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Synthesis and biological evaluation of some new schiff base metal complexes

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ABSTRACT

The present paper deals with the synthesis and characterization of metal complexes of new Schiff base derived from Pramipexole(4,5,6,7-Tetrahydro-N6-propyl,2,6-benzothiazolediamine). The bidentate ligand is derived from the inserted condensation of pramipexole with O-hydroxy benzaldehyde (OHB) in a 1:1 molar ratio. Using this bidentate ligand, complexes of Cu(II), Ru(II) with general formula ML_2 *have been synthesized. The synthesized complexes were characterized by several techniques using Elemental analysis, IR, NMR, UV-VisSpectrometry, ESR, Vibrational spin magnetometry, TG-DTA and Conductometric measurements. The elemental analysis data suggest that stoichiometry to be 1:2[M:L].All the complexes are non electrolytic in nature as suggested by molar conductance measurements. Infrared spectral data indicate the co-ordination between ligand and central metal ion through de protanated Phenolic oxygen and azomethine nitrogen. In addition the authors have been screened the compounds for biological activity. It was found that the compounds have shown activity against the organisms like Salmonella typhi, Enterococcus faecails and Escherichia coli.*

Keywords: Synthesis, Characterization, Schiff base (OHBP), pramipexole, Biological activity

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INTRODUCTION

Coordination complexes are gaining importance in recent years especially in the designing of long acting drugs in metabolism. The metal complexes from bidentate ligands have often been studied recently because of their technical applications [1,2] and applications in enhancement of drug action[3,4]. Transition metals are essential for normal functioning of living organism and are , therefore of great interest as potential drugs[5]. The coordination chemistry of nitrogen donor ligands is an active area of research. A great deal of attention in this area has been focused on the complexes formed by metals with bidentate ligands using both sulfur and nitrogen [6, 7]. The Schiff bases are an important class of ligands in coordination chemistry. The study of structural and binding features of various Schiff base complexes can play an important role in better understanding of the complex biological process. Schiff bases derived from salicylaldehyde are well known for their interesting ligational properties and exclusive applications in different fields[8–10]. It is well known from the literature that Schiff bases derived from pramipexole have a strong ability to form metal complexes [11]. The interaction of these donor ligands and metal ions gives complexes of different geometries, and literature survey reveals that these complexes are potentially more biologically active. Thus, in recent years Schiff bases and their metal complexes have attained much attraction because of their extensive biological activities [12, 13].Keeping the above fact in our mind and in continuation of our earlier work on transition metal complexes with Schiff bases [14, 15], the ligand OHBP Schiff base(L) has been synthesized. In the present paper, the synthesis and characterization of the ligand and its complexes with Cu(II), Ru(II) are being reported.

EXPERIMENTAL SECTION

Instrumentation

The percentage compositions of the elements (CHNO) for the compounds were determined using an element analyzer CHNO model Fison EA 1108.The Infra red spectra were recorded as potassium bromide (KBr) discs using

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a JASCO FT/IR-5300.The 1H (400Hz) nuclear magnetic resonance spectra were recorded using the ACF200 Broker Germany Spectrometer. Ultraviolet Spectra were recorded using Perkin-Elmer lab India UV-Vis Spectrometer. The Electron spin resonance spectra were recorded using the JES-FA Series and TG-DTA spectra were recorded using the SPTQ600 PA, Thermo gravimetric analyses of the metal complexes were carried out by using the Perkin Elmer system in thermal analysis centre Stick Cochin. Melting points were measured on a unimelt capillary melting point apparatus. All materials used in this investigation were purchased from Sigma/Aldrich and AR (Merck). Solvents used were of reagent grade and purified before use by the standard methods.

Preparation of the ligand and its metal complexes: (Preparation of Pramipexole and O-Hydroxy benzaldehyde Schiff base (OHBP))

Pramipexole 4.22g (0.02mole) and O- Hydroxy benzaldehyde 2.442g (0.02mole) were dissolved in 25ml of methanol were taken in 250ml borosil reflection flask and 1 ml of triethylamine .The mixture was refluxed for 3 hour on water bath and then cooled to room temperature, cream colored sharp needles were separated out and washed with methanol and dried in vacuum desiccators over CaCl2 anhydrous.

For the Preparation of Cu(II) and Ru (II) metal chloride salts were used. Dissolve 3.1532g(0.01 Mole)of newly synthesized ligand in adequate of methanol. To this solution, aqueous solution of 1.3434 g(0.01Mole) and1.718g (0.01Mole) metal chlorides, and 1 ml of Sodium acetate. The mixture was refluxed for 6hours in a water bath and then cooled to room temperature, light green colored, dark brown colored sharp needles were separated out. The coloured metal complexes were washed with water and then methanol, and were recrystalised from ether and dried in vacuum dessicator over CaCl2 anhydrous. The elemental analysis was carried out for the newly synthesized ligand metal complexes. The prepared metal complexes were in 1:2 ratio. Ligands and metal complexes analytical data was tabulated in Table-1.

Table-1: Analytical data of the ligand and their metal complexes

			Complex		
			OHBP	$Cu(OHBP)_2X_2$	$Ru(OHBP)_2X_2$
Molecular weight			315.324	730.188	767.17
Co lour			cream	Light green	Dark brown
Yield			73	72	74
M.P			160-162	182-184	190-192
Elemental Analysis	C %	Calculated	64.76	55.87	53.14
		Found	64.45	55.66	52.98
	H%	Calculated	6.66	6.29	5.99
		Found	6.06	6.12	5.44
	N %	Calculated	13.33	11.50	10.94
		Found	13.11	11.23	10.56
	O%	Calculated	5.079	8.764	8.342
		Found	4.999	8.223	8.001
	M%	Calculated	-	8.70	13.16
		Found	-	8.22	12.99

RESULTS AND DISCUSSION

Infrared spectral analysis

Infrared spectra were recorded with a JASCO FT/IR-5300 Spectrometer (4000-400cm^{-}1) using KBr pellets. By utilizing this spectroscopy, the presence of important functional groups in the compound can be identified. Table2 through light on the observation made in analyzing IR spectra of ligand and metal complexes. The typical IR spectra are presented in the Fig.1, 2 and 3.

Interpretation of OHBP and Cu(II) and Ru(II)complexes

The Infrared spectrum of the ligand was compared with the spectra of Cu(II) and Ru(II) complexes. The data was summarized in table along their assignment. The typical IR spectra were shown in Fig.1, 2 and 3. The IR spectrum of the ligand has shows broad band at 1648 cm^{-1} [[14], which was assigned to due υC=N stretching of azomethine group. In complexes this band was shifted to lower regions, 1621 cm^{-}1 and 1600 cm^{-1}[[15] for Cu(II) and Ru(II) complexes respectively, suggesting the involvement of azomethine group(>C=N) group in complexation. This was due to the reduction of electron density on Nitrogen. There by indicating the coordination of the metal in through the nitrogen atoms.

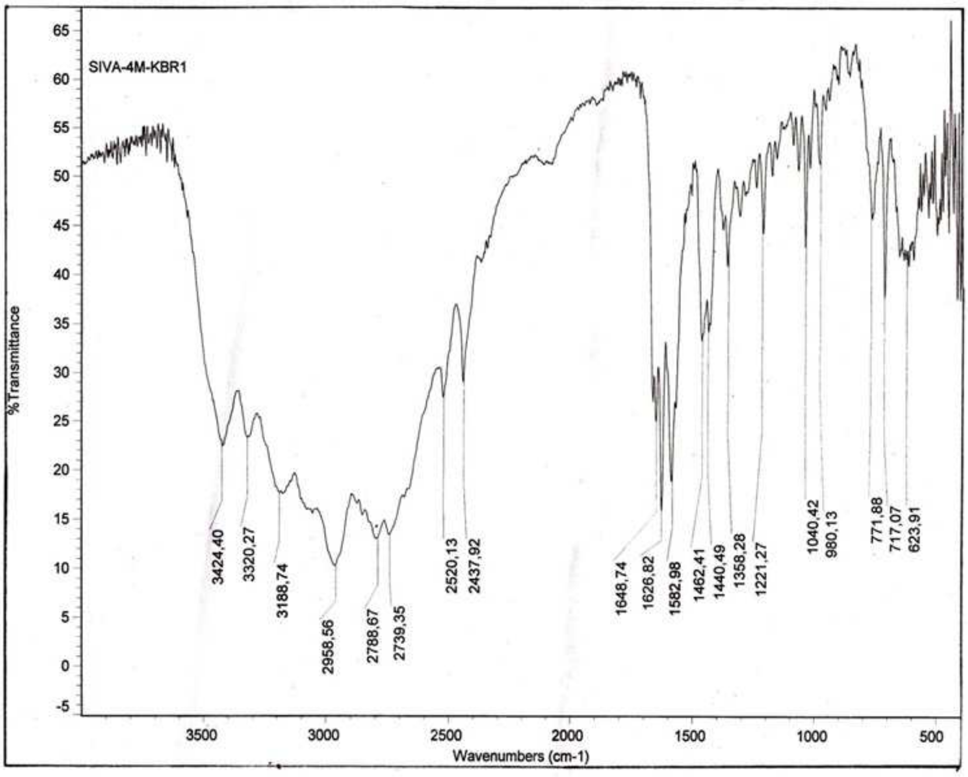
The IR spectra of metal chelates shows the disappearance of the υ(OH)[16] bond at 3424 cm^{-}1. It indicates the proton displacement from the Phenolic (OH) group on complexation. Thus bonding of the metal ions to the ligands under investigation takes place through a covalent link with oxygen of the Phenolic group. The IR spectra of Cu(II) and

Ru(II) metal complexes exhibit a broad band [17] around3440 cm^{-}1and 3419 cm^{-1} respectively, which can be assigned to υ(OH) of water molecules associated with complex formation. New bands were observed in the complexes, which were not observed in ligand. The bands at 744 cm^{-1} and 772 cm^{-1} were assigned to stretching frequencies of (M-O), the band at 480cm^{-}1and 490cm^{-1}[18] were assigned to the stretching frequencies (M-N) respectively[19-21].

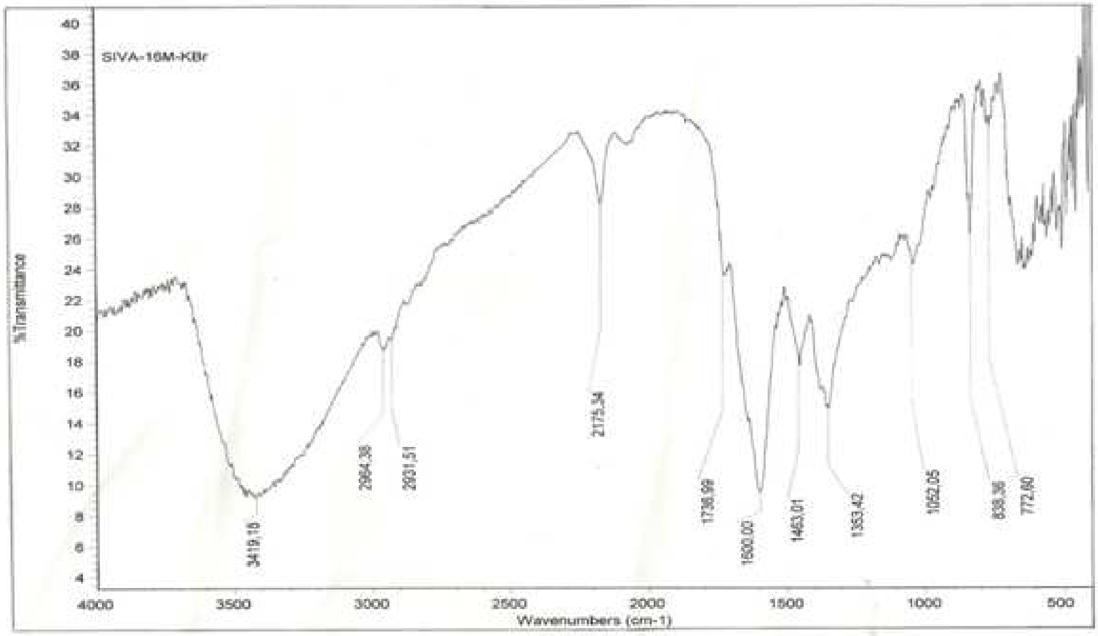
Table-2: The important IR bands of the Ligand and Their Metal Complexes

Compound	OH(Water)	υOH (Phenolic)	υ C=N	υ N-H	υ M-O	υ M-N	υ C-H
OHBP	-	3424	1648	3320	-	-	2958
Cu(OHBP)2	3440	-	1621	3347	744	480	2788
Ru(OHBP)$_2$	3419	-	1600	3322	772	490	2750

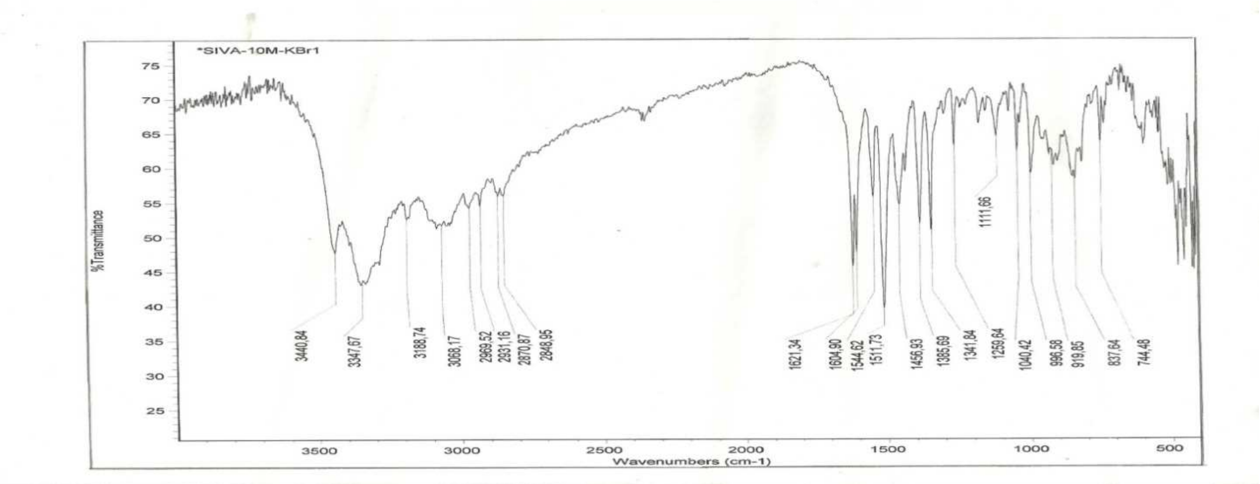
IR spectrum of OHBP ligand



IR spectrum of Cu(OHBP)2 complex



IR spectrum of Ru(OHBP)$_2$ complex



NMR Spectrum of OHBP Ligand and its Metal complexes

The ^{1}H NMR spectra of ligand and metal complexes in DMSO-d6 as solvent were given in fig,4and5.The chemical shift values of the ligand and metal complexes were shown in Table-3. Ligand showssingletat6.3ppm [21], which is due to protons bonded to Schiff base group. On complexation this band was shifted to low field regions 6.66ppm and 7.5ppm for Cu (II) and Ru (II) complexes respectively. This shift indicates the shielding of azomethine. The aromatic ring protons forms a multiplet at 6.37-6.41ppm, methelene protons forms a singlet in the region2.5ppm-1.70ppm hydroxyl proton [22] shows singlet at 5.71ppm, which was disappeared in the complexes.

In the 1H NMR spectrums of the Cu (II) and Ru (II) complexes the signal due to azomethine protons were shifted 6.3ppm to 6.66-7.5ppm respectively. This shifting indicates the deshielding of the azomethine group. The aromatic ring protons that are seen in the 7.2 - 7.3 ppm[22] become broad and less intense compared with the corresponding Schiff base. In complexes the aromatic ring protons at7.2-7.3ppm become broad and less intense, compared with Schiff base. The following complexation to the metal ion2.82ppm in the case of Cu(II) and Ru (II) complexes indicates the complexation of water molecules by coordination with metal ion.

Table-3: ^{1}H NMR Spectrum of the ligands and its metal complexes in DMSO-d6 in ppm

Compound	H-C=N	Ar-H	OH-H2O	CH2	Ar-OH	N-H
OHBP	6.3	6.37-7.65	-	2.5-1.70	5.71	8.53
Cu(OHBP) $_2$	6.66	6.9-7.32	4.717	2.82-292	-	9.56
Ru(OHBP) $_2$	7.5	6.41-7.0	4.699	1.14-2.85	-	8.35

Conductivity measurements

The molar conductance of complexes in DMF (~10^{-3} M) was determined at 27+20C using Systronic 303 direct reading conductivity bridge. A known amount of solid complexes is transferred into 25ml standard flask and dissolved in diethyl formamide (DMF). The contents are made up to the mark with DMF.

The complex solution is transferred into a clean and dry 100ml beaker. The molar conductances of the complexes were less than 20 Ohm^{-}1 cm2 mol^{-}1 indicating the Non-electrolytic nature. These values suggest non-electrolytic nature of the present complexes. The molar conductance values of these metal complexes are given in the Table 4.

Table-4: Conductance data for Metal-OHBP Complexes: Cell constant: 1.00

S. No.	Metal Complex	Conductance Ohm-^{1}	Specific Conductance Ohm^{-}1 cm^{-1}	Molar Conductance Ohm^{-}1 cm2 mol^{-1}
1.	Cu(OHBP)$_2$	0.00019 x 10^{-3}	0.00019 x 10^{-3}	19
2.	Ru(OHBP)$_2$	0.00020 x 10^{-3}	0.00020 x 10^{-3}	20

Electronic spectra

In UV-Visible electromagnetic radiation, the transitions are associated with the electronic energy levels of the compound under investigation. The electronic spectra were recorded on a Thermo Spectronic Heylosa spectrophotometer. The transition metal ions occur in a variety of structural environments. Because of this, the electronic structures are extremely varied. The electronic structures have been identified with UV-Visible spectroscopy OHBP and its metal complexes:

The electronic spectral of ligand and its metal complexes were given in the transitions were reported in the Table-5. Ligand shows signal band at 310 nm, assigned to ∏─∏* transition. In complexes this band was shifted to higher wavelength regions. New bands were observed in the complexes at corresponding to the charge transfer transitions. In high convention spectra of complexes d-d transitions were observed in visible region.

Table-5 : Electronic spectral data

Complexes	λmax of the complex in nm	λmax of the ligand in nm
Cu(OHBP) $_2$	326	310
Ru(OHBP) $_2$	337	310

Electronic spin resonance spectra

In the present study the X-band (~9.3GHz) ESR spectra of Cu(II) and Ru(II) complexes in DMF were recorded at room temperature and at liquid nitrogen temperature (LNT) on a JES-FA SERIES spectrometer. DPPH radical was used as a field maker.

Analysis of OHBP through ESR spectra of Cu (II) complex

The ESR spectra of the complex in poly crystalline state exhibit only one broad signal, which is attributed to dipolar broadening and enhanced spin lattice relaxation. Anisotropic spectra obtained for these complexes in DMF at LNT and representative ESR spectra of Cu (II) complexes were presented inFig.7.Inthislowtemperature spectrum, four peaks of small intensity have been identified which are considered to originate from $g_{\|}$ component. The spin Hamiltonian, orbital reduction and bonding parameters of the Cu(II) complex was presented in Table 6. The $g_{\|}$ and $g_{\perp}$ are computed from the spectrum using DPPH free radical as g marker. Kvelson & Neiman[23] have reported that $g_{\|}$ value is less than 2.3 for covalent character and is greater than2.3forioniccharacterofthe metal-ligand bond in complexes. Applying this criterion, the covalent bond character can be predicted to exist between the metal and the ligand complexes [24]. The trend $g_{\|}$>gave> $g_{\perp}$> 2.0023 observed for the complex suggest that the unpaired electron is localized in dx2- y2 and dz2 orbital of the cupper (II) ions for the complex. It is observed that G value for these complexes are greater than four and suggest that there are no interactions between metal-metal centers in DMF medium.

The ESR parameters $g_{\|}$, g┴, A‖*and A┴* of the complexes and the energies of d-d transitions are used to evaluate the orbital reduction parameters ($K_{\|}$,K┴) the bonding parameters (α^2), the dipolar interaction(P)[25]. The observed $K_{\|}$ <K┴ indicates the presence of out of plane Pi-bonding. The α^2 values for the present chelates lie in the range 0.42-0.48 and support the covalent nature of these complexes. Giordano and Bereman suggested the identification of bonding groups from the values of dipolar term P. The reduction of P values from the ion value (0.036cm-^{1}) might be attributable to the strong covalent bonding. The values of P obtained for the present complexes in between 0.029-0.036cm^{-1}and remain consistent with bonding of metal ions to oxygen and nitrogen donor atoms respectively. The shape of ESR lines, ESR data together with the electronic spectral data suggest an octahedral geometry for these complexes [26].

Table-6: Spin Hamiltonian and orbital reduction parameters of copper and Manganese complexes in DMF solution

Parameters	Cu(OHBP)$_2$
$g_{\|}$	2.04559
$g_{\perp}$	1.99000
gave	2.00853
G	3.8201
$A_{\|}$ *	0.0182
$A_{\perp}$*	0.0022
Aave *	0.0081
d-d	13500
$K_{\|}$	0.8891
$K_{\perp}$	0.9824
P*	0.036
α 2	0.420

Magnetic susceptibility measurements of copper (II) and Ruthenium (II) complexes

The effective magnetic moment values for all the complexes are represented in the Table.7.Thereare considerable orbital contribution and effective magnetic moments for octahedral complex at room temperature. The magnetic moments of the present (OHBP)$_2$ Cu complex is 4.82 B.M. and the value is less than the spin only value, it shows

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reduced Para magnetism, which suggest the formation of low-spin complex having octahedral geometry. The magnetic moments of the present $(OHBP)_2$ Ru complex is 1.79 B.M. and this value is less than the spin-only value, showing reduced paramagnetism, which suggest the formation of flow-spin complex having square planar.

Table-7: Magnetic moments of copper and Ruthenium

S. No.	Metal Complexes	Effect. In B.M.		Number of unpaired electron
		Theoretical	Observed	
1.	$Cu(OHBP)_2$	4.90	4.82	4
2.	$Ru(OHBP)_2$	1.68	1.79	1

Thermal analysis

The thermal studies of these complexes are carried out to know the stability of the complexes on thermal decomposition, as well as to know the different final products that are obtained in thermal decomposition having novel catalytic properties.

Study of OHBP and its Cu (II) and Ru (II)metal complexes by TGA-DTA spectra

Thermo analytical data of metal complexes were given in the Table.8. The representative thermo grams were shown in the fig.8 and 9. The Cu complexes are thermally stable up to 1000^0C. The first stage of the decomposition corresponding to endothermic dehydration of the complex and the two lattice water molecules are lost in the temperature range 120-140^0C to give anhydrous complex[28].The second decomposition stage with two endothermic is known as stable intermediate formed around 380-440 ^{0}C[29-30].Exothermic decomposition express to give the corresponding metal oxides as final decomposition product at a high temperature i.e. above 600^0C .The decomposition behaviour of the complexes was observed in nitrogen atmosphere. All the experimental mass loss has shown Table.8. The Ru complexes are thermally stable up to 1000^0C.

The first stage of the decomposition corresponding to exothermic dehydration of the complex and the two lattice water molecules are lost in the temperature range 220-260^0C to give anhydrous complexes. The second decomposition stage with two exothermic is known as stable intermediate formed between 270-440^0C. Exothermic decomposition express to give the corresponding metal oxides as final decomposition product at a high temperature i.e. above 520^0C. The decomposition behaviour of the complexes was observed in nitrogen atmosphere. All the experimental mass loss has shown Table.8. At high temperatures, the corresponding metal oxides were formed, as stable products. All the experimental percentage mass loss was compared with the calculated weights. Based on thermal data it was shown that the stability order of the complexes was Cu (II)> Ru (II).

Table 8 : Thermal analytical data of the Ligand and their metal complexes

Complex X=H2O	Molecular weight in gms	Weight of the complex take in mgs	Temperature Range during weight loss in 0C	%of fraction of weight	Probable assignment
[Cu.L2.X2] L= $C_{17}H_{21}N_3SO$	730.18	8.1910	120-140 380-440 Above 600	15.64 64.53 10.77	Loss of 2H2O molecule. Loss of two L molecules. Remaining residue Corresponds to CuO.
[Ru.L2.X2] L= $C_{17}H_{21}N_3SO$	767.17	12.1630	220-260 270-440 Above 650	11.33 52.81 7.55	Loss of 2H2O molecule. Loss of two L molecules. Remaining residue Corresponds to RuO.

Biological activity

The author in this present investigation attempted to find out antibacterial activity of ligand and their metal complexes against Salmonella typhi, Enterococcus faecails and Escherichia coli choosing serial paper disc method Table 9.The results of the biological activity of the metal complexes indicated the following facts. A comparative study of the ligand and their complexes indicates that the metal chelates exhibited higher antibacterial activity than that of the free ligand. The increase in the antibacterial activity of metalchelates was found due to the effect of metal ion on the metal chelates which could be explained on the basis of overtones concept and chelation theory. On chelation the polarity of the metal ion reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charges of metal ion with donor groups. It was further noted that the delocalization of electrons over the whole chelate ring enhanced the lipophillicity of the complexes. This increase dlipophillicity [31] hanced the penetration of the complexes into lipid membrane and blocking the metal sites on enzymes of microorganism.

The zones of inhibition of the ligand metal complexes were in the Table.9. The activity was compared with zone of inhibition was measured in mm and reported in of Cu (II) and Ru(II) Complexes of Schiff, is found to be more.

Table 9: Antibacterial Activity of the Metal complexes Total Area of Zone of clearance in mm

S.No.	Compound	*Salmonella Typhi*	*Enterococcus Faecails*	*Escherichia coli*
1	OHBP	10	12	15
2	$Cu(OHBP)_2$	12	18	18
3	$Ru(OHBP)_2$	14	16	17

CONCLUSION

The outcome of the above results confirms the stoichiometry of the complexes to be 1 : 2 [M: L] as indicated by elemental analysis and conductometric measurements. IR spectra suggest that the ligand behaves as bidentate and coordinates to the central metal ion through azomethine nitrogen and phenolic –OH group. This has been further confirmed on the basis of NMR spectral studies. Thus, on the basis of above physicochemical and spectral studies the complexes are found to have higher biological activities as compared to the respective ligand and the parent drug that, somehow, justifies the purpose of the research work. The present work will be further extended to the synthesis of metal complexes using other biologically active metals and evaluation of their biological activities. All the metal complexes carry on charge and are thermally stable. As such no single technique is independent of predicting final structures of the complexes.

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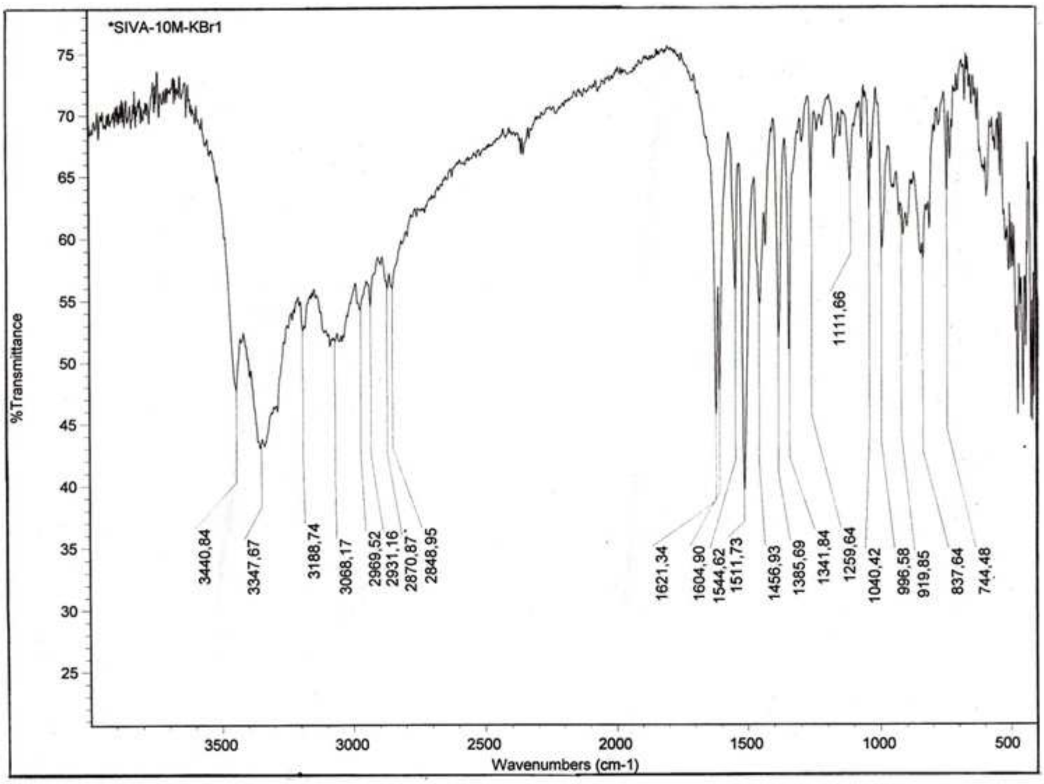
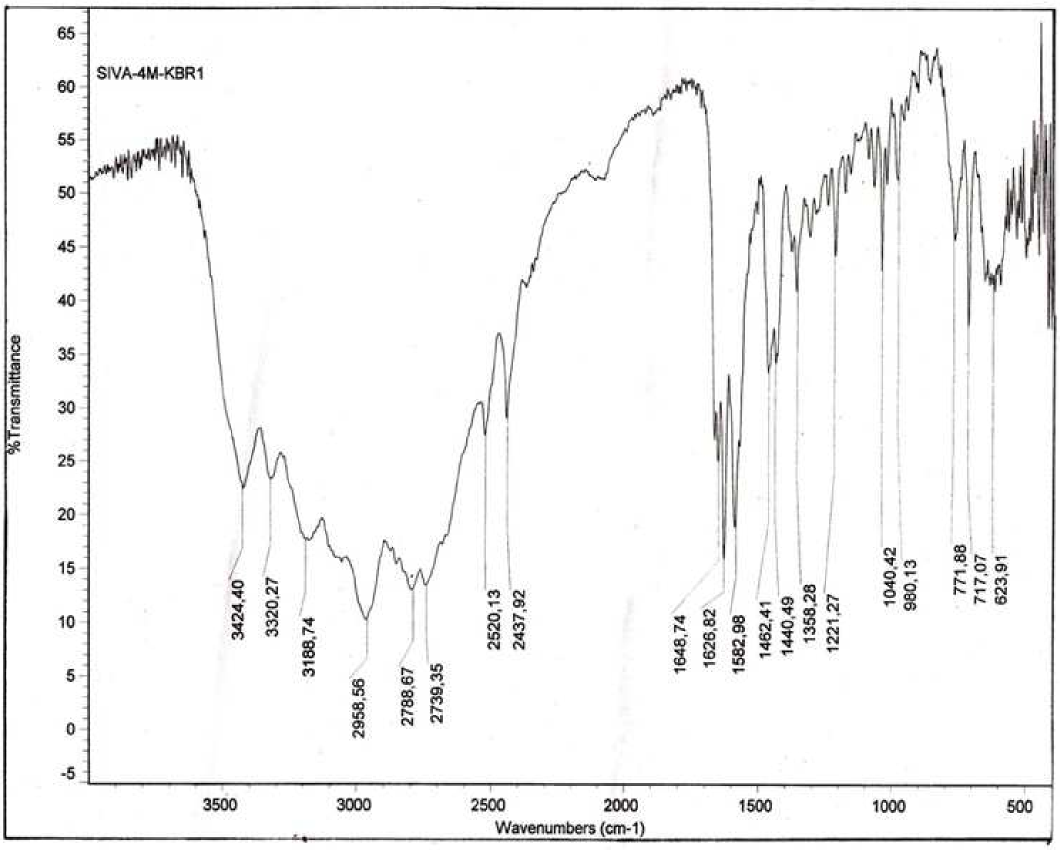
IR spectra of cu -OHBP complex



IR Spectra of OHBP ligand



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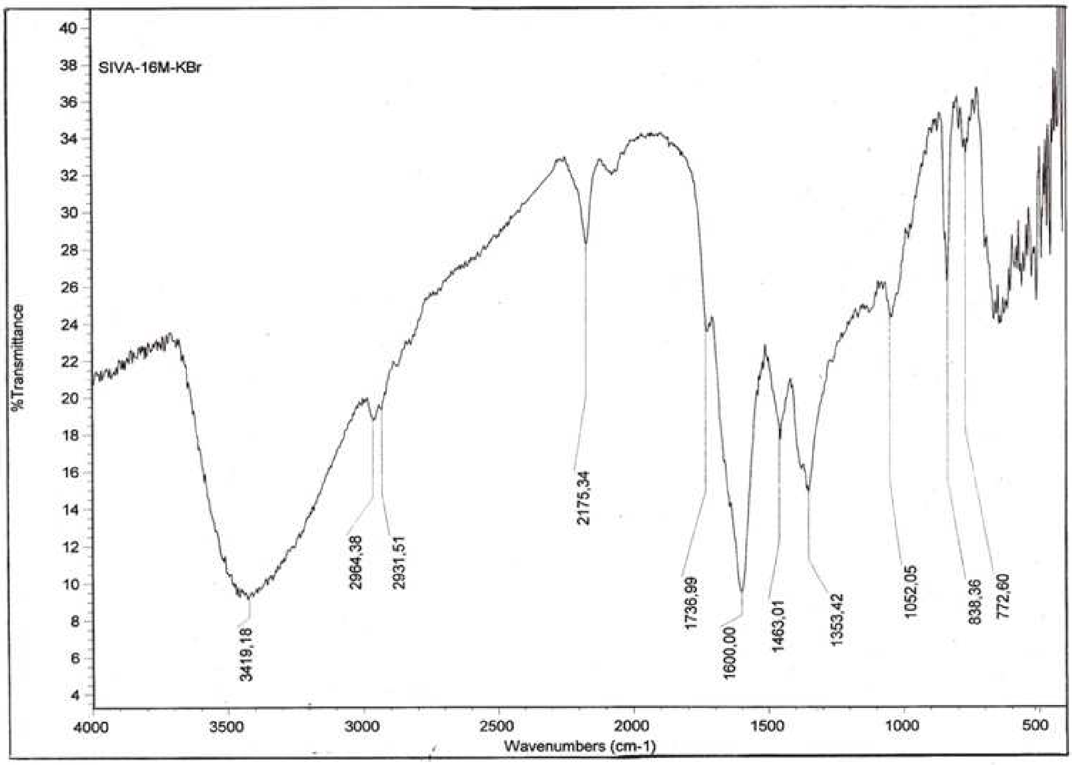

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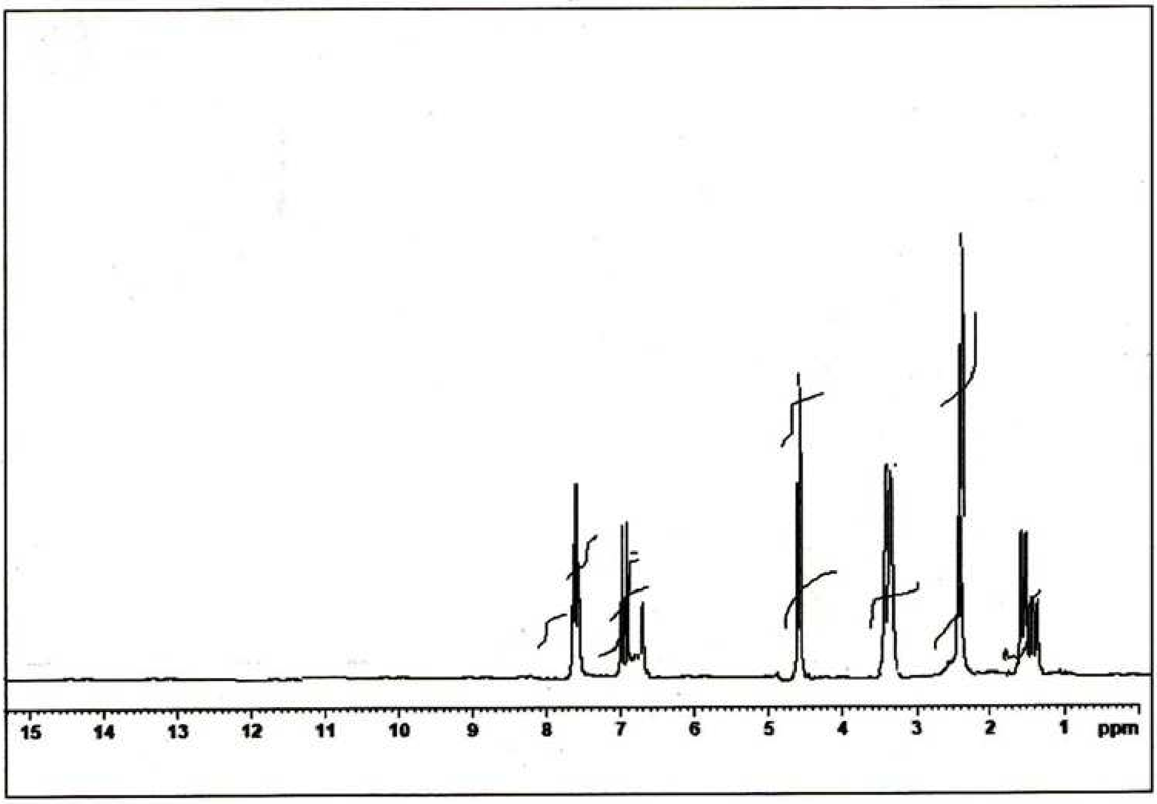
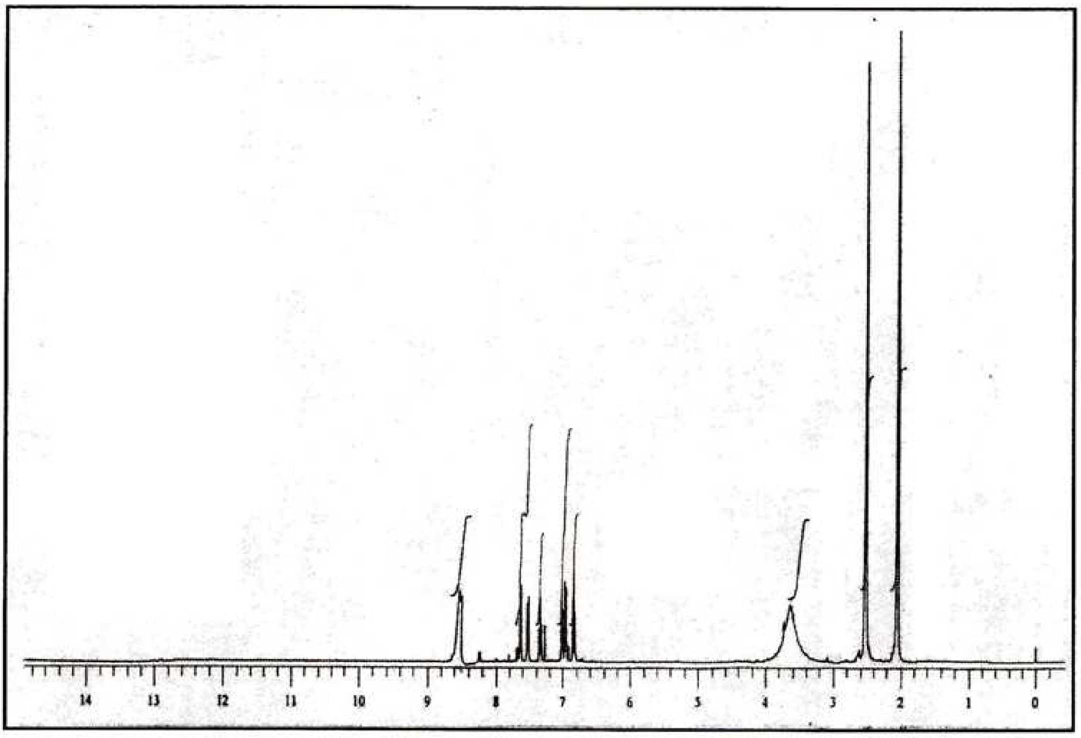
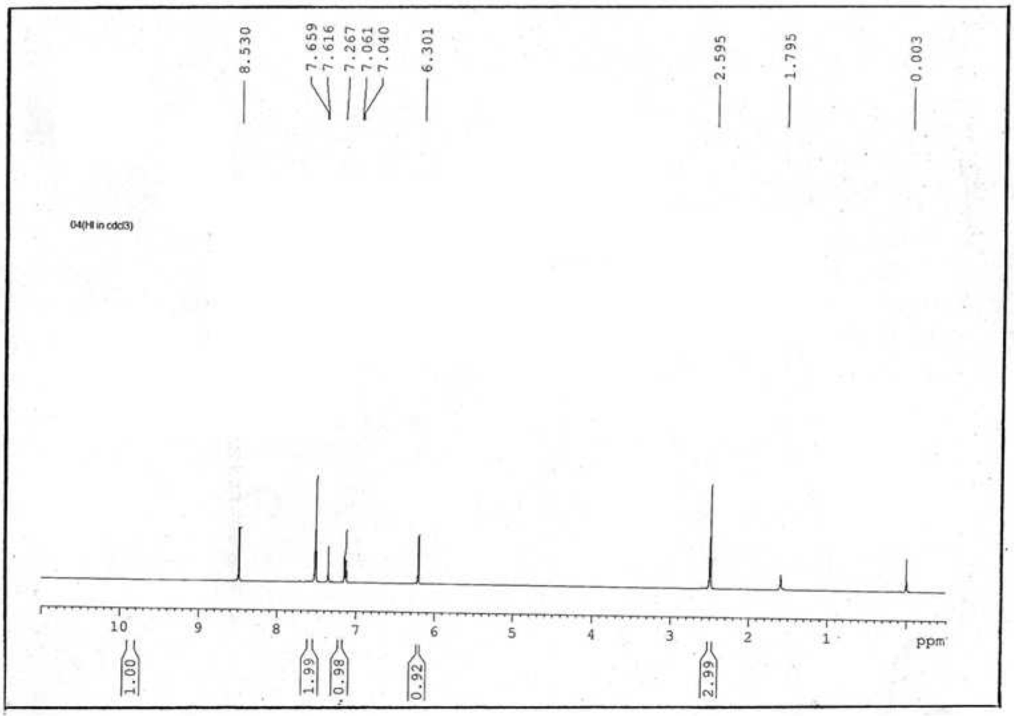


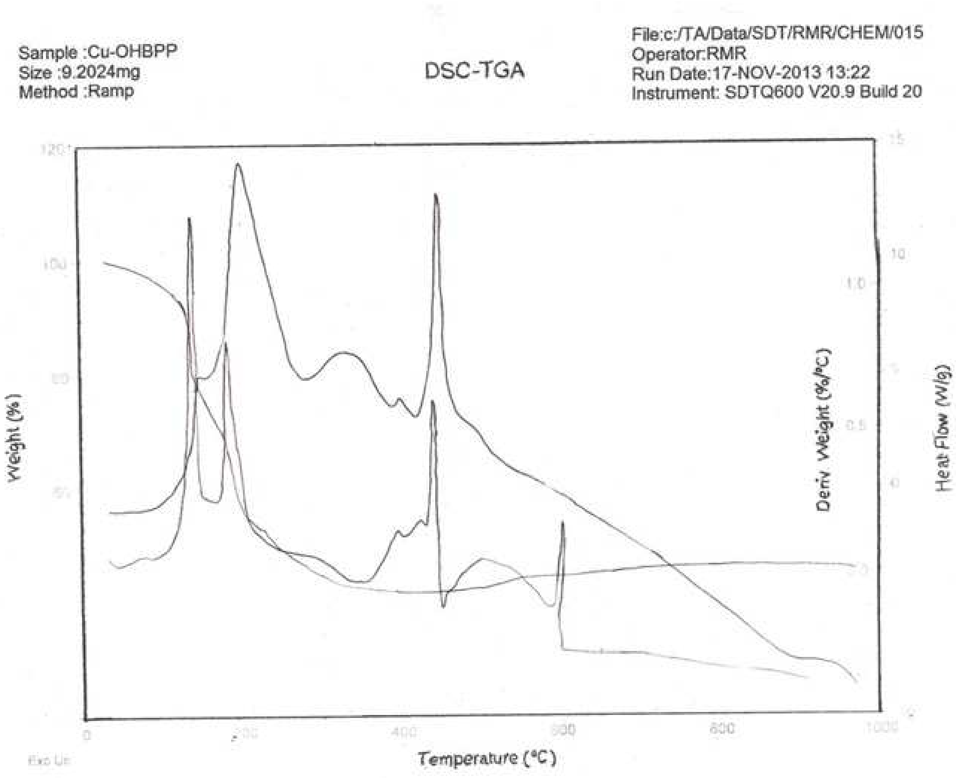
NMR spectra of Cu- OHBP complex



NMR spectra of OHBP Ligand



TG-DTA of OHBP Cu complex



TG-DTA of OHBP Ru complex