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### Synthesis, Characteristic, Catalytic and Antimicrobial activities of Imidazolo substituted Benzylidene imines with Ruthenium (III) complexes

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#### Abstract

The synthesis and characterization of several Hexa – coordinated Ru(III) complexes of the type  $[\text{RuCl}(\text{CO})(\text{EPh}_3)_2(\text{L})]$  ( $\text{E} = \text{P}$  or  $\text{As}$ ;  $\text{L} =$  monobasic bidentate anion) were reported. FTIR, Electronic, EPR, electrochemistry and catalytic activity of the complexes were discussed. An octahedral geometry has been tentatively proposed for all these complexes. The new complexes have been subjected to antimicrobial investigations are also discussed.

**Key words:** Synthesis, Catalytic activity, Antimicrobial activity, Ruthenium (III) complexes.

#### Introduction

In the recent years there has been considerable interest in the chemistry of transition metal complexes of Schiff bases<sup>1</sup>. The metal ruthenium possesses several favorable chemical properties that indicate it may be a strong candidate to form a basis for rational anticancer drug design<sup>2,3</sup>. Benzylidene imines have vast medicinal importance showing a number of activities such as tuberculostatic, bactericidal, fungicidal, anti-inflammatory etc.<sup>4-6</sup>. The pharmacological importance of imidazolones and imines are also reported<sup>7-10</sup>. Bidentate complexes have been employed as catalyst for many reactions and as biological models in understanding the structure of bio-molecules and biological process<sup>11, 12</sup>. They are increasingly important for designing metal complexes related to synthetic and

natural oxygen carriers<sup>13</sup>. The real impetus towards developing their coordination chemistry was their physicochemical properties and significant biological activities<sup>14,15</sup>. The chemistry of ruthenium is currently receiving a lot of attention, primarily because of the fascinating electron transfer properties displayed by the complexes of this metal<sup>16</sup>. Ruthenium offers a wide range of oxidation states and the reactivity of the ruthenium complexes depend on the stability and interconvertibility of these oxidation states, which in turn depend on the nature of the ligand bound to the metal complexation of ruthenium by ligands of different types has thus been of particular interest<sup>17,18</sup>. Herein, we are reporting the preparation, spectral, catalytic activity and antimicrobial activities of some Ru(III) complexes containing bidentate ligands. The general structure of the ligands is given in Fig. 1.

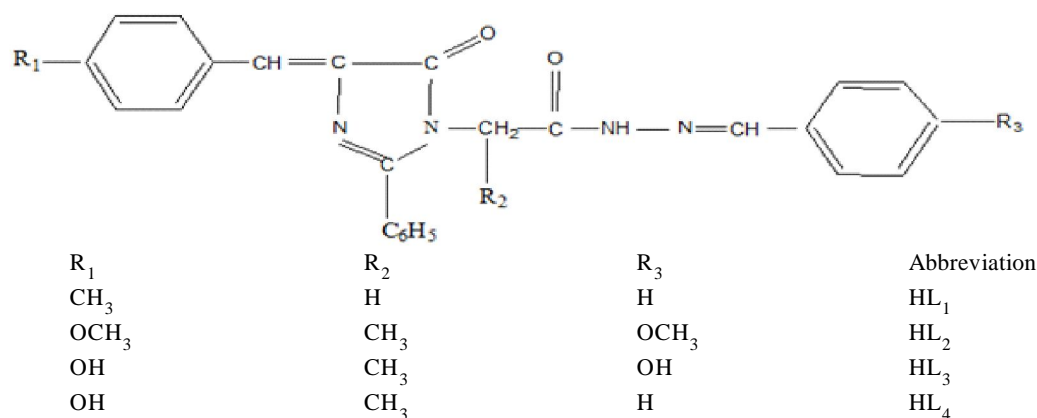


Fig. 1. Structure of ligands

### Experimental Procedure

#### Materials and Methods:

The entire reagents used were of AR grade. Solvents were purified and dried according to standard procedures.  $RuCl_3 \cdot 3H_2O$  purchased from Merck was used without further purification. The starting complexes  $[RuCl_3(PPh_3)_3]^{19}$ ,  $[RuCl_3(AsPh_3)_3]^{20}$ ,  $[RuBr_3(AsPh_3)_3]^{21}$  and  $[RuBr_3(PPh_3)_2(MeOH)]^{22}$  and the ligands<sup>23</sup> were prepared according to the literature procedures. Catalytic oxidation of alcohols and antibacterial studies were carried out according to our earlier procedures<sup>24,25</sup>.

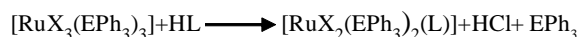
#### Preparation of new Ru (III) complexes:

All the reactions were carried out under anhydrous condition. To a solution of  $[RuX_3(EPh_3)_3]$  ( $E = P$  or  $As$ ,  $X = Cl$  or  $Br$ ) (0.1g, 0.08 - 0.01 mmol) or  $[RuBr_3(PPh_3)_2(MeOH)]$  (1.12 g, 0.01 mmol) in benzene (20  $cm^3$ ) the respective ligands (0.03-0.08g, 0.08 - 0.1 mmol) were added. The

resulting solution was concentrated to *Ca.* 3 $cm^3$  and the product was separated by the addition of the small amount of light petroleum (60 – 80°C). It was filtered and recrystallized from  $CH_2Cl_2$ /light petroleum (60 -80°C) and dried in Vacuum (yield = 70 - 85%).

### Results and Discussion

Light and air stable Ru(III) complexes of the general formula  $[RuX_2(EPh_3)_2(L)]$  ( $E = P$  or  $As$ ;  $L$  = monobasic bidentate anion) have been prepared by reacting  $[RuX_3(EPh_3)_3]$  and  $[RuBr_3(PPh_3)_2(MeOH)]$  with the respective ligands in a 1:1 molar ratio in benzene (Fig. 1).



The analytical data obtained for the new complexes (Table 1) agree very well with the proposed molecular formulae in all of the above reactions, the ligand behave as monobasic bidentate ligands.

Table 1. Analytical data of new Ru(III) Complexes

Complex	Mp (°C)	Yield (%)	Found (Calculated)(%)		
			C	H	N
$[RuBr_2(PPh_3)_2(L_1)]$	168	75	61.68(61.69)	4.24(4.22)	4.68(4.64)
$[RuCl_2(AsPh_3)_2(L_1)]$	171	85	61.76(61.79)	4.23(4.23)	4.64(4.65)
$[RuCl_2(PPh_3)_2(L_2)]$	179	74	65.31(65.31)	4.67(4.67)	4.74(4.75)
$[RuBr_2(PPh_3)_2(L_2)]$	168	72	60.92(60.95)	4.35(4.36)	4.43(4.44)
$[RuCl_2(AsPh_3)_2(L_2)]$	173	81	60.75(60.75)	4.32(4.35)	4.48(4.43)
$[RuCl_2(PPh_3)_2(L_3)]$	170	83	64.79(64.80)	4.45(4.44)	4.86(4.87)
$[RuBr_2(PPh_3)_2(L_3)]$	165	80	60.08(60.09)	4.12(4.12)	4.51(4.52)
$[RuCl_2(AsPh_3)_2(L_3)]$	168	77	60.20(60.21)	4.11(4.13)	4.53(4.53)
$[RuCl_2(PPh_3)_2(L_4)]$	166	79	65.72(65.72)	4.51(4.50)	4.93(4.94)
$[RuBr_2(PPh_3)_2(L_4)]$	175	80	60.87(60.88)	4.16(4.17)	4.56(4.58)
$[RuCl_2(AsPh_3)_2(L_4)]$	179	82	60.97(60.98)	4.18(4.18)	4.58(4.59)

*FTIR spectrum:*

The IR spectra of the free ligands were compared with those of the new complexes in order to confirm the coordination of benzylidene imine to the ruthenium metal. The IR spectrum of the free ligands showed a band in the absorption due to ( $>C=N$ ) appears in the 1600-1620  $\text{cm}^{-1}$  region undergoes a negative shift by 5 - 25  $\text{cm}^{-1}$  in the spectra of the complexes indicating the co-ordination of azomethine to the metal<sup>26</sup>. A strong band which appeared in the spectra of the ligands around 1610  $\text{cm}^{-1}$  due to  $-C=O$  completely disappeared and a new band was observed around 1570  $\text{cm}^{-1}$ . This may be due to the enolisation and subsequent coordination through the deprotonated oxygen atom of the  $-CH-C=O$  group<sup>27</sup>. In addition all the complexes show a new band at 1520  $\text{cm}^{-1}$  due to the cyclic  $-C=O$  group. This band remains unaltered in the corresponding metal complexes indicating the non-involvement of this group on complexation<sup>28</sup>. In addition to the above, the characteristic bands due to  $PPh_3$  or  $AsPh_3$  were also present in the expected region<sup>29,30</sup>.

*Electronic spectral analysis:*

The electronic spectra showed two to three bands in the 290-690 nm regions. The ground state of Ru(III) is  $2T_{2g}$  and the first excited doublet levels in order to increasing

energy are  $2A_{2g}$  and  $2T_{1g}$  which arises from  $t_{5g}e_{1g}$  configuration<sup>31</sup>. In the most of the Ru(III) complexes the electronic spectra showed only charge transfer bands<sup>32</sup>. The band in the 690 – 595 nm regions have been assigned to the d – d transition. Which is in conformity with assignments made for the similar Ru(III) complexes<sup>33,34</sup>. Other bands in the 375 – 230 nm region have been assigned to the charge transfer transitions<sup>35</sup>. In general the electronic spectra of all the complexes are characteristic of an octahedral environment around Ru(III) ions. The data of FTIR and electronic spectra are shown in Table 2.

*EPR spectrum:*

All the Ru(III) complexes are paramagnetic showing a +3 oxidation state for ruthenium ion. The solid state EPR spectra at X-band frequencies for several new Ru(III) complexes have been recorded at room temperature. The g value of the complexes is listed in Table 3. All the complexes show single isotropic resonances with a “g” value in the range. The isotropic lines of the type usually observed are either due to the intermolecular spin exchange, which may be broaden the lines or to the occupancy of the unpaired electron in degenerate orbital. The nature and pattern of the EPR spectrum suggests an almost perfect octahedral environment around ruthenium ion in these complexes<sup>36, 37</sup>.

Table 2. FTIR and Electronic spectral data of the new Ru(III) complexes

S.No	Complex	$\nu(C=N)\text{cm}^{-1}$	$\nu(C=O)\text{cm}^{-1}$	$PPh_3 / AsPh_3$	$\lambda_{\text{max}}(\text{nm})$
1.	$[RuCl_2(PPh_3)_2(L_1)]$	1545	1570	1434, 1085, 692	90,400,250
2.	$[RuBr_2(PPh_3)_2(L_1)]$	1595	1560	1433, 1086, 694	690,390,250
3.	$[RuCl_2(AsPh_3)_2(L_1)]$	1600	1565	1435, 1087, 693	680,360, 280
4.	$[RuCl_2(PPh_3)_2(L_2)]$	1605	1570	1434, 1085, 688	670,370, 260
5.	$[RuBr_2(PPh_3)_2(L_2)]$	1590	1565	1436, 1078, 670	680,350, 270
6.	$[RuCl_2(AsPh_3)_2(L_2)]$	1600	1560	1437, 1093, 682	690,350, 280
7.	$[RuCl_2(PPh_3)_2(L_3)]$	1595	1565	1435, 1086, 696	680,370, 270
8.	$[RuBr_2(PPh_3)_2(L_3)]$	1605	1565	1433, 1084, 692	680,370, 260
9.	$[RuCl_2(AsPh_3)_2(L_3)]$	1595	1570	1432, 1078, 694	690,350, 260
10.	$[RuCl_2(PPh_3)_2(L_4)]$	1585	1570	1431, 1092, 670	680, 280
11.	$[RuCl_2(AsPh_3)_2(L_4)]$	1600	1560	1436, 1076, 684	690,350, 270
12.	$[RuBr_2(PPh_3)_2(L_4)]$	1585	1570	1434, 1074, 690	680,370, 290

*Magnetic moments:*

The magnetic moments for Ru(III) complexes have been measured at room temperature using a vibrating sample magnetometer and diamagnetic corrections have been

applied. The value obtained were from 1.90 -2.00 BM indicating the presences of one unpaired electron, suggesting a low spin  $t_{2g}^5$  configuration for the ruthenium(III) ion an octahedral environment in all of these complexes<sup>38</sup>.

Table 3. EPR spectral data and magnetic moment of Ru(III) complexes

Complex	gx	gy	gz	g*	$\mu_m$
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>1</sub> ]	2.15	2.15	2.15	2.15	1.92
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>1</sub> ]	2.17	2.17	2.17	2.17	n
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> L <sub>1</sub> ]	2.19	2.19	2.19	2.19	1.90
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>2</sub> ]	2.3	2.3	2.3	2.3	n
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>2</sub> ]	2.28	2.28	2.28	2.28	1.96
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> L <sub>2</sub> ]	2.35	2.35	2.35	2.35	1.93
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>3</sub> ]	2.32	2.32	2.32	2.32	1.97
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>3</sub> ]	2.18	2.18	2.18	2.18	n
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> L <sub>3</sub> ]	2.20	2.20	2.20	2.20	2.00
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>4</sub> ]	2.27	2.27	2.27	2.27	n
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> L <sub>4</sub> ]	2.25	2.25	2.25	2.25	n
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> L <sub>4</sub> ]	2.22	2.22	2.22	2.22	1.98

$$g^* = (1/3g_x^2 + 1/3g_y^2 + 1/3g_z^2) \quad n = \text{not recorded}$$

#### Electrochemistry:

Complexes were electrochemically examined at a glassy carbon working electrode in dichloromethane solution using cyclic voltammetry. The potential data are listed in Table 4. The complexes display the Ru(III)–Ru(II) and Ru(III)–Ru(IV) couples in the potential ranges –0.43 to –0.67 and 0.83 to 1.17 V respectively vs SCE. In this, the Ru(III)–Ru(II) redox couple is quasi-reversible in nature, with a peak-to-peak separation (*E<sub>p</sub>*) of 120–370 mV, and

the Ru(III)–Ru(IV) couple is irreversible. The reason for the irreversibility of these complexes may be oxidative degradation or the short-lived oxidized state of the metal ion.<sup>38</sup> The *E<sub>1/2</sub>* value of a complex containing thiophenolato donor is more cathodic than that of a complex containing phenolato donor. Coordination of the Oxygen atom makes the metal center more electron-rich and shifts the oxidation potential towards more negative values<sup>39, 40</sup>.

Table 4. Cyclic voltammetry data for some Ru(III) complexes

Complex	Ru(IV) - Ru(III)				Ru(II)- Ru(III)			
	E <sub>pa</sub> (V)	E <sub>pc</sub> (V)	E <sub>f</sub> (V)	$\Delta E_p$ (mV)	E <sub>pa</sub> (V)	E <sub>pc</sub> (V)	E <sub>f</sub> (V)	$\Delta E_p$ (mV)
RuCl(PPh <sub>3</sub> ) <sub>2</sub> L <sub>1</sub>	-	-	-	-	-0.05	-0.2	-0.075	250
RuCl(PPh <sub>3</sub> ) <sub>2</sub> L <sub>2</sub>	0.06	0.45	0.525	150	-0.3	-0.25	-0.125	250
RuCl(PPh <sub>3</sub> ) <sub>2</sub> L <sub>3</sub>	-	-	-	-	-0.05	-0.3	-0.175	250
RuCl(PPh <sub>3</sub> ) <sub>2</sub> L <sub>4</sub>	0.05	0.4	0.225	350	-0.825	-0.66	-0.782	165
RuCl(PPh <sub>3</sub> ) <sub>2</sub> L <sub>5</sub>	-	-	-	-	-0.2	-0.55	-0.325	350

#### Catalytic activity:

The oxidation of alcohols was carried out with the ruthenium complexes as catalyst in the presence of *N*-methylmorpholine *N*-oxide(NMO) as co-oxidant in chloroform (Table 5). Benzaldehyde was formed from benzylalcohol and cyclohexanol was converted into cyclohexanone after stirring for 3 h at room temperature. The products formed were quantified as their 2,4-dinitrophenyl hydrazone derivatives. In no case, there was

any detectable oxidation of alcohols in the presence of NMO alone and without ruthenium complexes. All of the synthesized ruthenium complexes were found to catalyze the oxidation of alcohols to carbonyl compound but the yield and turnover were found to vary with different catalyst. The yield and turnover number are comparable with those reported for the oxidation of alcohols by similar Ru(III) complexes<sup>41</sup>. It has also been found that PPh<sub>3</sub> complexes possess higher catalytic activity than the AsPh<sub>3</sub>

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

Complex	Substrate <sup>a</sup>	Product	Yield <sup>b</sup>	Turnover <sup>c</sup>
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	Benzylalcohol	A	71	75
	cyclohexanol	K	63	67
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	Benzylalcohol	A	67	71
	cyclohexanol	K	56	60
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	Benzylalcohol	A	82	86
	cyclohexanol	K	75	79
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	Benzylalcohol	A	75	76
	cyclohexanol	K	65	69
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	Benzylalcohol	A	80	84
	cyclohexanol	K	73	77
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	Benzylalcohol	A	70	74
	cyclohexanol	K	62	66
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	Benzylalcohol	A	75	79
	cyclohexanol	K	66	70
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	Benzylalcohol	A	73	77
	cyclohexanol	K	59	63
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	Benzylalcohol	A	72	76
	cyclohexanol	K	65	65
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	Benzylalcohol	A	72	76
	cyclohexanol	K	65	65
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	Benzylalcohol	A	70	74
	cyclohexanol	K	63	68
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	Benzylalcohol	A	80	84
	cyclohexanol	K	74	78

<sup>a</sup>A: Benzaldehyde ; K: Cyclohexanone; <sup>b</sup>Yield based on substrate; <sup>c</sup> Moles of product per more of catalyst.

complexes<sup>42</sup>. This may be due to the higher donor ability of the arsine ligand compared to the phosphine ligand. The relative higher product yield obtained for the oxidation of benzyl alcohol than for cyclohexanol is due to the fact that the – CH moiety of benzyl alcohol is more acidic compared to that of cyclohexanol<sup>43, 44</sup>. The catalytic oxidation is expected to proceed via Ru(IV) = O intermediate as reported by us earlier<sup>45</sup>.

#### Antimicrobial study:

The invitro antimicrobial screening of the ligands and their ruthenium complexes have been carried out against *Escherichia coli*, *Aeromonashydrophila* and *Salmonella typhi* using a nutrient agar medium by disc diffusion method<sup>46</sup>. The results (Table 6) showed the complexes exhibit moderate activity against *Escherichia coli*, *Aeromonashydrophila* and

*Salmonella typhi*. The toxicity of ruthenium chelates increases on increasing the concentration<sup>47</sup>. The increase in the antimicrobial activity of the metal chelates may be due to the effect of the metal ion on the normal cell process. A possible mode of the toxicity increase may be considered in light of Tweedys chelation theory<sup>48</sup>. Chelation considerable reduces the polarity of the metal ion because of partial sharing of its positive charge with the donor groups and possible  $\pi$  - electron delocalization over the whole chelate ring. Such chelation could enhance the lipophilic character of central metal atom, which subsequently favours its permeation through the lipid layers of cell membrane. Furthermore, the mode of action of the compounds may involve formation of a hydrogen bond through the azomethine (>C=N) group with the active centers of cell constituents, resulting in interference the normal cell

Table 6. Antimicrobial activity of ligands and Ru(III) new complexes

Ligand/Complex	Diameter of inhibition Zones (mm)								
	<i>Escherichia coli</i>			<i>Aeromonashychofla</i>			<i>Salmonella typhi</i>		
	0.25%	0.5%	1%	0.25%	0.5%	1%	0.25%	0.5%	1%
HL <sub>1</sub>	10	12	13	9	10	12	8	10	11
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	12	14	16	11	14	17	10	12	15
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	11	13	15	10	11	13	10	12	14
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>1</sub> )]	12	13	14	11	12	12	9	14	15
HL <sub>2</sub>	10	11	13	10	12	13	10	12	13
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	12	14	17	12	14	19	12	14	15
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	11	13	16	11	13	15	11	13	14
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>2</sub> )]	12	13	14	12	13	14	11	13	15
HL <sub>3</sub>	10	11	12	9	10	11	9	11	12
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	13	14	16	10	12	14	10	12	15
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	12	15	16	10	11	13	10	12	14
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>3</sub> )]	12	13	12	10	11	12	13	14	15
HL <sub>4</sub>	9	10	12	8	10	11	8	11	12
[RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	11	13	14	10	12	13	11	12	13
[RuBr <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	11	12	13	10	11	13	10	11	14
[RuCl <sub>2</sub> (AsPh <sub>3</sub> ) <sub>2</sub> (L <sub>4</sub> )]	10	11	13	10	11	12	11	12	13
Streptomycin	22	23	28	21	27	29	29	21	25

process<sup>49</sup>. Though the complexes possess activity they could not reach the effectiveness of the standard drug streptomycin. The variation in the effectiveness of the different compounds against different organism depend either of impermeability of the cells or the microbe of

difference in ribosome of microbial cells<sup>50</sup>.

Based on the analytical and spectral (electronic), electrochemical data, an octahedral structure (Fig. 2) has been tentatively proposed for all of the new Ru(III) complexes.

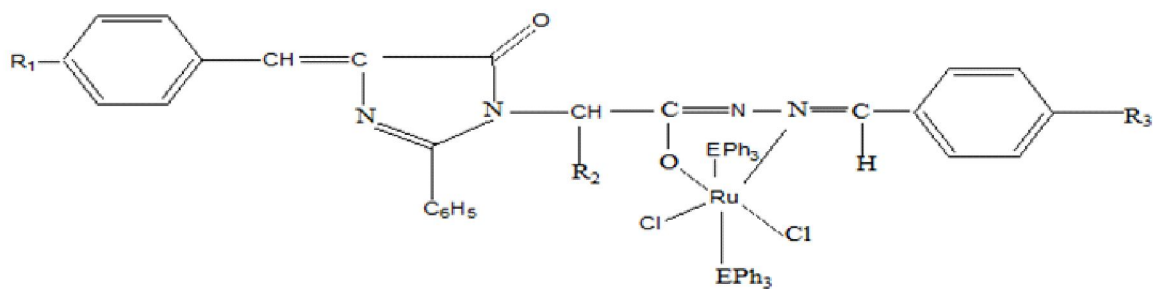


Fig. 2. Structure of new Ru(III) complexes

(R<sub>1</sub> = H, OCH<sub>3</sub>, OH; R<sub>2</sub> = CH<sub>3</sub>; R<sub>3</sub> = H, OCH<sub>3</sub>, OH; E = P or As)

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