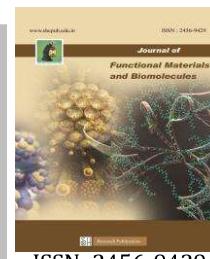




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## DEVELOPMENT OF NOVEL SCHIFF BASE LIGAND

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### Abstract

The Novel Schiff based ligand is expected to exhibit variety of characteristics such as biological and catalytic properties. Schiff bases are studied widely due to their synthetic flexibility, selectivity and sensitivity towards the central metal atom; structural similarities with natural biological compounds and also due to presence of azomethine group ( $-N=CH-$ ) which imports in elucidating the mechanism of transformation and racemization reaction biologically. The imine group present in such compounds has been shown to be critical to their biological activities. This paper concentrates on the synthesis of Schiff base. The main aim of the present work is to synthesize a novel ligand derived from Aminoantipyrine due to its wide range of application as antibacterial agents. The Schiff base displayed good activity against the Gram-positive bacteria and Gram-negative bacteria. The synthesized ligand was characterized by various physical and chemical methods of analysis such as UV-Vis, FTIR,  $^1H$ -NMR, and elemental analysis.

**Keywords:** Schiff base, Aminoantipyrine derivative.

### 1. Introduction

Inorganic and organic ligands have many interesting features in the study of coordination compounds. Heterocyclic compounds with nitrogen, amino nitrogen, and azomethine nitrogen or oxygen play the role of donor atoms, which has more interest in the field of coordination chemistry. When comparing the current research environments of various ligands, Schiff bases have always garnered a lot of attention in the fields of coordination chemistry and

complexion. It is because of their delocalized n-orbitals, multifunctional ligating sites, and flexible nature.[1]

Among the nitrogen containing synthetic compounds Schiff's bases are of the great interest which is due to their higher tendency to coordinate with transition and other metals to form chelating complexes. Schiff bases are obtained as a result of the condensation product of primary amine with a carbonyl compound (aldehyde/ketone). Schiff's base are compounds having ( $-C=N$ ), where nitrogen atom bonded to an aliphatic/aromatic group.[2-5] Due to outstanding complexation abilities, they exhibit a crucial role in the chemistry of coordination compounds.

Schiff's base formation is said to be reversible when it is carried out with aldehyde or ketone. In general they are prepared by refluxing with small quantity of dilute acid. Acetic acid is used predominantly due to its mild acidic nature which helps in the formation of desired product. Stronger acids are not preferred because they tend to break the molecule.[6] The mechanism behind the reaction is formation of hydrazones via nucleophilic addition of amine to the aldehydes and ketones.

Intermediates are formed in this reaction process in which carbinol amine is formed which on losing a molecule of water in presence of dilute acids results in the formation of Schiff's base. The rate determining step is the final step.[57] The resultant imine compounds obtained are basic in nature. Basically amine acts as a nucleophile to

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give carbinolamine which is unsaturated which undergoes dehydration in the rate determining step either in the presence of acid or base,[7-12] Schiff bases are formed in two steps that is addition of  $H^+$  followed by elimination of water.

The coordination of transition metal ions with the synthesized Schiff base ligand results in formation of coloured coordination complexes. These compounds have significant biological and catalytic properties. Metal complex of Rhodium is Wilkinson's catalyst used in the hydrogenation of olefins. Copper complex used in the catalytic epoxidation of styrene.

Schiff's bases are classified according to their complex formation properties with Schiff's base has valuable uses in the field of medicinal chemistry, biology, analytical chemistry, organic and inorganic chemistry[13]

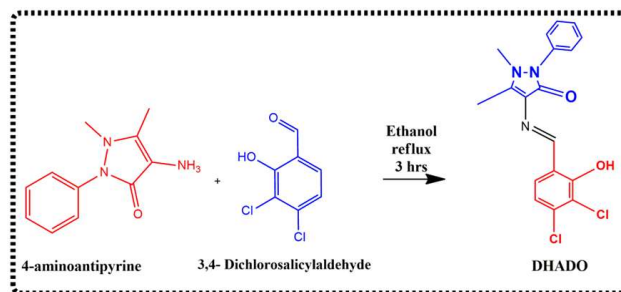
Because of the presence of azomethine group ( $-C=N$ ) in Schiff's base it is very much reliable in the field of medicinal chemistry which is due to the presence of two electrons on  $-N$  atom, which makes the Schiff's base a good chelating molecule with metal ions. Schiff's base metal complexes are also employed as anti-bacterial, anti-tumor, anti-cancer, DNA photocleavage and antioxidant properties.[14]

## 2. EXPERIMENTAL METHODS

### 2.1. Synthesis of ligand 3,4 dichloro-((2-hydroxybenzylidene)amino)-1,5-phenyl-1H-pyrazol-3(2H)-one [DHADO]

About 0.8g, (1mmol) 4-aminoantipyrine was taken and reacted with 0.8g, of 3,4-dichlorosalicylaldehyde (1mmol) in ethanol (40ml). This mixture was refluxed 3 min in microwave irradiation and gradually pale yellow precipitate starts separating out. It was filtered, washed with ethanol and recrystallized from ethanol. (Yield 80%)

#### Scheme 1 Synthesis of ligand DHADO



Compounds	F.W. g/mol	Color	Calculated (found) %				
			C	H	N	O	M
DHADO ( $C_{18}H_{15}N_3O_2$ )	375.05	Yellow	57.46	4.02	11.17	8.50	-

**Table 1. physical characterization, analytical data of the ligand (DHADO)**

### 3.1. Spectral Characterization

#### 3.1.1 Electronic Absorption Spectral Studies

The electronic absorption spectra are often very helpful in the evaluation of results furnished by other methods of structural investigation. The electronic spectral measurements were used for assigning the stereochemistry of metal ions in the complexes based on the positions and number of d-d transition peaks. The electronic absorption spectra of the Schiff base ligand (DHADO) was recorded at room temperature using ethanol as solvent. A peak at 350 nm as depicted in Fig 3.1 clearly shows the  $n-\pi^*$  transition present in ligand DHADO confirming the formation of ligand[15].

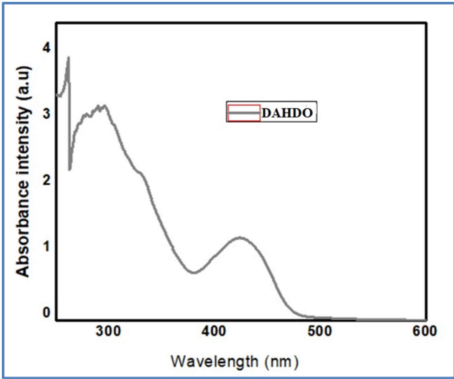


Fig. 3.1. Electronic Spectra of Ligand (DHADO)

3.2. Infrared spectral Studies

The IR spectra of both the free ligand and metal complexes were carried out in the range 4000-400 cm<sup>-1</sup> and the values are tabulated below. The spectral data for ligand and the complexes are presented in the Table 2. The sharp peak at 1636 corresponds to  $\nu(\text{C}=\text{N})$  (azomethine)[15] respectively. The broad band at 3423 cm<sup>-1</sup> complex is due to N-H stretching in the 4-aminoantipyrine ring. The FT-IR spectra are depicted in Fig. 3.2 .

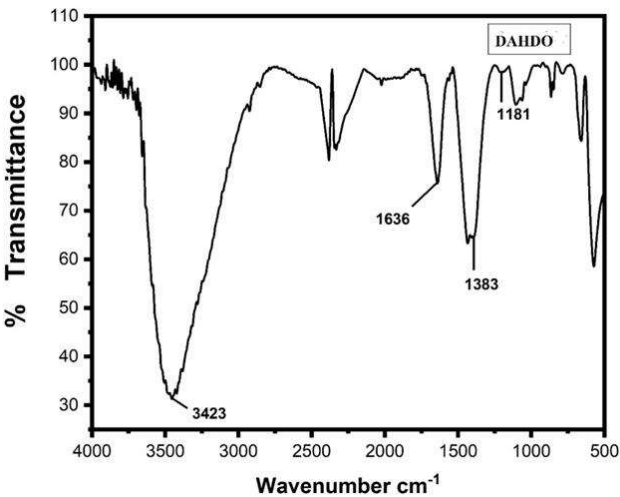


Fig.3.2. IR spectrum of Ligand (DOMPO)

Compounds	$\nu(\text{N-H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C-O})$	$\nu(\text{M-O})$
DAHDO	3423	1636	1383	-

Table 2. IR spectral data of ligand (DAHDO)

3.3. NMR Spectral Studies

Nuclear Magnetic Resonance spectroscopy involves the change of the spin state of a nuclear magnetic moment when the nucleus absorbs electromagnetic radiation in a strong magnetic field. The <sup>1</sup>H NMR spectrum of ligand is recorded in DMSO given in Fig.5.4 The peaks are assigned as follows: azomethine proton as singlet (s, 7.9  $\delta$ ), aromatic protons as multiplet at (7.558-7.621  $\delta$ ). Aminoantipyrine (-NH3) proton at (s, 3.323  $\delta$ )[16].

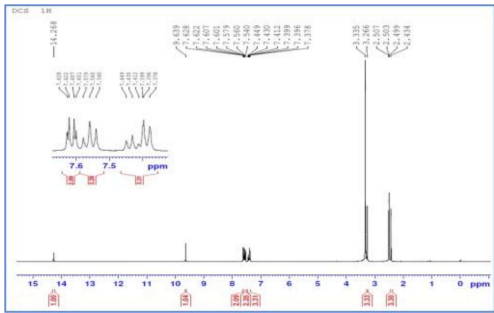


Fig 3.3 1H- NMR spectrum of ligand DAHDO

4. Conclusion

A novel Schiff base ligand DAHDO were synthesized and characterized by spectral and analytical methods. The synthesis was done with greener initiatives using microwave irradiation. The spectral and analytical data confirm the formation of ligand.

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