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Synthesis, spectroscopic characterization, electrochemical behaviour and antibacterial activity of Ru(III) complexes of 2-[(4-N,N′-dimethylaminophenylimino)-methyl]-4-halophenol

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ABSTRACT

The reaction of the chelating Schiff base ligands 2-[(4-N,N′-dimethylaminophenylimino)-methyl]-4-X-phenol with $[Ru(Cl)_3(EPh_3)_3]$; (E = P or As); (X = Cl, Br or I) in the benzene afforded new stable ruthenium complexes of the general formula $[Ru(Cl)_2(EPh_3)_2(L)]$ (L = anion of bidentate Schiff bases). The newly synthesized complexes were characterized using molar conductivity, spectral (UV–vis, FT-IR and EPR) and electrochemical studies. The molar conductance measurements proved that all these complexes are non-electrolytes. All complexes show strong d–d transition in the visible region. The coordination of imine nitrogen and phenolic oxygen of ligands to ruthenium metal was confirmed with the change in the IR stretching frequency values. The EPR spectral data showed that the complexes are paramagnetic with one unpaired electrons. The redox behaviour of the complexes have been investigated by the cyclic voltammetric technique. All the complexes display an irreversible reduction (Ru^{III}/Ru^{II}) in the range of −0.826 to −0.971 V. In view of the biological activity, the ligands and the complexes were observed that all the complexes showed moderate activity. Also the antibacterial activity of the ligand increased on chelation with metal ion.

1. Introduction

There has been considerable interest in the ruthenium Schiff base complexes now-a-days because of their redox stability, excited state life time and excited state reactivities [1,2], effective catalysts [3–8], their ability to act as probes in investigating the structure of DNA [9,10] and its antibacterial activity [11–14].

Schiff bases a class of chelators capable of forming coordinate bonds with many of metal ions through both azomethine group and phenolic group or via its azomethine or phenolic groups [12–15]. Chemotherapeutic Schiff bases and their complexes receives significant interest and attention of inorganic biochemists because of their biological activity including anti-tumor, antibacterial, fungicidal and anticarcenogenic properties [16,17]. However, literature survey reveals that little work has been done on biological studies of Schiff base complexes of Ru(III) [5,18,19].

In the present investigation, we describe the synthesis, characterization, redox behaviour and antibacterial screening of new class of hexacoordinated ruthenium complexes of Schiff bases containing O and N donor atoms and $PPh_3/AsPh_3$. The Schiff bases used to prepare the new ruthenium(III) complexes were shown in Fig. 1.

2. Experimental

2.1. Materials

All the reagents used were chemically pure and AR grade. Solvents were purified and dried according to standard procedure [20]. $RuCl_3{\cdot}3H_2O$ was purchased from Loba Chemie Pvt. Ltd., Bombay, India and was used without further purification. 5-Halo-2-hydroxy benzaldehyde and 4-N,N′-dimethylaniline were purchased from Aldrich. $[Ru(Cl)_3(EPh_3)_3]$ (where E = P or As) was prepared by reported literature method [21].

2.2. Physical measurements

The electronic spectra in MeCN were obtained on a JASCO V-550 UV–Vis spectrophotometer. The IR spectra (KBr disc) were recorded on a JASCO FT-IR 410 spectrophotometer in the range of 4000–400 cm^{-1} ^1H NMR spectra were recorded in $CDCl_3$ with TMS

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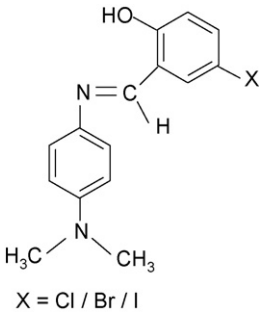

Fig. 1. Schiff base.

as an internal standard on a Joel GSX-400 MHz, FT NMR spectrophotometer. The ESR spectra in MeCN were obtained at 77 K on a E-112 varian ESR spectrometer. Cyclic voltammetric measurements were made in MeCN using a Bio Analytical System (BAS) model CV-50 W electrochemical analyser, with a glassy carbon working electrode, Pt auxiliary electrode and Ag/AgCl reference electrode: $[Bu_4N]ClO_4$ (TBAP) (0.1 M) was used as supporting electrolyte.

2.3. Synthesis of the ligands

The ligand was prepared by condensing 4-N,N′-dimethylaminoaniline in hot ethanolic solution of 4-halo-2-hydroxybenzaldehyde in 1:1 molar ratio. The purity of the compound was checked with TLC. The ^{1}H NMR spectra of all the Schiff bases provide compelling evidence of the presence of azomethine group.

2.3.1. 2-[(4-N,N′-dimethylaminophenylimino)-methyl]-4-chlorophenol (DMAPIMP-Cl)

Yield: 76%; m.p.: 142 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 13.759 (O–H, s, 1H); 8.541(–CH=N, s, 1H); 7.229–7.321(Ar, m, 4H); 6.732–6.754(Ar, d, 2H); 6.924–6.945(Ar, d, 1H); 3.009($N(CH_3)_2$, s, 6H).

2.3.2. 2-[(4-N,N′-dimethylaminophenylimino)-methyl]-4-bromophenol (DMAPIMP-Br)

Yield: 74%; m.p.: 145 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 13.805 (O–H, s, 1H); 8.561(–CH=N, s, 1H); 7.280–7.489(Ar, m, 4H); 6.888–6.932(Ar, d, 2H); 6.744–6.786(Ar, d, 1H); 3.031 ($N(CH_3)_2$, s, 6H).

2.3.3. 2-[(4-N,N′-dimethylaminophenylimino)-methyl]-4-iodophenol (DMAPIMP-I)

Yield: 78%; m.p.: 159 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 13.854 (O–H, s, 1H); 8.525(–CH=N, s, 1H); 7.262–7.634(Ar, m, 4H); 6.732–6.752(Ar, d, 2H); 6.774–6.796(Ar, d, 1H); 3.011($N(CH_3)_2$, s, 6H).

2.4. Synthesis of ruthenium(III) complexes

A solution of $[Ru(Cl)_3(PPh_3)_3]$ (0.1 mmol, 99.342 mg) or $[Ru(Cl)_3(AsPh_3)_3]$ (0.1 mmol, 112.542 mg) in C_6H_6 (20 cm^3) and a solution of the appropriate Schiff base (0.1 mmol, 27.4–36.6 mg) in $CHCl_3$(10 cm^3) was added, colour change was noticed on the addition of the ligand due to the formation of complexes (Fig. 2). The contents were refluxed for 4–5 h. The resulting solution was concentrated to small volume (3 cm^3) on a rotary evaporator and the product was separated by the addition of small amount of pet-ether (60–80 °C). The compound that separated was filtered, washed with benzene followed by ether, dried *in vacuo* over anhydrous $CaCl_2$, then recrystallised from 1:2 (v:v) chloroform-pet ether (60–80 °C) mixture.

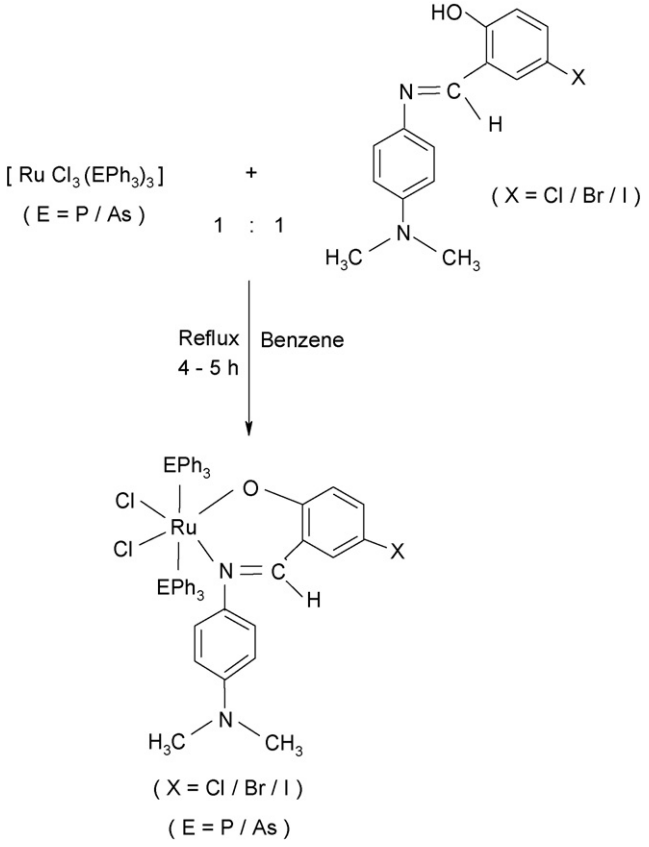

Fig. 2. Synthesis of ruthenium(III) complexes.

2.5. Antibacterial screening

The *in vitro* antibacterial screening effects of the investigated compounds were tested against the Gram +ve bacteria *Staphylococcus aureus* and Gram −ve bacteria *Proteus mirabilis* by the well diffusion method using agar nutrient as the medium at 37 °C for 18 h. The stock solutions of the Schiff bases and the complexes were prepared in 10% MeCN in methanol and were stored dry at room temperature. In a typical procedure [22] a well was made on the agar medium inoculated with microorganisms. The well was then filled with the test solution using a micropipette and the plates were incubated at 35 °C for 24 h for bacteria. During this period, the test solution diffused and the growth of the inoculated microorganisms was affected. The inhibition zone developed on the plate was measured and the data is summarized in Table 5. Ampicillin was used as the control.

3. Results and discussion

All the complexes are amorphous powder, insoluble in water and ether, but soluble in solvents such as $CHCl_3$, CH_2Cl_2, MeCN, DMF and DMSO. The molar conductance datas (Table 1) indicate that all the complexes are non-electrolytes [23].

3.1. Electronic absorption spectra

The electronic absorption spectral bands of the complexes were recorded over the range of 200–1100 nm in MeCN together with tentative assignments [24] (Table 1) are discussed in detail.

The low spin paramagnetic ruthenium(III), a d^5 system with $^2T_{2g}$ as the ground term and the first excited doublet levels in the order of increasing energy are $^2A_{2g}$ and $^2T_{1g}$, which arise from $^4T_{2g} \rightarrow {}^1E_g$ configuration. All complexes exhibit three bands in the range of

Table 1
Electronic spectral and molar conductance data.

Complex	λ_{max} (nm) (ε) ($dm^3/mol/cm$)[a]	Molar conductance ($\times10^{-3}$ Ω^{-1} cm^2 mol^{-1})	
		CH_3CN	CH_3OH
(1) $[Ru(Cl)_2(DMAPIMP\text{-}Cl)(PPh_3)_2]$	212 (20,320),[b] 369 (12,400),[c] 630 (510)[d]	27.6	44.8
(2) $[Ru(Cl)_2(DMAPIMP\text{-}Br)(PPh_3)_2]$	213 (20,700),[b] 374 (11,750),[c] 621 (530)[d]	26.9	42.1
(3) $[Ru(Cl)_2(DMAPIMP\text{-}I)(PPh_3)_2]$	203 (21,700),[b] 369 (12,600),[c] 618 (490)[d]	27.1	44.4
(4) $[Ru(Cl)_2(DMAPIMP\text{-}Cl)(AsPh_3)_2]$	214 (20,450),[b] 406 (12,380),[c] 616 (495)[d]	23.4	44.2
(5) $[Ru(Cl)_2(DMAPIMP\text{-}Br)(AsPh_3)_2]$	223 (19,400),[b] 365 (11,600),[c] 619 (550)[d]	22.1	43.0
(6) $[Ru(Cl)_2(DMAPIMP\text{-}I)(AsPh_3)_2]$	211 (19,800),[b] 355 (12,100),[c] 618 (520)[d]	20.8	44.6

[a] In acetonitrile.
[b] π–π^* transition.
[c] n–π^* transition.
[d] d–d transition.

Table 2
FT-IR spectral data (cm^{-1}) of the ligands and Ru^{III} complexes.

Compound	$\upsilon_{(C=N)}$	$\upsilon_{(C-O)}$	$\upsilon_{(O-H)}$	$\upsilon_{(Ru-O)}$	$\upsilon_{(Ru-N)}$	Bands for PPh_3/$AsPh_3$
DMAPIMP-Cl	1619	1369	3464	–	–	–
DMAPIMP-Br	1619	1370	3454	–	–	–
DMAPIMP-I	1619	1355	3430	–	–	–
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(PPh_3)_2]$	1611	1386	–	423	472	1428,1021,692
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(PPh_3)_2]$	1611	1361	–	425	469	1434,1078,695
$[Ru(Cl)_2(DMAPIMP\text{-}I)(PPh_3)_2]$	1611	1394	–	425	467	1434,1077,694
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(AsPh_3)_2]$	1611	1440	–	411	453	1433,1071,697
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(AsPh_3)_2]$	1611	1453	–	424	457	1435,1067,692
$[Ru(Cl)_2(DMAPIMP\text{-}I)(AsPh_3)_2]$	1611	1440	–	407	474	1434,1077,694

203–630 nm. The spectral profiles below 400 nm are very similar and are ligand centered transitions. The intense bands appeared in the range of 203–223 nm (ε = 19,400–21,700 dm^3/mol/cm) which can be assigned to π–π^* transition was due to transitions involving molecular orbitals located on the phenolic chromophore [25,26]. Another band appeared in the region 355–400 nm (ε = 11,600–12,400 dm^3/mol/cm) can be assigned to n–π^* transition was due to transitions involving molecular orbitals of the C=N chromophore and the benzene ring [26,27]. The other broad appeared in the range of 616–630 nm (ε = 490–530 dm^3/mol/cm) which can be attributed to d–d transitions in the metal orbitals [5,26].

3.2. Infrared spectra

The IR spectra (Table 2) of the free Schiff bases were compared with their respective ruthenium complexes in order to determine the coordination mode of the ligands [28]. The free ligands show a broad band of medium intensity observed at ca. 3430–3464 cm^{-1} is assigned to $\upsilon_{(O-H)}$ of the phenolic group. In complexes the two intense bands centered at ca. 1611 and 1361–1453 cm^{-1} assigned to $\upsilon_{(C=N)}$ and $\upsilon_{(C-O)}$, respectively [13,25]. The bands observed in the region of 407–425 cm^{-1} has been assigned to $\upsilon_{(Ru-O)}$ and $\upsilon_{(Ru-N)}$ [25,29]. The disappearance of $\upsilon_{(O-H)}$, the downward shift of $\upsilon_{(C-O)}$ and the lower frequency of $\upsilon_{(C=N)}$ [30] on complexation proves the bonding of ligands through imine nitrogen [31] and deprotonated phenolic oxygen [23]. Also the characteristic bands due to triphenylphosphine or triphenylarsine were observed in the expected regions [13].

3.3. Magnetic moment and EPR studies

The solid state EPR spectra of ruthenium(III) complexes [5,11] recorded at liquid nitrogen temperature (77 K) shows a simple three line spectra indicating magnetic anisotropy [13] in these systems and the complexes are in a low spin octahedral geometry. The average 'g' values (g_x, g_y, g_z) with $g_x \neq g_y \neq g_z$ lie in the 2.219–2.226BM range (Table 3) and these values fit very well with the values obtained for other similar octahedral ruthenium(III) complexes [32–34].

Table 3
ESR spectral and magnetic moment data of ruthenium(III) complexes.

Complex	g_x	g_y	g_z	$\langle g \rangle$[a]	μ_{eff}[b]
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(AsPh_3)_2]$	2.415	2.243	1.979	2.219	1.916
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(AsPh_3)_2]$	2.385	2.248	2.03	2.226	1.923
$[Ru(Cl)_2(DMAPIMP\text{-}I)(AsPh_3)_2]$	2.335	2.354	2.146	2.2803	1.975

[a] $[1/3\,g_x^2 + 1/3\,g_y^2 + 1/3\,g_z^2]^{1/2}$.
[b] $g_{av}\sqrt{s(s+1)}$.

The effective magnetic moments μ_{eff} (Table 3) lie in the range of 1.916–1.923BM also suggesting that these complexes are one electron paramagnetic low spin $^5t_{2g}$ (s = 1/2) configuration of the ruthenium(III) ion in an octahedral environment which is similar to reported O,N–coordinated Ru(III) complexes [5].

3.4. Electro chemical study

The redox properties of the mixed ligand complexes of ruthenium in MeCN were studied by cyclic voltammetry under nitrogen

Table 4
Electrochemical redox data of ruthenium(III) complexes.[a].

Complex	Ru^{II}/Ru^{III}				
	Potential (V)			Current (A)	
	E_{pa}	E_{pc}	ΔE_p	$E_{1/2}$	I_{pa}/I_{pc}
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(PPh_3)_2]$	−0.591	−1.147	0.556	−0.869	−0.2
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(PPh_3)_2]$	−0.667	−1.183	0.516	−0.925	−0.32
$[Ru(Cl)_2(DMAPIMP\text{-}I)(PPh_3)_2]$	−0.576	−1.076	0.505	−0.826	−0.98
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(AsPh_3)_2]$	−0.567	−1.122	0.555	−0.844	−0.64
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(AsPh_3)_2]$	−0.646	−1.296	0.65	−0.971	−0.2
$[Ru(Cl)_2(DMAPIMP\text{-}I)(AsPh_3)_2]$	−0.596	−1.288	0.619	−0.942	−0.14

[a] Solvent—acetonitrile; supporting electrolyte—$[Bu_4N]ClO_4$ (TBAP) 0.1 M; reference electrode—SCE; $E_{1/2} = 0.5(E_{pa} + E_{pc})$ where E_{pa} and E_{pc} are anodic and cathodic peak potential, respectively; $\Delta E_p = E_{pa} - E_{pc}$; scan rate = 100 mV s^{-1}.

Table 5
Antibacterial activities of Schiff base ligands and ruthenium(III) complexes.

Compound	Diameter of inhibition zone (mm)					
	Staphylococcus aureus			*Proteus mirabilis*		
	0.15%	0.2%	0.25%	0.15%	0.2%	0.25%
DMAPIMP-Cl	–	–	–	–	–	–
DMAPIMP-Br	–	–	–	–	–	–
DMAPIMP-I	–	–	–	–	–	–
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(PPh_3)_2]$	10	12	13	15	15	16
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(PPh_3)_2]$	9	10	13	14	15	16
$[Ru(Cl)_2(DMAPIMP\text{-}I)(PPh_3)_2]$	10	11	12	13	14	16
$[Ru(Cl)_2(DMAPIMP\text{-}Cl)(AsPh_3)_2]$	9	12	12	16	16	17
$[Ru(Cl)_2(DMAPIMP\text{-}Br)(AsPh_3)_2]$	8	9	10	14	15	17
$[Ru(Cl)_2(DMAPIMP\text{-}I)(AsPh_3)_2]$	10	12	12	16	17	18
Control (acetonitrile)	–	–	–	–	–	–
Standard (ampicillin)	30	32	34	22	23	24

Symbol "–" denotes no activity.

atmosphere and the relevant electrochemical data are given in Table 4. All Ru(III) complexes (Fig. 2) exhibit redox peak ($E_{1/2}$ values) in the range of −0.826 to −0.971 V has been assigned to metal-based reduction Ru(III)/Ru(II) [11] with peak-to-peak separation value (ΔE_p) ranging from 505 to 650 mV suggesting that an irreversible one electron transfer process [35]. Representative cyclic voltammogram of $[Ru(Cl)_2(PDMAAS\text{-}Cl)(PPh_3)_2]$ is shown in Fig. 3. The presence of such redox waves seems to be typical for ruthenium salicyliminato complexes [5,13,36]. The Schiff bases, when co-ordinated through N and O, stabilise ruthenium(III) rather than ruthenium(II). That is, the hard oxygen atom stabilises the higher oxidation state of ruthenium and lower valencies are stabilised by π-acid ligands like triphenyl phosphine and triphenyl arsine.

3.5. Antibacterial investigation

The Schiff base ligands and ruthenium(III) complexes were screened *in vitro* for their microbial activity against two pathogenic bacterial species *S. aureus* and *P. mirabilis* using the well diffusion method. The test solutions were prepared in MeCN and the results are summarized in Table 5. Blank experiments with $RuCl_3{\cdot}3H_2O$ and the Ru(III) precursors were carried out under identical experimental conditions and show the inability of these complexes to inhibit the bacterial growth. The effectiveness of an antimicrobial agent in sensitivity is based on the zones of inhibition. The diameter of the zone is measured to the nearest millimeter (mm). These compounds were found to exhibit moderate activity against both the organisms. The complexes are more active than their parent ligands, which is consistent with earlier reports [37,38]. Such an increased activity for the metal chelates as compared to the free ligands can be explained on the basis of Tweedy's chelation theory [39]. The activity increases with increase in concentration of test solution containing the new complexes. The different compounds exhibit microbial activity with small variations against the bacterial species and this difference in activity could be attributed to the impermeability of the cell of the microbes or differences in the ribosomes of the microbial cells [40]. Although the complexes are active, they did not reach the effectiveness of the conventional bacteriocide ampicillin.

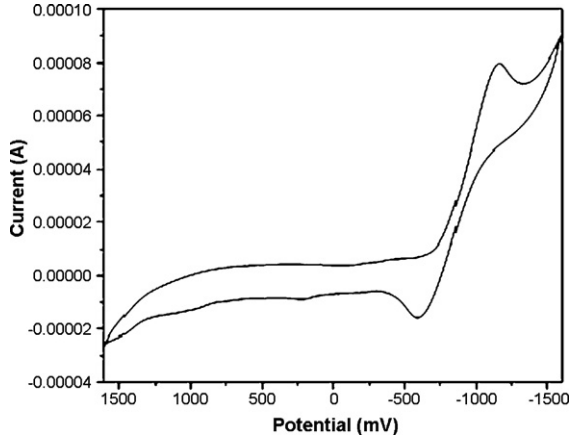

Fig. 3. Cyclic voltammogram of $[Ru(Cl)_2(PDMAAS\text{-}Cl)(PPh_3)_2]$.

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