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SYNTHESIS AND CHARACTERIZATION OF A NOVEL AMINOANTIPYRINE BASED SCHIFF BASE LIGAND

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Abstract

The versatile ligation behavior of Schiff base compounds had evoked considerable interest in the past. It is our aim to synthesize new Schiff base derivative. We have demonstrated that metal ions acting as modulators (or inputs, in digital design parlance) can generate absorbance changes in accordance with various operation. It was also obtained exploiting differential binding affinities of metal ions for different ligands. The present work focuses on the synthesis of ligand. The synthesized ligand was characterized using various physical and chemical methods of analysis such as UV-Vis, FTIR, ^{1}H-NMR.

Keywords: Aminoantipyrine, Synthesis and Characterization

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

In general they are prepared by refluxing with small quantity of dilute acid. Acetic acid is used predominantly

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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 [5-7]. The resultant imine compounds obtained are basic in nature.

Basically amine acts as a nucleophile to 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.

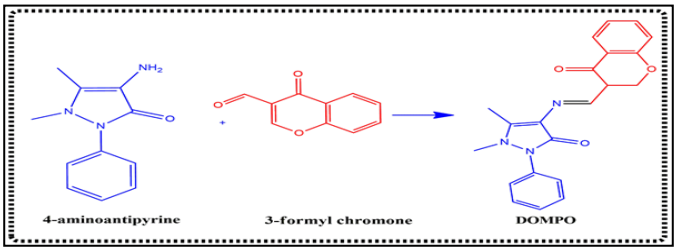
The coordination of transition metal ions with the synthesized Schiff base ligand results in formation of coloured coordination complexes. These compounds have significant biological and catalytic properties. Metal complex of Rhodium is Wilkinson‟s catalyst used in the hydrogenation of olefins. Copper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

2.1.Synthesis of ligand 1,5-dimethyl-4-((4-oxochroman-3-yl)methylene)amino)-2-phenyl-1H-pyrazol-3(2H)-one [DOMPO]

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



Scheme 1 Synthesis of ligand DOMPO

3. Results and Discussions

All the synthesized compounds are stable in air and non-hygroscopic in nature.

Table 1.1. physical characterization, analytical data of the ligand (DOMPO)

Compounds	F.W. g/mol	Color	Calculated (found) %				
			C	H	N	O	M
DOMPO ($C_{21}H_{19}N_3O_3$)	361.14	Yellow orange	47.46	4.43	16.66	14.27	-

They are freely soluble in organic solvents such as Ethanol, DMSO, DMF and acetonitrile. The Co(II) complex was obtained in good yield through the reaction of (DOMPO) with the corresponding Cobalt chloride salts.

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 (DOMPO) was recorded at room temperature using ethanol as solvent. A peak at 255 nm and 402 nm as depicted in Fig 1.1, clearly shows the π -π* and n-π* transition present in ligand DOMPO confirming the formation of ligand [15].

3.2. Infrared spectral Studies

The IR spectra of both the free ligand and metal complex were carried out in the range 4000-400 cm^{-1} and the values are tabulated below. The spectral data for ligand and the complexes are presented in the Table 2. The spectrum of the ligand shows a peak at 3283 cm^{-1} which may be due to ν(N-H) stretching [16] . The bands at 1623 cm^{-1} are assigned to ν(C=N) (azomethine) stretching which confirms the formation of ligand DOMPO.In the Co(II) metal complex of ligand DOMPO the azomethine nitrogen is involved in coordination and undergoes a lower shift to 1608 cm^{-1}.

The broad band at 1372 cm^{-1} complex is due to C=O stretching water. In addition, the IR spectra of Co(II) complex show a sharp peak at 601 cm^{-1} assigned to *ν*(M-Cl) mode respectively.

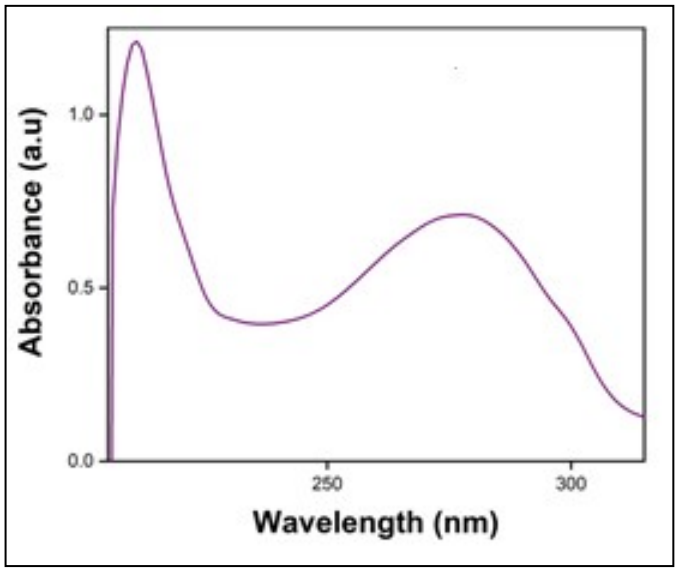


Fig. 1.1. Electronic Spectra of Ligand (DOMPO)

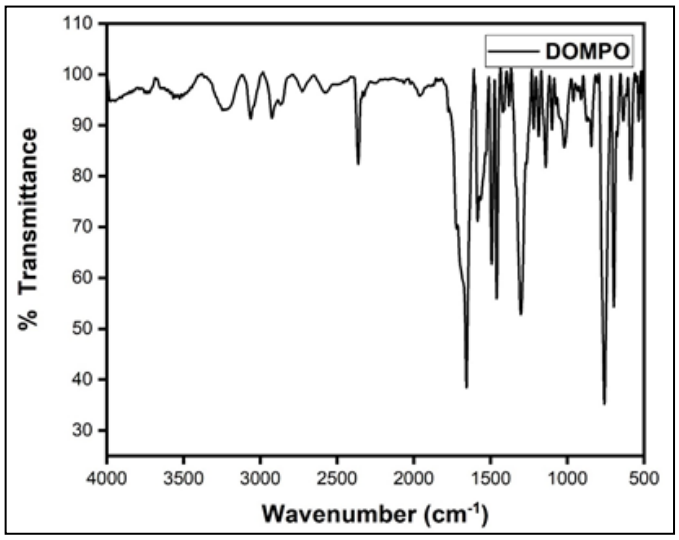


Fig.1.2.IR spectrum of Ligand (DOMPO)

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 ^{1}H NMR spectrum of ligand is recorded in DMSO given. The peaks are assigned as follows: azomethine proton as singlet (s, 8.122 δ), aromatic protons as multiplet at (7.358-7.830 δ). Aminoantipyrine (-NH_3) proton at (s, 3.012 δ)[17,18].

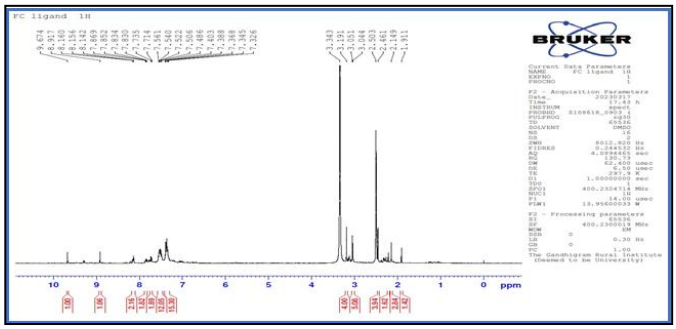


Fig 1.3 ^{1}H- NMR spectrum of ligand DOMPO

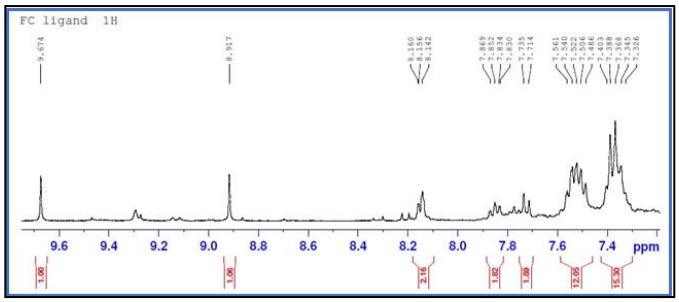


Fig 1.4 ^{1}H- NMR spectrum of ligand DOMPO (from7.3 ppm-10 ppm)

Table 2. IR spectral data of ligand (DOMPO) and its Co(II)complex

Compounds	ν(C=O)	ν (C=N)	ν (N-H)	ν (M-O)
DOMPO	1372	1623	3283	-
[Co(DOMPO)Cl_2]	1360	1608	3283	601

4. Conclusion

A novel Schiff base ligand DOMPO 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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