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## Synthesis, Characterization and Antimicrobial Studies of Schiff Base Complexes of 1-(5-Chloro-2-Hydroxy-4- Methylphenyl)Ethanone

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### ABSTRACT

The chemistry of metal complexes has fascinated and inspired the chemist all over the world. Among the chelating ligand, Schiff base have attracted the attention of the chemist due to the ease of the preparation and complexation. Schiff base complexes have remained an important and popular area of research due to their simple synthesis versatility and diverse range of applications. Schiff bases are able to stabilize many different metals in various oxidation states. In the present study the Schiff base of 1-(5-chloro-2-hydroxy-4-methylphenyl) ethanone with 4-(2-aminoethyl)phenol has been synthesized. The complexes of this Schiff base ligand have been synthesized by using Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) ions under refluxed in ethanol. These compounds have been investigated by elemental analysis, infra-red, <sup>1</sup>H nuclear magnetic resonance, diffuse reflectance spectroscopic techniques and magnetic susceptibility measurements. The free ligand and their metal complexes have been screened *in vitro* for their antimicrobial activity against gram-positive bacteria *S. aureus*, gram negative bacteria, *E. coli*, *S. typhi* and fungi *P. notatum* and *R. oligosporus*.

**Keywords:** 4-(2-aminoethyl) phenol, infra-red, magnetic susceptibility measurements, antimicrobial activity etc.

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## INTRODUCTION

Schiff bases play an important role in inorganic chemistry as they easily form stable complexes with most transition metal ions. The development of the field of bioinorganic chemistry has increased the interest in Schiff base complexes, since it has been recognized that many of these complexes may serve as models for biologically important species. Schiff base metal complexes were investigated for fungicidal, bactericidal activities<sup>1-2</sup>. Schiff base derived from amines and aromatic ketones are important due to their behavior. They behave as a chelating agent<sup>3</sup> on one hand and important biocide on the other<sup>4</sup>. Schiff base complexes are considered to be among the most important stereo chemical models in main group and transition metal co-ordination chemistry due to their preparative accessibility and structural variety<sup>5</sup>. The metal complexes show higher activity than the free ligand against the same organism under identical experimental condition. Such increased activity can be explained on the basis of chelation theory<sup>6-8</sup>. Schiff bases appear to be an important intermediate in a number of enzymatic reactions involving interaction of an enzyme with an amino or a carbonyl group of the substrate one of the most important types of catalytic mechanism is the biochemical process, which involves the condensation of a primary amine in an enzyme<sup>9-10</sup>.

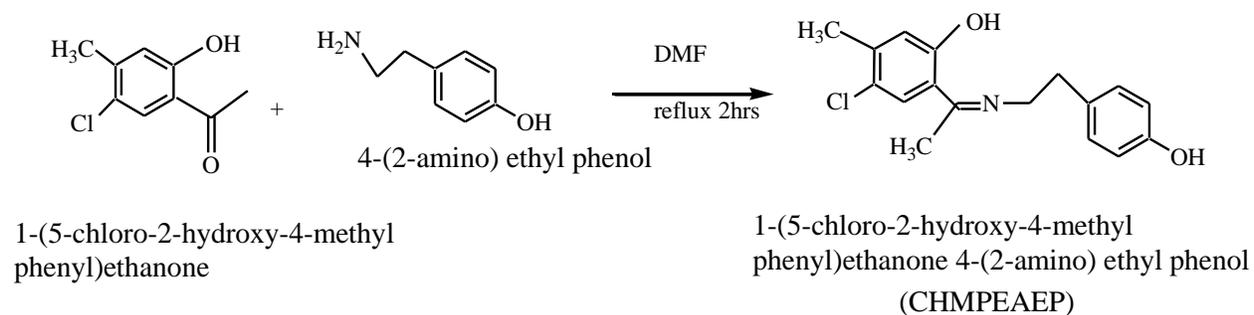
## MATERIALS AND METHOD

The reagents required for the given synthesis is as follows:

### Synthesis of Schiff base (CHMPEAEP)

The ligand (CHMPE) was synthesized by taking equimolar quantities of 1-(5-chloro-2-hydroxy-4-methyl phenyl) ethanone and 4-(2-amino) ethyl phenol in DMF was refluxed for 2 hours with occasional shaking (Figure 1).

### Synthesis of Schiff base (CHMPEAEP)



**Figure 1:**

### <sup>1</sup>H NMR spectrum of CHMPEAEP:

7.2 (7H, m, Ar-H), 6.7 (1 H, s phenolic -OH), 3.8 (2H, T, -CH<sub>2</sub>-CH<sub>2</sub>), 2.3 (1 H, s, -CH<sub>3</sub>), 2.1 (1H, s, -N=C-CH<sub>3</sub>).

### Synthesis of Metal Complexes

The metal complexes were complexes by mixing both solution of Schiff base and Metal (II) acetate in DMF-ethanol in molar ratio 2: 1. The resulting solution was refluxed for 4 to 9 hours on water bath. Colored complexes formed have been filtered and then dried in *vacuum*.

All metal complexes are colored, stable in air. The solids do not melt sharply and undergo decomposition. These are insoluble in water and soluble in organic solvent such as DMF and DMSO giving respective colors to the solution.

### RESULTS AND DISCUSSION

All compounds gave satisfactory elemental analysis. Values are in the close agreement with the values calculated for expected molecular formulae assigned to these complexes, suggesting 1:2 (M:L) stoichiometry. The physical data of ligand and metal complexes are given. (Table 1)

**Table 1: Elemental analysis of CHMPEAEP and its metal complexes**

Sr. No.	Ligand/ complex	Time of Reflux in hours	Colour	Elemental analysis % Found (Calcd).			
				M	C	H	N
1	CHMPEAEP	2	Yellow	--	76.11 (76.02)	6.71 (6.57)	5.22 (5.00)
2	Mn(CHMPEAEP) <sub>2</sub>	9	Dark Brown	9.30 (9.15)	69.03 (69.00)	6.09 (6.00)	4.70 (4.10)
3	Co(CHMPEAEP) <sub>2</sub>	6	Pinkish Brown	9.91 (9.20)	68.91 (67.75)	5.05 (5.97)	4.70 (4.00)
4	Ni(CHMPEAEP) <sub>2</sub>	5	Pale Green	9.61 (9.43)	68.80 (68.35)	6.07 (6.00)	4.72 (3.95)
5	Cu(CHMPEAEP) <sub>2</sub>	4	Dark Green	10.60 (10.00)	68.00 (67.85)	6.00 (5.93)	4.66 (4.01)
6	Zn(CHMPEAEP) <sub>2</sub>	6	Yellowish White	10.81 (10.50)	68.88 (68.02)	5.99 (5.48)	4.65 (4.00)
7	Cd(CHMPEAEP) <sub>2</sub>	6	Opaque	17.28 (17.00)	62.96 (62.00)	5.55 (4.95)	4.32 (3.99)

The IR spectra of the Schiff base ligand was compared with that of the rest of metal complexes to obtain the information about the binding mode of the ligand to the metal complexes. A strong band is observed at 1640-1620 cm<sup>-1</sup> in the spectra of the free Schiff bases which is the characteristic of the azomethine group.<sup>11</sup> Coordination bonding from nitrogen to the metal atom would reduce the electron density in azomethine linkage and due to this lower the -HC=N- absorption. In the spectra of complexes this band is shifted to the region at 1653 to 1570 cm<sup>-1</sup>. A strong band obtained at

2873-2758 $\text{cm}^{-1}$  in all metal complexes has been assigned to chelate  $\nu(\text{O-H})$  absorption. The new bands in the range of 590-540  $\text{cm}^{-1}$  and 470-410  $\text{cm}^{-1}$  which is not present in the free Schiff base are due to  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  vibration<sup>12</sup>. (Table 2)

**Table 2: Infrared spectral data ( $\text{cm}^{-1}$ ) of CHMPEAEP and its Metal Complexes**

Sr. No.	Ligand/ complex	$\nu$ (O-H) Chelate	$\nu$ (C=H) Aromatic	$\nu$ (C=C)	$\nu$ (C=N)	$\nu$ (M-O)	$\nu$ (M-N)
1	CHMPEAEP	--	3022	1454	1620	--	--
2	Mn(CHMPEAEP) <sub>2</sub>	2758	2981	1521	1653	478	526
3	Co(CHMPEAEP) <sub>2</sub>	2765	2991	1506	1683	447	516
4	Ni(CHMPEAEP) <sub>2</sub>	2800	3020	1558	1653	457	567
5	Cu(CHMPEAEP) <sub>2</sub>	2574	3026	1492	1678	486	567
6	Zn(CHMPEAEP) <sub>2</sub>	2922	3061	1496	1653	445	507
7	Cd(CHMPEAEP) <sub>2</sub>	2873	3059	1494	1612	445	505

The magnetic moment was recorded at room temperature and is shown in Table 3. Magnetic studies also help to look into the structure of the complexes. The observed magnetic moment of Mn(II) and Ni(II) found to be 5.78 and 3.00 B.M. indicating octahedral environment around central metal ion. The magnetic moment value of Co(II) and Cu(II) complexes is found to be 3.95 and 1.80 B.M. which is well within the expected range of square planar complexes<sup>13</sup>. The electronic spectrum of Mn(II) complex exhibited three bands at 17200 $\text{cm}^{-1}$ , 20809 $\text{cm}^{-1}$  and 22025 $\text{cm}^{-1}$  assignable to  ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$ ,  ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$  and  ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}$  transition respectively for an octahedral geometry. The Co(II) complex exhibited bands at 9020 $\text{cm}^{-1}$ , 17890 $\text{cm}^{-1}$  and 19150 $\text{cm}^{-1}$  respectively. (Table No.4) which indicate square planar geometry. The electronic spectrum of Ni(II) complex shows absorption bands at 9480 $\text{cm}^{-1}$ , 15385 $\text{cm}^{-1}$  and 25975 $\text{cm}^{-1}$  corresponding to the  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$ ,  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$  and  ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$  transition supporting square planar structure of the complex<sup>14</sup>. The electronic spectrum of Cu(II) complex exhibited bands at 17690 $\text{cm}^{-1}$ , 18680 $\text{cm}^{-1}$  corresponding  ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$ ,  ${}^3\text{B}_{1g} \rightarrow {}^2\text{E}_g$  respective transition. The other high intensity band at 23881 $\text{cm}^{-1}$  give charge transfer transition<sup>15</sup> exhibit square planar complex. (Table 3)

**Table 3: Electronic and Magnetic spectral data of complexes of CHMPEAEP**

Sr. No.	Complex	$\mu$ eff. (in B.M.)	Absorption bands (nm) ( $\text{cm}^{-1}$ )		Assignments
1	Mn(CHMPEAEP) <sub>2</sub>	5.78	581	17200	${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$
			480	20809	${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{1g}$
			454	22025	${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}$
2	Co(CHMPEAEP) <sub>2</sub>	3.95	1108	9020	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$
			558	17890	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$
			522	19150	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$
3	Ni(CHMPEAEP) <sub>2</sub>	3.00	1054	9480	${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{2g}$
			649	15385	${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g}(\text{F})$

4	Cu(CHMPEAEP) <sub>2</sub>	1.80	384	25975	<sup>3</sup> A <sub>2g</sub> → <sup>3</sup> T <sub>1g</sub> (P)
			565	17690	<sup>2</sup> B <sub>1g</sub> → <sup>2</sup> A <sub>1g</sub>
			535	18680	<sup>3</sup> B <sub>1g</sub> → <sup>2</sup> E <sub>g</sub>
			418	23881	C.T.
5	Zn(CHMPEAEP) <sub>2</sub>	Diamagnetic	–	–	–
6	Cd(CHMPEAEP) <sub>2</sub>	Diamagnetic	–	–	–

## ANTIMICROBIAL ACTIVITY

### Antibiogram Test (By Agar well diffusion):

Complexes may have antimicrobial activity against selected various microbes. To check the resistivity and sensitivity of synthesized compounds against selected pathogens antibiogram test was performed by Agar well diffusion method. Muller-Hinton Agar and Potato dextrose Agar plate were spread with selected test pathogen *E. coli*, *S. aureus* and *S.typhi* etc. Well were made at appropriate distance into the Muller-Hinton and PDA Agar plates with the help of gel puncture and filled by the sample. Petri plates were incubated at 37<sup>0</sup>C for 24-48 hours. Then the zone of inhibition was measured and noted. The antimicrobial activity of sample was determined by measuring the clear zone around the well.

The *in vitro* antifungal assay was performed by the disc diffusion method. The complexes and ligand were tested against the fungi *P. notatum*, and *R. oligosporus*, cultured on potato dextrose agar as the medium. The inhibition zone which developed on the plate was measured in mm. (Table 4)

**Table 4: Antimicrobial analysis of CHMPEAEP and its Metal Complexes**

Ligand/ complex	Antibacterial			Antifungal	
	Zone of inhibition in mm			Zone of inhibition in mm	
	<i>S.aureus</i>	<i>E.coli</i>	<i>S.typhi</i>	<i>P.notatum</i>	<i>R.oligosporus</i>
CHMPEAEP	R	R	R	20	R
Mn(CHMPEAEP) <sub>2</sub>	R	12	R	R	R
Co(CHMPEAEP) <sub>2</sub>	25	30	22	30	12
Ni (CHMPEAEP) <sub>2</sub>	15	R	15	R	R
Cu(CHMPEAEP) <sub>2</sub>	R	18	20	R	R
Zn(CHMPEAEP) <sub>2</sub>	20	12	R	35	R
Cd(CHMPEAEP) <sub>2</sub>	R	R	R	20	R

R-Resistance, Diameter of well 6mm, *S.aureus*-*Staphylococcus aureus*, *E.coli*-*Escherichia coli*, *S.typhi*-*Salmonella typhi*, *P.notatum*-*Penicillium notatum*, *R.oligosporus*-*Rhizopus oligosporus*.

## CONCLUSION

The compound synthesized in the present investigation has been subjected to various antimicrobial screening programs based on their structural features so as to ascertain their activity against

different microorganism. For this purpose DMSO was used as a solvent. Antimicrobial and antifungal data for *S.aureus*, *E.coli*, *S.typhi*, *P.notatum* and *R.oligosporus* have shown in Table No. 4 for the purpose of comparison. It has been observed that compound show very good antibacterial and antifungal activity for Co(II) and Cd(II) complexes. The result of preliminary study on antibacterial activity indicated that most of the compounds were highly active against *E.coli* as well as *S.typhi* and same for antifungal activity against *P. notatum* also.

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