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# Synthesis and characterization of novel Schiff's base ligands derived from 1,8 diamino naphthalene and 5-bromo-2-hydroxyacetophenone.

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**ABSTRACT:** Schiff base ligands are synthesized through the acid-catalyzed condensation reaction between aromatic amine and a carbonyl compound (aldehyde or ketone). Typically, equimolar amounts of the reactants are dissolved in a solvent like ethanol or methanol, and the mixture is refluxed to drive the dehydration that forms the central imine (C=N) bond and the Schiff base. The solid product is then isolated by cooling, filtration, washing, and drying, often followed by recrystallization for purification.

Due to their flexible coordination chemistry and broad range of applications in catalysis, material science and bioinorganic chemistry Schiff bases have attracted notable attention. In this study, a sequence of novel Schiff base ligands were synthesized by reacting aromatic ketones with aromatic amines under mild conditions. The synthesized ligands were characterized using spectroscopic techniques such as infrared spectroscopy (IR), nuclear magnetic resonance (NMR) and ultraviolet-visible (UV-Vis) spectroscopy. The results confirmed the successful formation of the Schiff base ligands, and their potential applications in coordination chemistry are discussed.

**KEY WORDS** : 5-bromo-2-hydroxyacetophenone, diaminonaphthalene, Schiff base, etc.

## I. INTRODUCTION

The scientist Hugo Schiff in 1864 first reveals Schiff bases are a class of organic compounds formed by the condensation between primary amines and aldehydes or ketones, resulting in the formation of an imine (-C=N-) bond[1]. These are also called imines or azomethine which are analogue of aldehyde or ketone in which carbonyl group (C=O) replaced by azomethine (C=N) group[2].The complexing properties of imines with metals have led to the formation of various complexes in which imines act as a ligand. Imines can be a good ligand if they bear –OH group closer to the azomethine (C=N) group. The study of imine metal complexes is very interesting because of their unusual stability, sensitivity, selectivity, chemical and magnetic properties. The chemical and biological importance of azomethine (C=N) group is due to presence of lone pair in $sp^2$ hybridized orbital which make them a good chelating agents. Generally the bi and tri dentate ligands are more capable of forming stable complexes with S and D block elements.

This class of organic compounds has great importance in the field of coordination chemistry because of potentially capable of forming stable complexes with metal ions[3]. Therefore, imine metal complexes were significantly investigated for their antifungal, antibacterial, antimicrobial, antitumor and enzymatic activities[4-8]

Recently the study of chemistry of imines continuously is increasing because of their applicability in various biological systems, polymer stabilizer, homogeneous and heterogeneous catalysis, medicine, pharmacy and other technologies[9]. Schiff's bases exhibit a wide range of biological activities such as antibacterial, antifungal, anti-inflammatory, antiviral and antipyretics[10-11].

Literature survey shows that many researchers have made their attempt to synthesize imine derivatives by condensation of substituted 2-hydroxy acetophenone with substituted aromatic amines and evaluated their biological properties. Patil et.al.[12] synthesized imine derivatives from substituted 2-hydroxy acetophenone with substituted aromatic amines and evaluated their antibacterial and antifungal properties. But however, there has been less information about the study of binary and ternary metal complexes of imine derived from substituted 2-hydroxy acetophenone and substituted aromatic amines.

So keeping these views in consideration to understand the chelating nature of imines towards transition metal ions, present work is undertaken.

## II. RELATED WORK

Scientists worldwide have synthesized various Schiff bases derived from aromatic aldehydes and ketones. Schiff base metal complexes show various biological properties. Esther,I.Ville et.al[13] synthesized a Schiffbase derived from 2-hydroxy-1- naphthaldehyde and 1,8- diaminonaphthalene in ethanol and studied the antimicrobial activities of the Schiff base ligand.

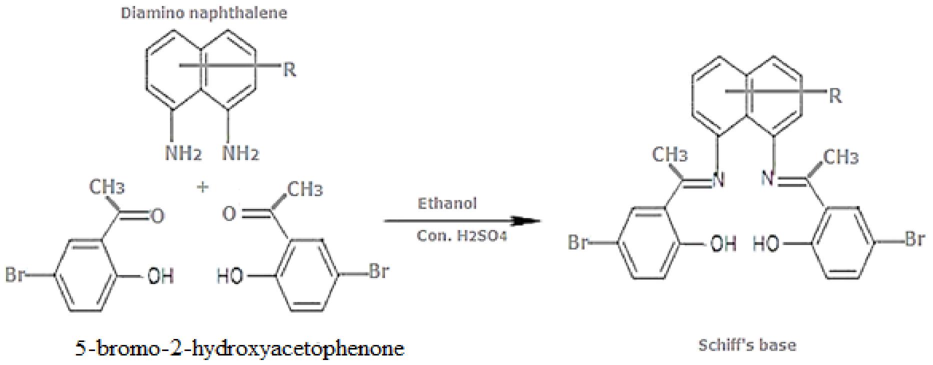
## III. MATERIALS AND METHODS

### Materials

All chemicals used in this study were of analytical grade and used without further purification. Aromatic ketones (e.g., 5-bromo-2-hydroxyacetophenone) and primary amines (e.g., Diamino naphthalene) were purchased from Sigma-Aldrich. Solvents such as ethanol and methanol were used for the synthesis and purification of the ligands.

### Synthesis of Schiff Base Ligands

The Schiff base ligands were synthesized by reacting equimolar amounts of an aromatic ketone and diamino naphthalene in ethanol followed by reflux conditions. For example, 5-bromo-2-hydroxyacetophenone (2mmol) was mixed with 1, 8 diamino naphthalene (1 mmol) in 20 mL of ethanol, and the mixture was refluxed for 5 hours. The reaction progress was monitored using thin-layer chromatography (TLC). After completion, the reaction mixture was cooled to room temperature, and the resulting solid product was filtered, washed with cold ethanol, and dried under vacuum.



| Compound | R Group | Colour | Molecular weight | M.P./B.P. | Yield |
|---|---|---|---|---|---|
| Schiff Base 1 | R= -H | Pale Yellow | 552.26 gm/mole | 142$^0$C | 83% |
| Schiff Base 2 | R= 4 -Cl | Dark Yellow | 586.70 gm/mole | 154$^0$C | 74% |
| Schiff Base 3 | R= 4 –Br | Orange | 631.15 gm/mole | 148$^0$C | 71% |
| Schiff Base 4 | R= 2 –Cl | Dark Yellow | 586.70 gm/mole | 163$^0$C | 69% |

## IV. RESULTS AND DISCUSSION

The aromatic ketones on reaction with the diaminonaphthalene formed Schiff's base with good yields ranging from 69-83%. All the ligands formed are air stable. The substituted Schiffs bases showed various colours ranging from light yellow to dark yellow and orange (Table 1). The molecular weight of substituted Schiffs base ligands ranging from 552.26 to 631.15 gm/mole. The substituted Schiffs base ligands melted in the range of 142-163$^0$C. This indicates the high thermal stability of the compounds. The results indicated that the experimental data are in close agreement with the theoretical values and agrees with the formation of Schiff's base ligands.

**$^1$H NMR spectra:** The $^1$HNMR spectra of the Schiff's base ligand was recorded in DMSO solvent. The free ligand (HL) exhibited a singlet signal at $\delta$ 12:00 ppm due to hydrogen-bonded phenolic proton.The multiplet signals in the range of $\delta$ 6.32 - 8.89 ppm indidicates aromatic proton. 2.30 – 2.40 ppm indicates CH3 groups and DMSO protons appeared at $\delta$ 2.53-3.33 ppm.

**Infrared Spectra:** The relevant IR frequencies exhibited by the substituted Schiff base ligand showed a broad band at 3415 cm$^{-1}$ which is due to the orthohydroxyl group,(-OH) of the Schiff base ligand. The azomethine (-C=N) band in the spectra of the ligand appeared at 1658cm$^{-1}$ as a broad band.The phenolic C – O stretching frequency appeared at 1250 cm$^{-1}$ in the Schiff base ligand, this indicate the higher acidity of the OH group in the ligand which in turn affects the strength of the C – O bond.

**Electronic Spectra**

The UV-Vis spectra of the ligands were recorded in methanol solution in the wavelength range of 200-850 nm. In the ligand , the two absorption bands at 27777 and 25641cm$^{-1}$ are attributed to $\pi - \pi^*$ and $n - \pi^*$ transition of the non-nonbonding electrons present on the nitrogen of the azomethine group.

## V. CONCLUSION

From the IR spectral interpretation, it may be concluded that the Schiff base ligands are tetradentate and can bind the metal ions through the phenolic -OH and azomethine nitrogen atoms. From the electronic spectral interpretation of the complexes in methanol suggested that Schiff base ligands are tetradentate.

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