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## Synthesis, characterization and biological activity of a novel p-toulic hydrazone and resacetophenone schiff base (RAPPTH) ligand and their metal complexes

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### ABSTRACT

A novel Schiff base like RAPPTH ligand ( p-Toulic Hydrazone and resacetophenone ) and their Mn (II), Co (II), Ni (II) and Cu (II) metal complexes have been prepared and characterized by various physio chemical methods like FT-IR, <sup>1</sup>H-NMR, UV-Visible, ESR, VSM, Molar conductance, XRD and micro analytical data and found to be biological activity. The magnetic properties of these Mn (II), Co (II), Ni (II) and Cu (II) complexes are 5.28 B.M. 4.82 B.M., 3.15 B.M and 1.81 B.M respectively. This result clearly shows that the metal complexes of this ligand show octahedral geometry. The IR spectrums of these Schiff base metal complexes gives bands at 1596cm<sup>-1</sup>, 1585cm<sup>-1</sup> 1606cm<sup>-1</sup> and 1609cm<sup>-1</sup> respectively, which is assigned to (C=N) stretching vibrations, a fundamental feature of azomethine group. The structural assessment of these metal complexes has been carried out based on above physio-chemical and spectroscopic methods. From the elemental analyses data, 1:2 metal complexes are formed.

**Keywords:** Schiff base, p-Toluic hydrazone, Resacetophenone, Anti bacterial activity and characterization.

### INTRODUCTION

The Schiff bases are play an important role in chemistry. The transition metal complexes of Schiff bases are very fascinating because of the presence of both hard nitrogen and oxygen and soft sulphur donor atoms in the backbones of these ligands, some of these complexes have been exhibit remarkable physiochemical properties and potentially useful biological activities.[1-2] Schiff base have also been used for analytical purposes in the determination of metal ions, and some Schiff base derivatives have been used in the extraction of metal solvents. The applications of such complexes depend to a great extent on their molecular structure. Schiff base ligands are able to synchronize different metals, and to stabilize them in various oxidation [3-6] states. The Schiff base complexes have been used [7-8] in catalytic reactions and as models for biological systems. During the last 20 years extensive awareness on the metal complexes of Schiff bases [9-10] containing nitrogen and other donors. This may be endorsed to their stability, biological activity and huge applications [11] in many fields such as oxidation catalysis, electrochemistry and biological studies. Transition metal complexes of Schiff bases derived from benzil with different amines had

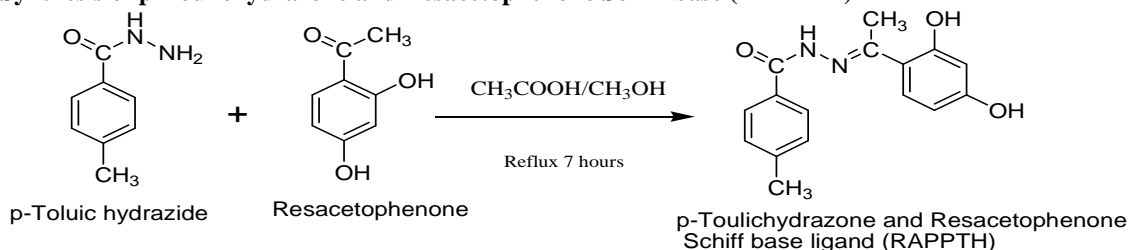
been prepared and characterized by physical and chemical techniques. Schiff bases derived from condensation of various aldehydes with triethylene tetra amine and their complexes were reported.

The present paper deals with a series of metal complexes of Mn(II), Co(II), Ni(II) and Cu(II) with Schiff base ligand (RAPPTH) derived from P-Toluic hydrazide and Resacetophenone (2,4 Dihydroxy acetophenone). These complexes were characterized by elemental analysis, IR, NMR, UV, ESR spectroscopy, TG-DTA, Powder X-Rd, VSM and Conductivity measurements to verify the mode of bonding, geometry and biological activities of the metal complexes were also studied. These metal complexes are shows the octahedral structure.

## MATERIALS AND METHODS

### 2. Experimental

#### 2.1 Synthesis of p-Toulichydrazone and Resacetophenone Schiff base (RAPPTH)

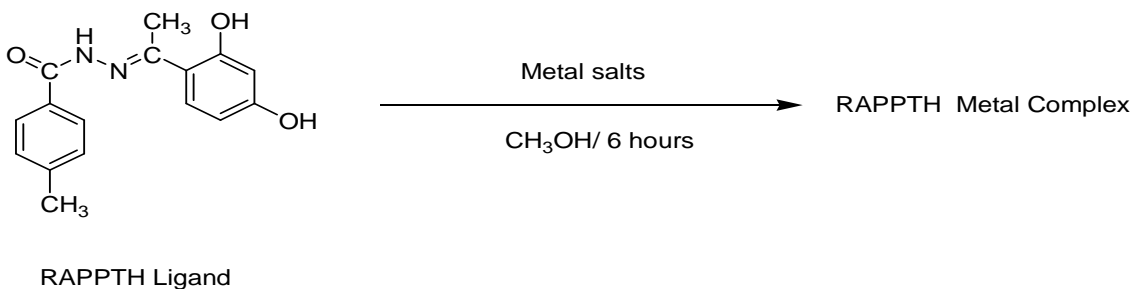


Scheme 1: Synthesis of RAPPTH Ligand

p-Toluic hydrazide (1.5018g) and Resacetophenone (2,4Dihydroxyacetophenone)(1.5215g) were dissolved in methanol and Refluxed in the presence of few drops of acetic acid for 7 hours on water bath with constant stirring. The resultant mixture was transferred in a china dish and allowed to cool naturally. Light brown crystals were obtained. The compound was recrystallized from water. The % of yield was 80% and melting point of the compound was 218-220<sup>0</sup>C.

#### 2.2 Synthesis of Mn (II), Co (II), Ni (II) and Cu (II) metal complexes of p-Toluic hydrazone and Resacetophenone Schiff base (RAPPTH):

The reagent p-Toluic hydrazone and Resacetophenone(2,4dihydroxyacetophenone) Schiff base was dissolved in 40ml of 50% methanol and Mn (II), Co (II), Ni (II) and Cu (II) metal ions dissolved in 20ml of distilled water, this compounds mixture was stirred for 6 hours in the presence of sodium acetate. Thick Green, Brown, Light yellow and Green colour metal complexes were formed respectively with good yield. These products were washed several times with hot water and cold methanol. Metal complexes are free from un reacted metal slats and ligand respectively and finally dried in vacuo over calcium chloride dessicator.



Scheme 2: Synthesis of RAPPTH Metal Complexes

## RESULTS AND DISCUSSION

### 3.1 IR & NMR Spectral Studies

IR & NMR Spectral data of free RAPPTH ligand and its metal complexes are shown in Fig 1-6. In <sup>1</sup>H NMR spectrum a signal is observed at 11.10ppm due to N-H proton in the ligand is shifted to δ 11.25 and 11.29ppm respectively for RAPPTH-Co and RAPPTH-Cu metal complexes.

A broad band exhibited at  $3342\text{ cm}^{-1}$  in the IR spectrum of the ligand due to N-H stretching vibration. On complexation this band shifted to  $3347$ ,  $3349$ ,  $3350$ , and  $3352\text{ cm}^{-1}$  for Mn (II), Co (II), Ni (II) and Cu (II) complexes respectively. The IR spectrum of the ligand has shown a sharp band at  $1698\text{ cm}^{-1}$  due to C=O stretching vibration. On complexation C = O stretching vibration band shifted to  $1671$ ,  $1666$ ,  $1658$ , and  $1662\text{ cm}^{-1}$  for Mn (II), Co (II), Ni (II) and Cu (II) Complexes respectively. These results are indicating the formation of complex. The IR spectra of Mn (II), Co (II), Ni (II) and Cu (II) complexes exhibited broad bands at  $3430\text{ cm}^{-1}$ ,  $3480\text{ cm}^{-1}$ ,  $3415\text{ cm}^{-1}$  and  $3420\text{ cm}^{-1}$  respectively, which can be assigned to the OH stretching vibration of the coordinated [12-14] water molecules. These results are clearly indicate that the ligand coordinate with the metal ion through the azomethine nitrogen and the oxygen of the deprotonated hydroxyl group.

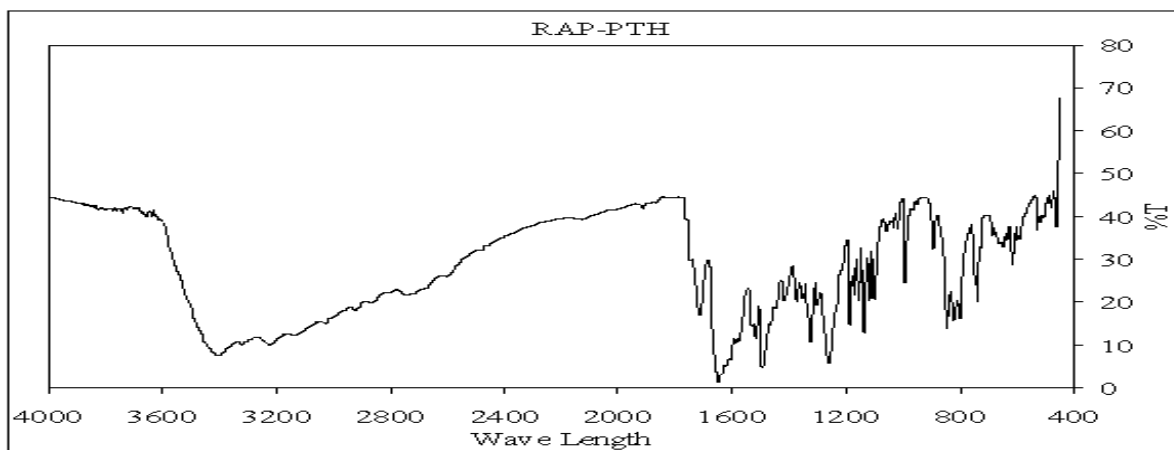


Fig 1: IR Spectrum of RAPPTH Ligand

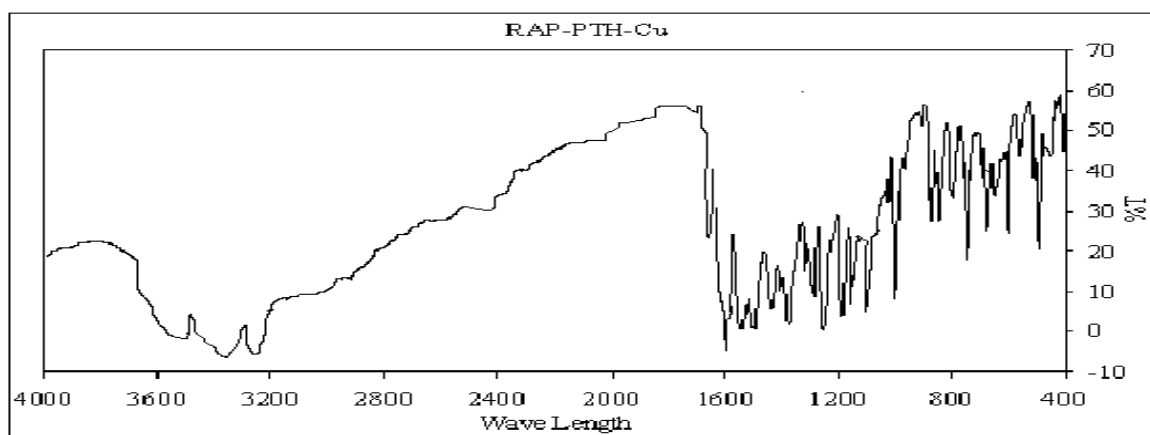


Fig 2: IR Spectrum of RAPPTH-Co Metal Complex

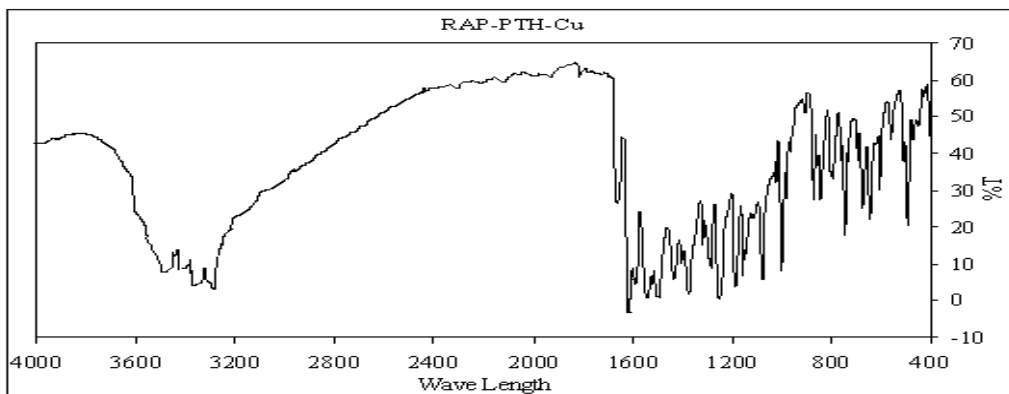


Fig.3. IR Spectrum of RAPPTH- Cu (II) Complex

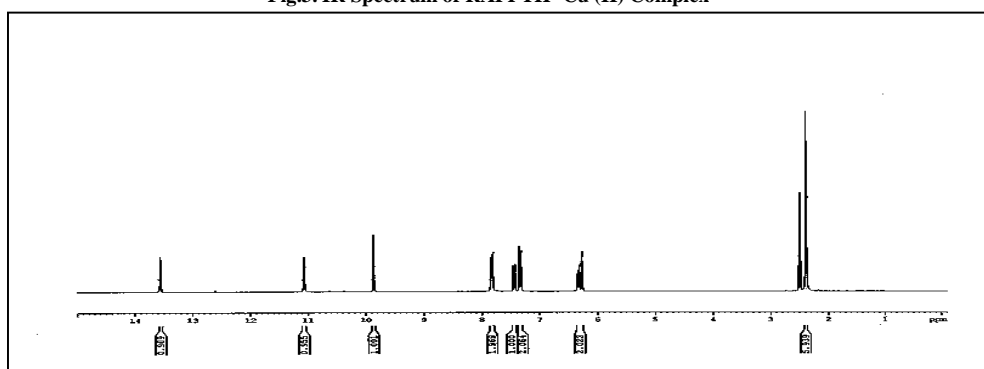


Fig 4 : <sup>1</sup>H NMR Spectrum of RAPPTH Ligand

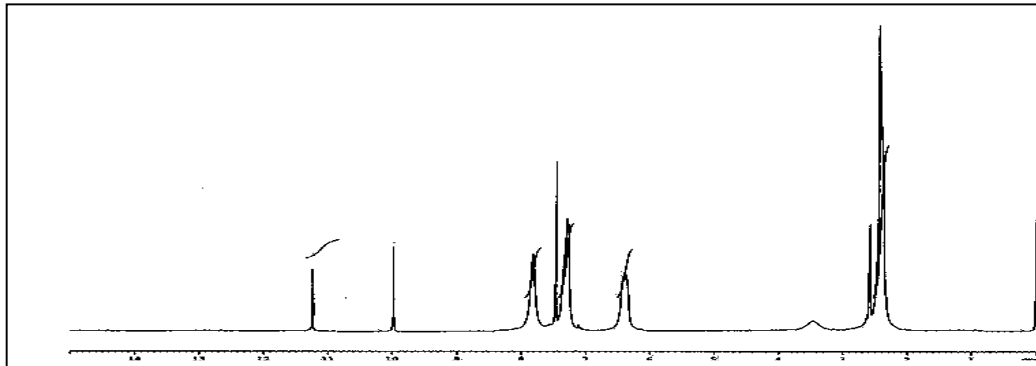


Fig 5: <sup>1</sup>H NMR Spectrum of RAPPTH-Co Complex

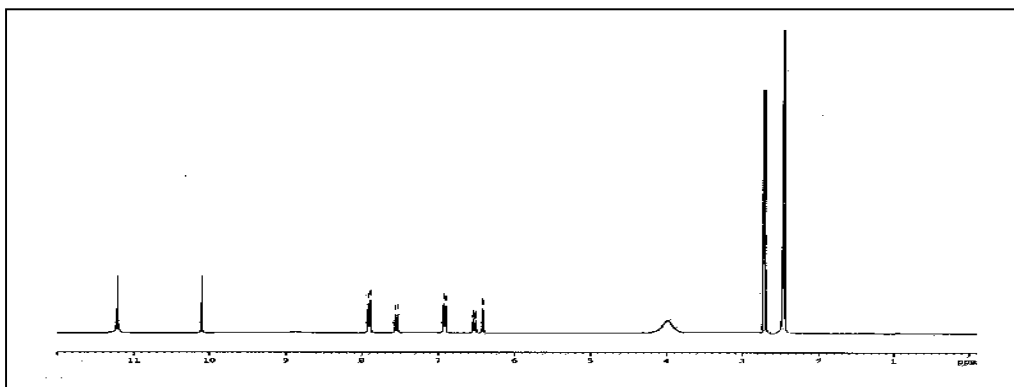


Table 1: Thermal Analytical Data of the RAPPTH metal complexes

Complex X=H <sub>2</sub> O	M.Wt in grams	Wt of the complex in mgs	Stage	Temp in range °C	Probable assignment	Mass loss (%)	Total loss (%)
MnL <sub>2</sub> 2X L=C <sub>16</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub>	657.57	8.0	1 2 3	120.12-221.90 284.46-575.03 Above 640.47	Loss of 2H <sub>2</sub> O molecule Loss of 2L molecule Corresponds to MnO	5.474 83.541 9.864	89.0
CoL <sub>2</sub> 2X L=C <sub>16</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub>	661.560	7.50	1 2 2	130.41-230.23 320.23-509.48 Above 600	Loss of 2H <sub>2</sub> O molecule Loss of 2L molecule Corresponds to CoO	5.44 85.583 9.877	91.0
NiL <sub>2</sub> 2X L=C <sub>16</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub>	661.269	8.00	1 2 3	145-230 290-720 Above 792	Loss of 2H <sub>2</sub> O molecule Loss of 2L molecule Corresponds to NiO	5.44 83.218 11.14	88.6
CuL <sub>2</sub> 2X L=C <sub>16</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub>	666.086	8.00	1 2 3	120-230 298-793 Above 839	Loss of 2H <sub>2</sub> O molecule Loss of 2L molecule Corresponds to CuO	5.404 78.816 15.64	84.2

Table 2. Molar conductivity of RAPPTH -Metal complexes:

Metal complexes	Molar conductance(ohm <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup> )
RAPPTH-Mn	6.50
RAPPTH-Co	5.00
RAPPTH-Ni	7.20
RAPPTH-Cu	4.80

Table 3. Analytical data of the RAPPTH ligands and its metal complexes

Molecular Formula X=H <sub>2</sub> O	Molecular Weight	Colour	Yield in %	Melting Point in °C	Elemental analysis							
					Carbon %		Hydrogen %		Nitrogen %		Metal %	
					Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found
L=C <sub>16</sub> H <sub>16</sub> O <sub>3</sub> N <sub>2</sub> RAPPTH	284.315	Light brown	84	218-220	67.53	67.12	5.62	5.40	9.84	9.47	-	-
[Mn L <sub>2</sub> ] <sub>2</sub> X (RAPPTH-Mn)	651.57	Thick green	72	318-340	58.39	58.04	5.17	4.67	8.52	8.13	8.35	8.01
[Co L <sub>2</sub> ] <sub>2</sub> X (RAPPTH-Co)	661.560	Light Yellow	81	315-322	58.04	57.64	5.14	4.82	8.46	8.07	8.90	8.73
[Ni L <sub>2</sub> ] <sub>2</sub> X (RAPPTH-Ni)	661.269	Brown	69	298-310	58.07	57.66	5.14	4.82	8.47	7.98	8.86	8.29
[Cu L <sub>2</sub> ] <sub>2</sub> X (RAPPTH-Cu)	66.086	Green	78	312-320	57.65	57.42	5.10	4.91	8.40	8.19	9.52	9.09

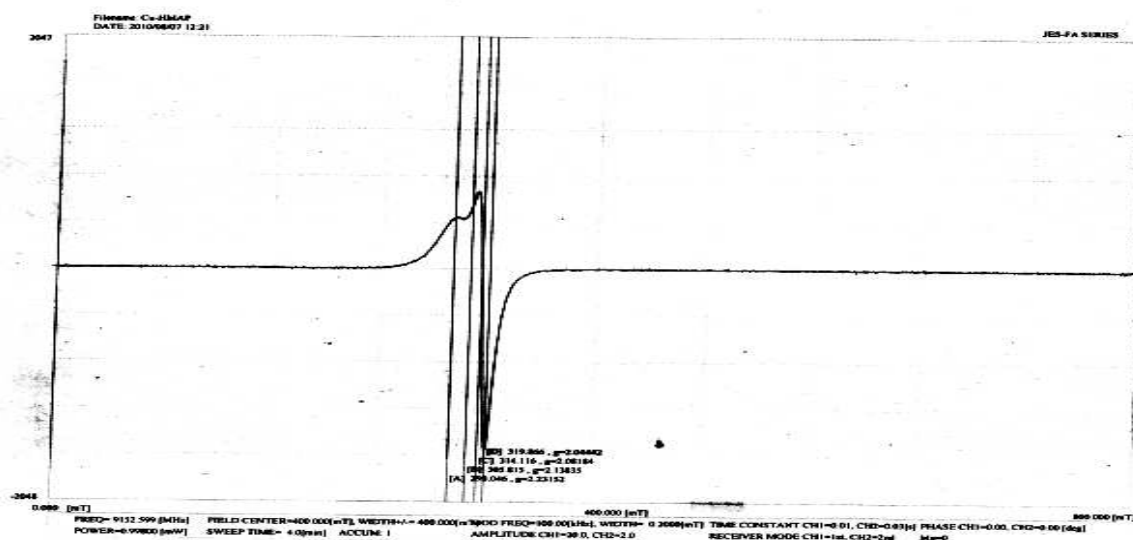
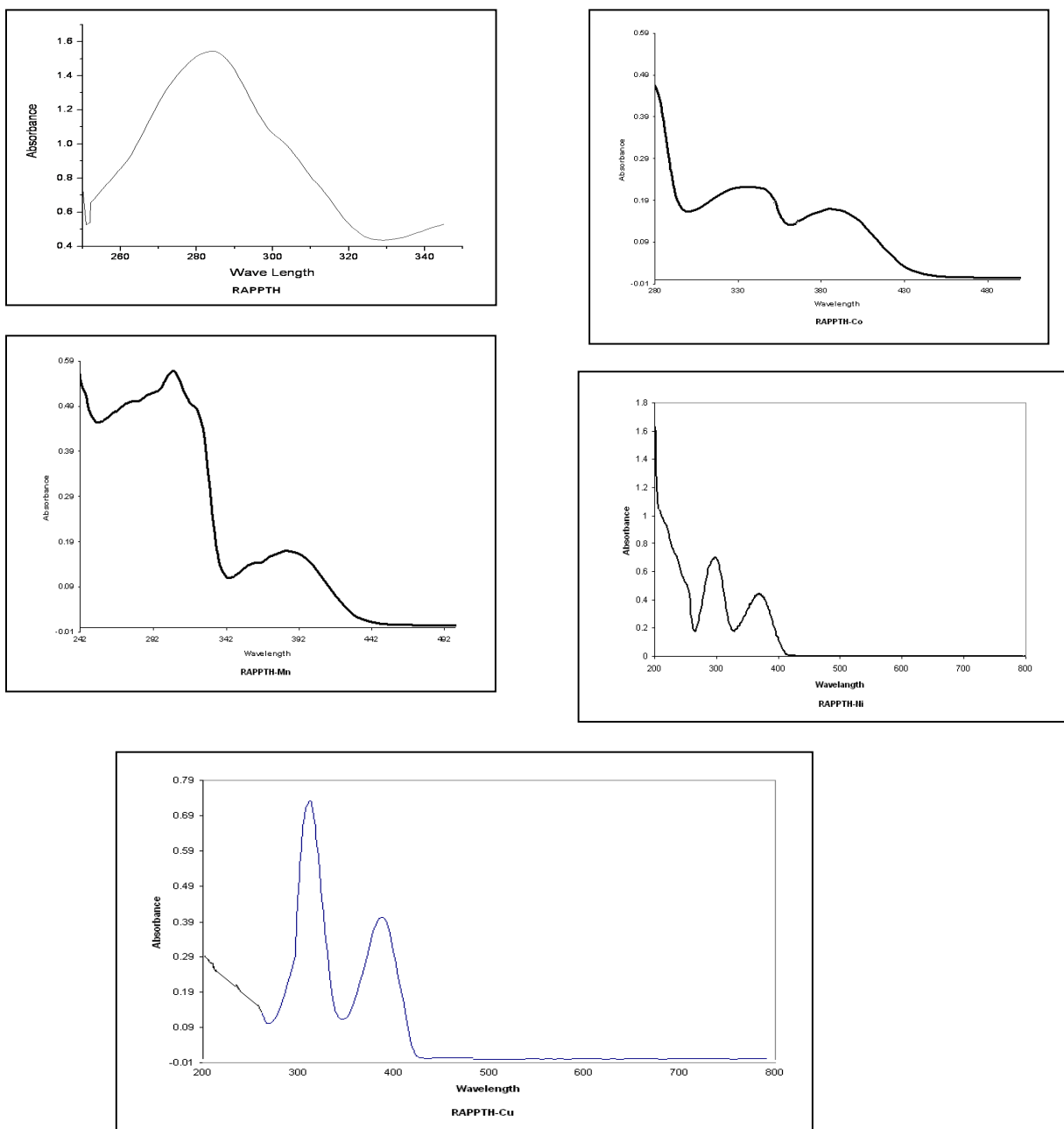


Fig 7: ESR Spectrum of RAPPTH-Cu (II) Complex



**Fig 9: Electronic Spectral data of RAPPTH ligand and its Metal Complexes**

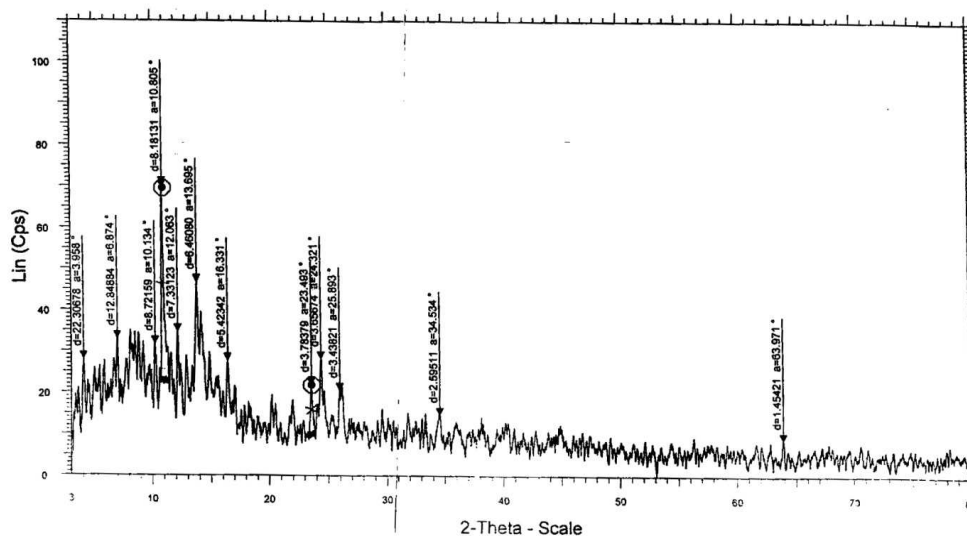


Fig 8: RAPPTH-Cu (II) Complex

### 3.2 UV Spectral & Magnetic measurements studies

UV spectrum shows the electronic transition for the ligand at 284 nm. On complexation of ligand with the different metal ions like Mn (II), Co (II), Ni (II) and Cu (II) new bands are appeared at 385 nm, 388 nm, 372 nm and 391 nm for different metal ions like Mn (II), Co (II), Ni (II) and Cu (II) corresponding to the transitional charge transfer from the ligand to the different metal ions. Bands are appeared in the region of 370-391 nm for all metal complexes [15-16], which are assigned to charge transfer transition (L→M). Based on the results octahedral structure is proposed for Mn (II), Co (II), Ni (II) and Cu (II) complexes. The magnetic moments of the RAPPTH Mn (II), Co (II), Ni (II) and Cu (II) complexes are 5.28, 4.82, 3.15 and 1.81BM respectively. Octahedral geometry is proposed [17] for Mn (II), Co (II), Ni (II) and Cu (II) complexes by Magnetic measurements & UV Spectral studies.

### 3.3 Thermal behavior of Mn (II), Co (II), Ni (II) and Cu (II) Metal complexes of RAPPTH

The Thermo gravimetric studies of all the complexes were carried out in air at a heating rate of 10°C per minute. The thermo analytical data is summarized in Table.1. The thermal decomposition of the complexes proceeds in three stages. The Mn (II), Co (II), Ni (II) and Cu (II) complexes are thermally stable up to 120, 130, 145 and 120 respectively [18-19]. The first stage of decomposition to endothermic dehydration of complexes by the loss of two water molecules occur in the temperature range 120-221°C, 130-230°C, 145-230°C and 120-230°C respectively. The intermediates formed are stable up to 230, 284, 320, 290 and 300°C. The second decomposition with exothermic peak by the loss of ligand moiety occurs in the temperature range 290-575°C, 320-510°C, 290-722°C and 300-793°C. The solid residues above 664, 530, 650 and 620°C were recognized as Mn, Co, Ni and Cu metal oxides respectively. In all the complexes [20] the final products are metal oxides.

### 3.4 Conductivity Measurements of RAPPTH metal complexes

The p-Toluic hydrazone and Resacetophenone (RAPPTH) metal complexes are freely soluble in dimethyl formamide (DMF). Perform conductivity measurements by using DMF. The solid metal complexes were transferred into 25 ml standard flask and dissolved in DMF and made up to the mark. This complex solution was pour into a clean and dry 100 ml beaker and measures the molar conductance values. The molar conductance values are 6.50, 5.00, 7.20 and 4.80 respectively for RAPPTH -Mn (II), Co (II), Ni (II) and Cu (II) metal complexes [21]. These values are predictable non-electrolytic nature for these complexes.

### 3.5 ESR Spectral studies of RAPPTH-Cu complex

ESR spectra of RAPPTH-Cu (II) ion complex is presented in Fig.7. and data is presented in table.4. The ESR anisotropic spectrum obtained for this metal complex in DMF at LNT..

In the low temperature spectrum, four small intensity peaks are identified which are considered to originate from  $g_{\parallel}$  component. The G value of the present complex is greater than four and suggests that there is no interaction between Copper-Copper centers in DMF medium. The ESR spectroscopic parameters  $g_{\parallel}$ ,  $g_{\perp}$ ,  $A_{\parallel}^*$  and  $A_{\perp}^*$  of the complexes and the energies of d-d transitions which are used to evaluate the orbital reduction parameters ( $K_{\parallel}$ ,  $K_{\perp}$ ), the bonding parameters ( $\alpha^2$ ), the dipolar interaction (P). The observed  $K_{\parallel} < K_{\perp}$  indicates the presence of out of plane  $\pi$ -bonding. The  $\alpha^2$  value for the present chelate is 0.524. It indicates that the complex has covalent character. This shows a significant covalency in the inplane  $\sigma$  bonding. The reduction of P values from the free ion value ( $0.036\text{cm}^{-1}$ ) might be attributable to the strong covalent bonding. The value of P obtained for the present complex is  $0.0159\text{cm}^{-1}$  and remain constant with bonding of Copper ions to oxygen and nitrogen donor atoms respectively. The shape of ESR lines, ESR data together with the electronic spectral data proposed [22-24] an octahedral geometry for RAPPTH-Cu complex.

Table.4. ESR spectral data of RAPPTH-Cu complex

Parameters	$g_{\parallel}$	$g_{\perp}$	$g_{ave}$	G	$A_{\parallel}^*$	$A_{\perp}^*$	$A_{ave}^*$	d-d	K <sub>  </sub>	K <sub>⊥</sub>	P <sup>*</sup>	$\alpha^2$
RAPPTH-Cu	2.252	2.063	2.126	4.006	0.0198	0.0035	.0089	1622	0.616	0.742	0.0159	0.524

Table 5. X-ray Diffraction data of RAPPTH-Cu complex

S.No.	d expt	d Calc	2 $\theta$ expt	2 $\theta$ Calc	h k l
1	22.3067	22.3015	3.9577	3.9263	1 0 0
2	12.8488	12.8239	6.8737	6.8526	2 0 0
3	8.7215	8.6959	10.1337	10.1257	3 3 0
4	8.1831	8.1734	10.8049	10.8026	2 2 1
5	7.3312	7.3215	12.0621	12.0517	3 2 2
6	6.4608	6.4579	13.6941	13.6927	4 3 0
7	5.4234	5.4198	16.3304	16.3298	4 4 2
8	3.7839	3.7697	23.4919	23.3926	6 5 3
9	3.6567	6.6478	24.3197	24.3086	7 5 2
10	3.4382	3.4297	25.8916	25.8912	7 6 1
11	2.5951	2.5902	34.5335	34.5296	9 6 5
12	1.4542	1.4523	63.9676	63.9521	9 9 8

### 3.6 Powder XRD study of RAPPTH-Cu complex

The diffractogram (12 diffractions) reflects Fig. 8 between 3-80 ( $2\theta$ ) values for Cu complex. Where  $\theta$  is Brages angle all the main peaks are indicted and calculated values of Miller indeces (h k l) along with observed d-specified and reveled intensities are specified in the Fig. 8. All the peaks have been indexed  $2\theta$  values compared in graph. Comparison values revels that there are good agreement values of  $2\theta$  and d-values. The powder x-ray diffraction data showed identical features with very poor crystalinity. The patterns are qualitative and dispersive in intensity for Cu complex. The X-RD patterns [25-26] are used to explain qualitatively the degree of crystalinity. X-ray Diffraction data of RAPPTH-Cu complex is presented in table 5.

### 3.7 Anti bacterial activity

The biological activity studies of the RAPPTH ligand and their metal complexes [27-28] were tested against bacteria. All the synthesized compounds were evaluated for their *in vitro* antibacterial activity studies against four bacteria's namely *E.coli*, *staphylococcus aureus*, *pseudomonas aereuginosa* and *B. subtiles*. The comparison studies of antibacterial activity these metal complexes [29-30] and ligand with the reference of streptomycin drug. The biological activity data present in Table 6. The antibacterial activity studies clearly suggest that the most of the metal chelates [31] are shows a good antibacterial activity when compared to streptomycin. RAPPTH-Cu (II) complex showed high activity compared to all the remaining synthesized compounds.



Table 6. Antibacterial Activity of RAPPTH Schiff base Ligand and its metal complexes

Compound	<i>E. Coli</i>	<i>B. Subtilis</i>	<i>Staphylococcus aureus</i>	<i>Pseudomonas aereuginosa</i>
RAPPTH Ligand	6	6	7	5
RAPPTH-Mn(II)	14	10	12	11
RAPPTH-Co(II)	9	7	6	8
RAPPTH-Ni(II)	10	9	10	9
RAPPTH-Cu (II)	18	15	17	15
Streptomycin	22	24	20	22

### CONCUSION

In this paper, the coordination chemistry of a Schiff base ligand obtained from the reaction of p-toulic hydrazide and resacetophenone. The RAPPTH - Mn (II), Co (II), Ni (II), and Cu (II) metal complexes have been characterized by spectral and analytical data. The IR, electronic transition and g tensor data lead to the conclude, that the geometry of the complexes of Mn (II), Co (II), Ni (II), and Cu (II) are octahedral in nature and hence the structure of RAPPTH Schiff base metal complexes are given in Fig.9. In all the complexes, the ligand acts as bidentate. The results of in-vitro biocidal activities of the ligand and its metal complexes clearly show antibacterial activity against the tested organisms. The antibacterial activity indicates the metal complexes have more biological activity than free ligands. All the metal chelates are found to be non-electrolytes

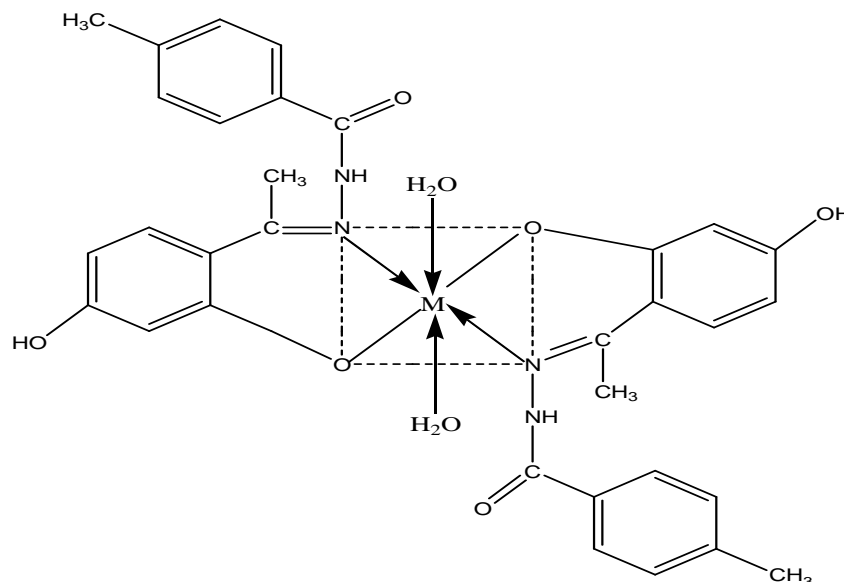


Fig 11: Structure of RAPPTH Metal Complex  
 $M = Mn^{+2}, Co^{+2}, Ni^{+2}$  and  $Cu^{+2}$ .

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