

**INTERNATIONAL JOURNAL OF ADVANCES IN PHARMACY,  
BIOLOGY AND CHEMISTRY****Research Article****Synthesis and Characterization of Complexes of Schiff  
Bases of Transition Metals****Seema I Habib and Prafullakumar A Kulkarni**

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**ABSTRACT**

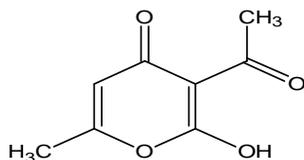
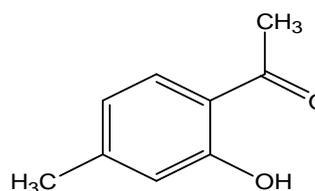
The Ni (II) and Co (II) Schiff base complex derived from dehydroacetic acid 4-chlorophenyl aniline, dehydroacetic acid naphthyl aniline, dehydroacetic acid phenyl aniline and metal halides has been prepared and characterized. Several physical tools in particular, elemental analysis, molar conductance, magnetic moment, infrared and <sup>1</sup>H NMR spectroscopies were used to investigate the chemical structure of the prepared complex. The elemental analysis data shows 1:3 [M: L] ratio. The molar conductance measurement reveals the presence of a non electrolytic nature. The infrared spectral data of the complex display the complexation behavior of the Schiff base towards Ni (II) and Co (II) ion. The free Schiff bases and their complexes have been tested for their antibacterial and antifungal activities against several human pathogenic bacteria and fungi, the obtained result showed enhancement in activity on coordination of metals with ligands.

**Keywords:** Schiff bases, metal complexes, spectral study, antimicrobial activity.**INTRODUCTION**

Schiff bases are important class of ligands and have got wide applications in various fields<sup>1, 2</sup>. In recent years schiff bases and their metal complex have played very important role in coordination chemistry and therefore more attention has been paid on them. They are regarded as model system of biochemical interest. Various studies have shown that the azomethine group (>C=N-) in Schiff base complex has considerable biological significance and responsible for biological function such as fungicidal and insecticidal activity.

Number of metal chelates was synthesized in last few decades using variety of ligands. Schiff bases due to their great flexibility in structure have a major contribution and therefore limited scope in the earlier studies have enlarged to manifold.

The heterocyclic dehydroacetic acid (DHA) [3-acetyl, 6-methyl (2H) pyran, 2, 4-(3H) dione], which is used in the present work has an analogous structure with substituted 2-hydroxy acetophenone.

**DHA****2-hydroxy-4-methyl acetophenone**

Literature survey reveals that many organic compounds in particular heterocyclic aromatic compounds are physiologically active. Number of heterocyclic compounds like benzoxazole, benzimidazole, benzothiazole are reported to possess a variety of physiological activity such as fungicidal, insecticidal, antimicrobial, anticonvulsant and anesthetic properties. One of such heterocyclic compound is DHA which has proved its significance in industries due to its physiological activity. In view of this it was thought worthwhile to synthesize the Schiff bases of DHA and their metal complexes.

**Chemicals**

All the chemicals used to carry out experimental work were of AR grade. The purity of chemicals was checked by routine tests like M.P, B. P and thin layer chromatography.

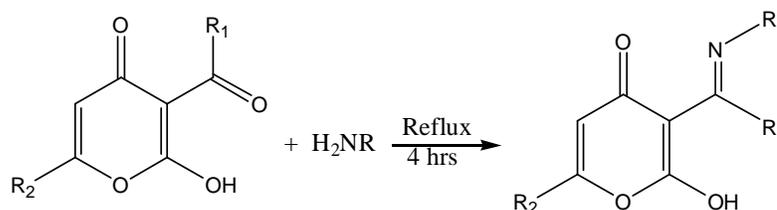
**EXPERIMENTAL**

Synthesis of Schiff bases: Schiff bases were prepared by refluxing equimolar mixture of DHA and amine in ethanol.

0.1 mol of each DHA and primary aromatic amine in 100ml ethanol was taken in RB flask. The contents were refluxed for 4 hours. After refluxion, the content in RB flask were cooled. The Schiff

bases was separated out as solid, it was washed by ethanol, recrystallised from ethanol and dried in a vaccum desiccator.

These ligands are stable to air and moisture; they are soluble in alcohol, chloroform, dioxane, DMF and DMSO.

**Scheme:**

Where

| Sr.no. | R               | R <sub>1</sub>  | R <sub>2</sub> |
|--------|-----------------|-----------------|----------------|
| 1)     | CH <sub>3</sub> | CH <sub>3</sub> |                |
| 2)     | CH <sub>3</sub> | CH <sub>3</sub> |                |
| 3)     | CH <sub>3</sub> | CH <sub>3</sub> |                |

**Table 1: Physical data of Schiff bases**

| Sr. No. | Compound code  | Molecular formula                                  | Colour        | Melting point °C |
|---------|----------------|--|---------------|------------------|
| 1       | L <sub>1</sub> | C <sub>14</sub> H <sub>11</sub> O <sub>3</sub> NCl | Yellow        | 140              |
| 2       | L <sub>2</sub> | C <sub>18</sub> H <sub>14</sub> O <sub>3</sub> N   | Pinkish White | 168              |
| 3       | L <sub>3</sub> | C <sub>14</sub> H <sub>12</sub> O <sub>3</sub> N   | Light yellow  | 118              |

**Synthesis of metal complexes**

In the present work the metal complexes are synthesized by refluxing the ethanolic solution of ligands and metal chlorides in 3:1 molar ratio, i.e 0.03 moles of ligand [in slight excess] was taken in RB flask containing 30 ml of ethanol and heated 0.01 moles of metal chloride was dissolved in 20 ml of ethanol. This metal solution was added dropwise in hot solution of ligands. The contents were refluxed for two hours. 10% alcoholic ammonia solution was added. The complexes of different metal were precipitated at different PH. The precipitate of complex formed was filtered in hot condition. It was washed with ammonia followed by petroleum ether 40-60°C and dried in desiccator over fused CaCl<sub>2</sub>.

**Table 2: Physical data of metal complexes**

| Sr. No. | Molecular formula   | Colour      | D. P °C | Molar Conductivity S Cm <sup>2</sup> Mol <sup>-1</sup> |
|---------|---|-------------|---------|--|
| 1       | C <sub>42</sub> H <sub>39</sub> O <sub>12</sub> N <sub>3</sub> Cl <sub>3</sub> Ni | Orange      | 268     | 20.49  |
| 2       | C <sub>42</sub> H <sub>39</sub> O <sub>12</sub> N <sub>3</sub> Cl <sub>3</sub> Co | Brown       | 192     | 6.84   |
| 3       | C <sub>54</sub> H <sub>48</sub> O <sub>12</sub> N <sub>3</sub> Ni                 | Light brown | >280    | 22.17  |
| 4       | C <sub>54</sub> H <sub>48</sub> O <sub>12</sub> N <sub>3</sub> Co                 | Brown       | >280    | 17.34  |
| 5       | C <sub>42</sub> H <sub>42</sub> O <sub>12</sub> N <sub>3</sub> Ni                 | Yellow      | 246     | 26.22  |
| 6       | C <sub>42</sub> H <sub>42</sub> O <sub>12</sub> N <sub>3</sub> Co                 | Dark brown  | >280    | 15.28  |

**RESULT AND DISCUSSION**

All the complexes are stable to air and moisture insoluble in water and most of the common organic solvent but soluble in CHCl<sub>3</sub>, DMF and DMSO. The analytical data of the complexes indicates that

their stoichiometry may be represented as 1:3 metal to ligand ratio.

### IR Spectra

On comparison of the IR spectra of the metal complexes with that of its ligand, it was found that the  $\nu(\text{C}=\text{N})$  stretching vibration is found in the Schiff base at near  $1633\text{ cm}^{-1}$ , this band is shifted to lower wave numbers in the complex indicating the participation of nitrogen in coordination<sup>3</sup>. A new band at  $\nu\text{ M-O}$  and  $\nu\text{ M-N}$  stretching vibration<sup>4</sup> were appeared in between  $500\text{-}400\text{ cm}^{-1}$  in the spectra of metal complex.

### <sup>1</sup>H NMR

The <sup>1</sup>H NMR spectra of complexes and ligand show well resolved signals. Due to presence of

metal ion there observed broad peaks indicating the formation of the complex.

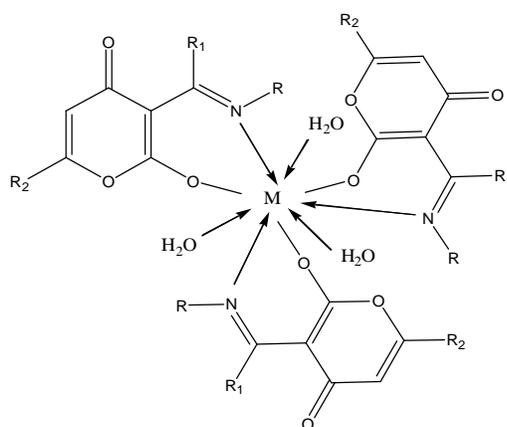
### Magnetic moment

The  $\mu_{\text{eff}}$  values at room temperature for Ni (II) and Co (II) are in the range of 2.94-3.16 and 4.36-4.89 B.M respectively. These values are expected for octahedral geometry of Ni (II)<sup>5, 6</sup> and Co (II) complexes at 25°C.

### Molar Conductivity

The molar conductance values of the complexes in DMF solvents are in the range of 6.84-26.22 S  $\text{cm}^2\text{ mol}^{-1}$  suggesting their non electrolytic nature<sup>7</sup>.

On the basis of analytical data, magnetic moment, electronic spectral data, IR and <sup>1</sup>H NMR, the Ni (II) and Co (II) complexes of the selected ligands have been concluded a octahedral geometry.



M= Ni (II), Co (II)

### Antimicrobial activity

Antimicrobial activity was assayed by cup plate agar diffusion method<sup>8</sup> by measuring inhibition zones in mm. in vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against four strains of bacteria which include E. coli, S.typhi, S.aureus and B. subtilis and against four fungal strains like A. niger, A. flavus, F.moneliforme and p.chrysogenum. The standard used was penicillin and grysofulvin.

From the results of antimicrobial activity of ligand and complex it is clear that the complexes shows enhance activity than the ligands. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligand<sup>9</sup>.

### CONCLUSION

The analytical data shows 1:3 metal to ligand stoichiometry and from antimicrobial activity it is found that the complexes are more active than their parent ligand and hence may serve as vehicle for activation of the ligand as principle cytotoxic species

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