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Synthesis and characterisation of Ruthenium (III) complexes from Schiff base synthesized from 1-phenyl-3-methyl-4-acetyl/benzoyl-5-pyrazolone with 4,4'-diaminodiphenyl methane and 4,4'-diamino diphenyl ether

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ABSTRACT: Schiff base ligands 4',4''-bis [(1'-phenyl-3'-R-5'-oxodihydropyrazol-4-yl) methyl imino] diamino diphenyl methane and 4',4''-bis [(1'-phenyl-3'-R-5'-oxodihydropyrazol-4-yl) methyl imino] diamino diphenyl ether where R is either methyl or benzoyl are complexed with Ruthenium (III) metal in basic conditions. The synthesized complexes are then characterised on the basis of elemental analysis, thermogravimetric analysis, ¹H-NMR, magnetic behaviour, electronic and infrared spectral studies. The complexes confirm to 1:1 stoichiometry. The complexes are found to be hexa coordinated with donor atoms being azomethine nitrogen, phenolic oxygen and oxygen of coordinated water molecule.

KEY WORDS: [Ruthenium (III), pyrazolone, schiff bases, N(azomethine group) & O(phenolic) donor atoms]

I. INTRODUCTION

Schiff base metal complexes have remained an important and popular area of research due to their simple synthesis, versatile and diverse range of applications¹. In recent years, the design and synthesis of multidentate phenoxy Schiff base ligands and their metal complexes have been a fascinating area of research because of their importance in basic and applied science^{2,3}. There have been many reports on their applications in biology including antibacterial, antifungal etc⁴. A great deal of attraction in this area has been focussed on the complexes formed by transition metal ions with Schiff bases because of the presence of both Nitrogen and oxygen donor atoms in the back bone of these ligands⁵.

For thousands of years, metal complexes have played important and diverse roles in medicine⁶. From the antiseptic properties of copper complexes, to the long-standing application of gold complexes in Chinese and Arabic medicines⁷. In a number of studies, ruthenium complexes have been shown to exhibit highly encouraging anti-cancer activity^{8,9}.

In recent years, several pyrazolone derivatives have been synthesized such as 4-acyl-pyrazolones¹⁰. It has been observed from literature survey that these 4-substituted pyrazolone derivatives mainly exhibit bidentate co-ordinate behaviour, in which keto-enol tautomerism plays an important role in the chelate formation with numerous metal ions. The acyl pyrazolones in turn are utilized in the synthesis of Schiff base pyrazolone^{11,12}, which act as either tridentate or tetra dentate ligands^{13,14}.

Ruthenium properties are well suited towards pharmacological applications. It can access a range of oxidation states (II, III and IV) under physiologically relevant conditions¹⁵. Much attention has been drawn towards the chemistry of Ru and Rh (acharya et al., 2006; Fairlie, Ilan & Taube, 1997) in different coordination spheres²¹. Ruthenium is a transition metal in group 8-the same chemical group as iron. Ruthenium binds tighter and more slowly than iron and has a preference for softer ligands¹⁶.

**II. RELATED WORK**

There have been multiple studies where complexes of ruthenium have been synthesized. Ru(III), Pd(II) and Co(II) complexes of thiosemicarbazone ligands were synthesized (Samar A. Aly 2018), characterised and were found to exhibit remarkable antibacterial study. Novel Schiff base ligands (dihydroxy benzaldehyde) and their Ru(II) complexes have been synthesized and were found to cleave nucleic acid and extent of cleavage was dose dependent (S. Saththiyaraj, G. Ayyannan, C. Jayabalakrishnan 2014).

The main objective of this research is to prepare and study the chemical composition of the new complexes of ruthenium (III). In this study we reported the synthesis and spectroscopic characterization of the Schiff base ligands and their Ru(III) complexes. The structure of ligands (synthesized) and their Ru(III) complexes were characterized using various spectral methods.

III. EXPERIMENTAL**A. Materials and Physical Measurements**

All the chemicals and solvents used in this research were of AR grade. Elemental analysis (C,H,N & O) was performed on a Thermo-finnigan CHNO analyser (IIT-B). The FTIR spectra was recorded on Shimadzu IR-Prestige-21 (Ruia College Mumbai) spectrophotometer in the range of 350cm^{-1} - 4500cm^{-1} and electronic spectra was recorded using Shimadzu UV-1800 in the range of 190-1100 nm in DMSO. ^1H NMR spectra was recorded with from NMR(300 Mhz-NMR2) Instrument of University of Mumbai. Samples were dissolved in DMSO(deuterated). Room temperature magnetic susceptibility was carried out on powdered samples using a Sherwood Scientific Magnetic Susceptibility balance (Banasthali University Jaipur). TGA of samples were carried out on Perkin Elmer, USAA, Model Diamond TG/DTA (Saif-IITb). Samples were heated from 30°C to 1020°C at $10^\circ\text{C}/\text{min}$.

B. Synthesis of Schiff bases**a. Synthesis of 3-methyl-1-phenyl pyrazol-5-one.**

3-methyl-1-phenylpyrazolone was prepared by the reported method¹⁷. The yield of 3-methyl-1-phenylpyrazol-5-one was about 52 g (80-85% yield).

b. Synthesis of 4-benzoyl-3-methyl-1-phenyl pyrazol-5-one and Synthesis of 4-acetyl-3-methyl-1-phenyl pyrazol-5-one.

The 4-substitued-3-methyl -1- phenyl pyrazolones were prepared by the reported method¹⁸. The yield of 4-benzoyl-3-methyl-1- phenyl pyrazol-5-one was about 65-70% and the yield of 4-acetyl-3-methyl-1- phenyl pyrazol-5-one was about 55-60%.

c. Ligand synthesis

- i. Ligand 1(L1) synthesis – 4',4''-bis [(1'-phenyl-3'-methyl-5'-oxodihydropyrazol-4'-yl) methyl imino] diamino diphenyl methane i.e. (HPMPZM)₂dpm.
- ii. Ligand 2(L2) synthesis- 4',4''-bis [(1'-methyl-3'-methyl-5'-oxodihydropyrazol-4'-yl) phenyl imino] diamino diphenyl methane i.e. (HPMPZP)₂dpm.
- iii. Ligand 3(L3) synthesis-4',4''-bis [(1'-phenyl-3'-methyl-5'-oxodihydropyrazol-4'-yl) methyl imino] diamino diphenyl ether i.e. (HPMPZM)₂dpe
- iv. Ligand 4(L4) synthesis-4',4''-bis [(1'-methyl-3'-methyl-5'-oxodihydropyrazol-4'-yl) phenyl imino] diamino diphenyl ether i.e. (HPMPZP)₂dpe.

The ligands L1(i), L2(ii), L3(iii) & L4(iv) were obtained by the reaction of 4-acetyl/benzoyl-3-methyl-1-phenyl pyrazol-5-one with 4,4'di amino diphenyl methane/4,4'di amino diphenyl ether in a 2:1 molar proportion in ethanol in the presence of traces of concentrated hydrochloric acid. The reaction mixture was refluxed on water bath for 18-22 hrs. on cooling the product was separated out and was recrystallized from alcohol. The yield of the product was 55-60%.

d. Synthesis of complexes

The ligand was dissolved in DMF. The equimolar quantity of trivalent metal chloride was dissolved in minimum quantity of distilled water. The ligand solution in DMF was slowly added to an aqueous solution of trivalent metal chloride with constant stirring (magnetic stirrer plate). The metal to ligand stoichiometry is (1:1). The pH of the solution was maintained by adding 0.1 N sodium hydroxide. The reaction was stirred for 4-5 hours (periodically checking pH and adding 0.1 N NaOH to maintain pH of 8-9). The solid complex separated out, filtered and air dried. This is the general method of preparation of four complexes of ruthenium ([RuL1(H₂O)₂]Cl, [RuL2(H₂O)₂]Cl₂.H₂O, [RuL3(H₂O)₂]Cl, and [RuL4(H₂O)₂]Cl. The proposed structure and stoichiometry of all the complexes is shown in figure 1 and table 1. Also Table 2 shows the elemental analysis of all the complexes.

IV. RESULTS AND DISCUSSION**A. IR Spectra**

Selected IR spectral bands for the ligands (L1, L2, L3 & L4) and its complexes are given in Table 3.

The IR spectra of the free ligands are characterized mainly by the broad bands at 3260-3000 cm⁻¹, 2340-3000 cm⁻¹ which can be assigned to symmetric and antisymmetric vibrations ν_{OH} . Wherein the ν_{CH} aromatic peaks were observed from 2820-2930 cm⁻¹ and ν_{CH} aliphatic peaks were observed from 2300-2380 cm⁻¹. The free ν_{OH} is normally observed between 3500 and 3600 cm⁻¹. The observed low value is due to intermolecular or intra molecular hydrogen bonding^{13,14}. The IR spectra of the Schiff bases (all ligands) shows bands in the region 1580-1630 cm⁻¹, corresponding to $\nu_{C=N}$ azomethine group and $\nu_{C=N}$ pyrazoline groups^{19,20,21}. The Schiff base ligands also show distinct bands between 1220-1250 cm⁻¹ due to δ_{OH} ¹³. There are bands observed at 1360-1388 cm⁻¹ because of ν_{C-O} phenolic^{13,21}. In the IR spectra of ruthenium complexes with schiff base ligands all the bands shift through a range of 10-2 cm⁻¹ indicating complexation. Presence of coordinated water in complexes is indicated by presence of band in the region of δ 820-880 cm⁻¹^{13,19}. Metal complexes shows broad strong absorption peaks at 3400-3880 cm⁻¹ showing the presence of coordinated water molecules^{5,13,20,22}. New bands with medium to weak intensities appear in the regions 570-500 cm⁻¹ and 490-400 cm⁻¹, supporting the formation of the Ru-O(ν_{M-O}) and Ru-N(ν_{M-N}) respectively (Bayoumi et. Al., 2013; Vinodo & Ankita, 2007)^{5,19,20,21,22}.

B. TGA analysis^{13,19}. (Table 4)

TGA of all complexes was recorded. There was not much weight loss observed from 80-120°C indicating the absence of water of crystallisation, except for the complex of ruthenium with ligand L2 where one molecule of water crystallisation is observed. Weight loss occurred between 150-300 °C is attributed to the coordinated water molecules (2 water molecules). All the four complexes showed significant loss of weight due to two molecules of coordinated water. After 300 °C till 600 °C there was loss of about 60-68% which indicates the disintegration of the whole complex. The conclusions of TGA spectra were supported by presence of spectral peaks in IR.

C. NMR studies of ligands and complexes of Ruthenium²³(Table 5)

¹HNMR spectra of ligands and complexes was carried out in solvent DMSO (both ligands and complexes were insoluble in commonly used solvents). The ligands L1, L2, L3 & L4 show signals for phenolic OH at δ 12.662-12.933ppm¹³. Phenolic 'H' peaks were not observed in the spectra of complexes indicating deprotonation of phenolic OH. Peaks due to methylene(-CH₂-) group were seen in ligands L1 and L2 at δ 3.9-4.066ppm²⁰ and on complexation methylene peaks underwent shift δ (3.9-4.072) ppm in [RuL1(H₂O)₂]Cl and [RuL2(H₂O)₂]Cl.H₂O. In ligands L3 & L4 the methylene peaks were not seen as -CH₂- group was replaced by -O- (non-coordinating). The ligands L2 and L4 showed extra peaks at δ 6.157-6.988ppm showing replacement of acetyl group by benzoyl groups.

D. Electronic spectra and magnetic susceptibility²⁰

The electronic spectra of the new ruthenium (III) complexes have been recorded in DMSO solution in the region 200-1100 nm. All ruthenium complexes display bands in region 350-410 nm which are due to, metal to ligand charge transfer. Magnetic moment values calculated for ruthenium complexes indicate spin d^5 configuration. So, the synthesized ruthenium (III) complexes belongs to the d^5 system and has 5 electrons in the outer orbital. After the pairing of d electrons, the d orbital has only single unpaired electron which is supported by the magnetic moment values in the range of 1.2-1.4 B.M. Complex $[RuL_2(H_2O)_2]Cl_2 \cdot H_2O$ showed an exceptional magnetic moment of 3.84 B.M. indicating oxidation of Ru(III) to Ru(IV) with four unpaired electrons in the d orbital.

V. CONCLUSION

A new family of Schiff base complexes of ruthenium incorporating 4-acetyl/benzoyl derivative of 1-phenyl-3-methyl-5-pyrazolone with 4,4'-diamino diphenyl methane and 4,4'-diamino diphenyl ether, were synthesized and characterized. The results of this investigation support the suggested structures of the metal complexes (figure 1). It can be concluded from this study that this stoichiometry of the complexes is 1:1 with ligand. The ligand coordinates with ruthenium (III) through 'N' donor atom which is part of the azomethine group and the phenolic O of the ligands. Ruthenium metal ion has +3 oxidation state. Deprotonated phenolic 'O', which has negative charge balances the +2, leaving +1 charge on the coordination sphere which is balanced by the chloride ion. In complex $[RuL_2(H_2O)_2]Cl_2 \cdot H_2O$ ruthenium metal ion has +4 oxidation state, so in this complex coordination sphere developed +2 charge which was balanced by two chloride ions (Table 1). All complexes also had two coordinated water molecules.

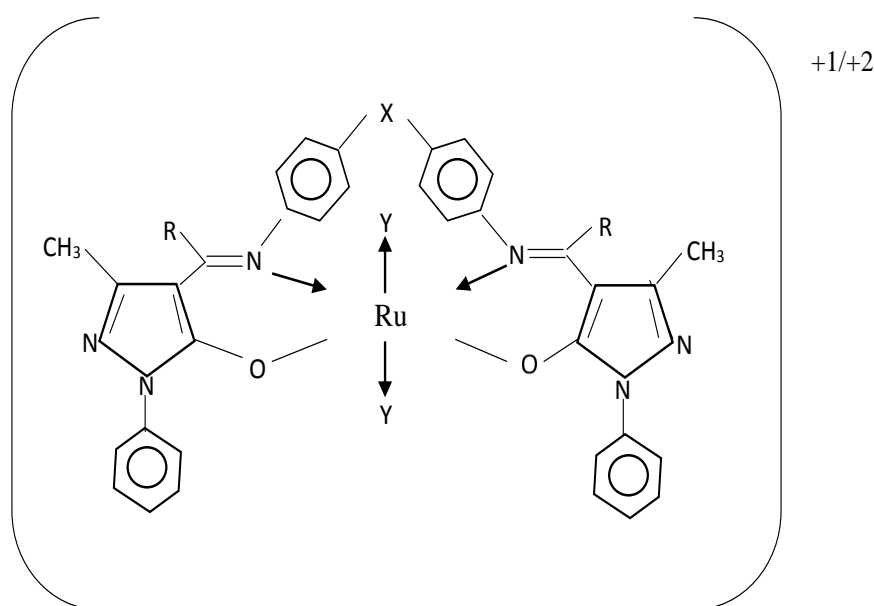
Figure: - 1

Table 1

S.No.	Coordination sphere	R	X	Y	Water of crystallisation	Cl	Complex Formula
1.	[RuL1(H ₂ O) ₂] ⁺	-CH ₃	-CH ₂ -	H ₂ O	None	One	[RuL1(H ₂ O) ₂]Cl
2.	[RuL2(H ₂ O) ₂] ²⁺	-C ₆ H ₅	-CH ₂ -	H ₂ O	One	Two	[RuL2(H ₂ O) ₂]Cl ₂ .H ₂ O
3.	[RuL3(H ₂ O) ₂] ⁺	-CH ₃	-O-	H ₂ O	None	One	[RuL3(H ₂ O) ₂]Cl
4.	[RuL4(H ₂ O) ₂] ⁺	-C ₆ H ₅	-O-	H ₂ O	None	one	[RuL4(H ₂ O) ₂]Cl

Table 2- Elemental Analysis

Complexes	%age C Result	%age C structure	%age H Result	%age H structure	%age N Result	%age N structure	%age O Result	%age O Structure
[RuL2(H ₂ O) ₂]Cl	57.64	57.9	5.05	4.96	10.953	10.96	13.12	8.35
[RuL2(H ₂ O) ₂]Cl ₂ .H ₂ O	59.6	59.8	4.13	4.66	9.05	8.9	9.34	8.48
[RuL2(H ₂ O) ₂]Cl	57.889	56.21	4.45	4.68	10.77	10.93	13.67	10.41
[RuL2(H ₂ O) ₂]Cl	59.32	61.82	4.33	4.48	9.1	9.41	11	8.96

Table 3:-IR peaks

Ligand/ Complex	ν_{OH} cm ⁻¹	ν_{H_2O} cm ⁻¹	$\nu_{C=N}$ cm ⁻¹ azomethine	$\nu_{C=N}$ cm ⁻¹ pyrazoline	ν_{C-O} cm ⁻¹ phenolic	δ_{OH} cm ⁻¹	δ_{H_2O} cm ⁻¹	ν_{M-N} cm ⁻¹	ν_{M-O} cm ⁻¹
(HPMPZM) ₂ dpm L1	3027-3065		1618.35	1580	1384.95	1225.82			
[RuL1(H ₂ O) ₂]Cl	Broad peak at 3028	3624-3847	1626.06	1573.98	1386.88	1227.74	825.33	397.34	470.63
(HPMPZP) ₂ dpm L2	3027.41 - 3065.02		1628.95	1592.31	1384.95	1227.4			
[RuL2(H ₂ O) ₂]Cl ₂ .H ₂ O	3334.92	3828.70	1630.88	1591.27	1382.96	1232.57	885.33	407	470.63
(HPMPZM) ₂ dpe L3	3400-broadness		1626.06	1540.23	1384.9	1250.89			
[RuL3(H ₂ O) ₂]Cl	3415.93 - 3498.87	3520.09 - 3888.49	1631.78	1556.55	1382.96	1247.94	817.82	403.12	509.21
(HPMPZP) ₂ dpe L4	3033.19		1630.88	1537.33	1383.02	1246.07			
[RuL4(H ₂ O) ₂]Cl	3003.17 - 3095.75	3660-3954	1640.53	1546.91	1382.96	1245.1	815.89	397.34	530.42

Table 4- Thermogravimetric Analysis

Complex	Mass by Formula	Water of crystallization Loss of weight b/w 80°C -100°C	Coordinated water Loss of weight b/w 150°C -300°C	By calculation %age of coordinated water molecules
[RuL2(H ₂ O) ₂]Cl	766.5	%age in TGA=not significant	%age in TGA = 4%	2 Water molecules= 36 36/766.5=4.69%
[RuL2(H ₂ O) ₂]Cl ₂ H ₂ O	943	%age in TGA = 1.98% 1 Water molecules= 18 18/943= 1.91%	%age in TGA =4.686%	2 Water molecules= 36 36/943= 3.82%
[RuL2(H ₂ O) ₂]Cl	768.5	%age in TGA=not significant	%age in TGA = 4.686%	2 Water molecules= 36 36/ 768.5 = 4.684%
[RuL2(H ₂ O) ₂]Cl	892.5	%age in TGA=not significant	%age in TGA = 3.5%	2 Water molecules= 36 36/ 892.5 = 3.89%

Table 5- NMR Peak Analysis

Ligand/complex	δ -CH ₃ protons at 3, 3'	δ -CH ₂ -	δ Phenyl	δ Phenolic proton
(HPMPZM) ₂ dpm L1	2.501-3.358 ppm	4.066 ppm	7.136-8.014 ppm	12.933 ppm
[RuL1(H ₂ O) ₂]Cl	2.500-3.367 ppm	4.072 ppm	7.134-8.013 ppm	
(HPMPZP) ₂ dpm L2	2.501-3.370 ppm	3.8-3.9 ppm	New peaks at 6.844,6.872&6.974 ppm Besides 7.001-8.013 ppm	12.706 ppm
[RuL2(H ₂ O) ₂]Cl ₂ H ₂ O	1.439-3.368 ppm	3.8 from doublet in ligand to singlet in complex	6.754, 6.781&6.985 ppm 7.157-8.008 ppm	
(HPMPZM) ₂ dpe L3	1.00-2.389-3.369 ppm		7.141-8.022 ppm	12.912 ppm
[RuL3(H ₂ O) ₂]Cl	1.1-2.388-3.362 ppm		7.141-8.022 ppm	
(HPMPZP) ₂ dpe L4	1.447-2.496-3.362 ppm		New peaks at 6.755,6.784&6.988 ppm Besides 7.017-8.022 ppm	12.662 ppm
[RuL4(H ₂ O) ₂]Cl	1.446-2.499-3.364 ppm		6.754,6.781&6.985 ppm 7.013-8.018 ppm	



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