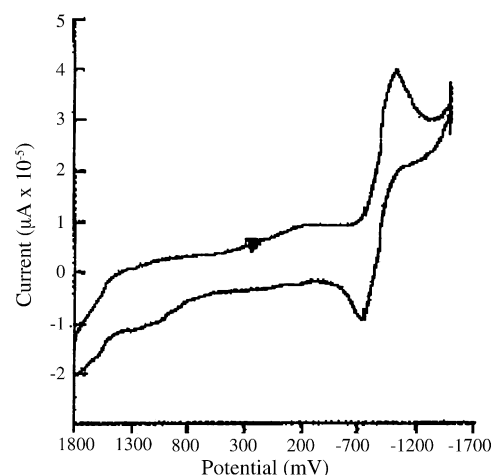


Fig. 2. Cyclic voltammogram of [RuCl₂(AsPh₃)₂(NCA)].

examination on the data reveals that the redox potentials do not vary much due to replacement of triphenylphosphine by triphenylarsine.

The E_f (oxidation) values of the complexes [11] containing one phenyl ring in the aldehyde part of the Schiff base ligands range from 0.56 to 0.79 V and E_f (reduction) ranges from –0.22 to –0.31 V [11]. When these values are compared with that of new complexes, it has been observed that with the addition of another phenyl ring in the ligand system, there is a positive shift in the E_f values (both oxidation and reduction). This can be explained by the fact that the additional phenyl ring, by its electron withdrawing nature decreases the electron density around the metal centre [34].

3.3. Catalytic oxidation

A few of the new ruthenium(III) complexes were tested for their catalytic activity in the oxidation of benzyl alcohol using NMO as oxidant (Table 5, Entry 2–5). Benzyl alcohol was converted to benzaldehyde. The product was quantified as its 2,4-dinitrophenyl hydrazone derivative [35]. In no case, there was any detectable yield of oxidized product in the absence of catalyst. From the comparative yield, it is evident that triphenylphosphine complexes possess more catalytic activity than triphenylarsine complexes because Ru–P bond is more labile than Ru–As bond. It has already been established that the catalytic cycle passes through a Ru^V=O species [36]. The char-



Fig. 1. EPR spectrum of [RuCl₂(AsPh₃)₂(NMOA)].

Table 5
Catalytic oxidation of alcohols by Ru(III) complexes in the presence of NMO

Entry	Complex	Yield ^a of benzaldehyde (%)	Turnover ^b
1	[RuCl ₂ (PPh ₃) ₂ (sal- <i>p</i> -toluidine)]	39.86	109.54
2	(1)	65.18	179.10
3	(6)	42.61	117.08
4	(7)	46.73	128.39
5	(12)	40.34	110.86

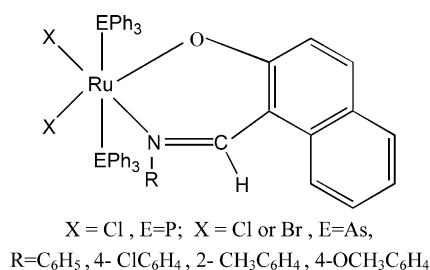
^a Yields based on substrate.

^b Moles of product per mole of catalyst.

acteristic peak at 856 cm⁻¹ (Ru=O) in the IR spectrum of the reaction mixture (after stirring for 3 h) supports the above.

Further, to study the effect of additional phenyl ring in the aldehyde part of the Schiff base ligands, the catalytic activity of the reported complex [11] (Table 5, Entry 1), which contains one phenyl ring in the aldehyde part of the ligand was also tested. On comparing the percentage yields, it has been observed that the new complexes reported in this paper show better catalytic activity than that of the complex reported earlier. The increased catalytic activity of the new complexes may be attributed to the increase in electron withdrawing nature of the ligand due to the increased resonance by the additional phenyl ring [37].

Based on the analytical, spectral (IR, electronic and EPR), magnetic and electrochemical data, the following octahedral structure has been tentatively proposed for all of the new ruthenium(III) complexes.



4. Conclusions

Some new ruthenium(III) complexes have been synthesized using the Schiff bases formed by condensing some derivatives of aniline with 2-hydroxy-1-naphthaldehyde. The new complexes have been characterized by analytical, spectral (IR, electronic and EPR) and magnetic studies. The redox behavior of the complexes has been studied by cyclic voltammetry. An octahedral structure has been tentatively proposed for all the complexes. The catalytic activity of some of the complexes (in the oxidation of benzaldehyde) has also been evaluated.

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