

Synthesis and Characterization of Some Transition Metal Complexes derived from 3-methoxysalicylaldehyde and 3-aminopyridine (MSAP) of Schiff base ligand

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Abstract: The Schiff base compounds play a significant role in pharmaceutical chemistry. The metal complexes of Co (II), Ni (II), Cu (II) and Zn (II) have been synthesized from the Schiff base ligand which was derived by the condensation of 3-methoxysalicylaldehyde and 3-aminopyridine were analyzed. The synthesized compounds have been characterized by using elemental analysis, molar conductance, magnetic susceptibility, FTIR, UV-Vis, and ^1H NMR. The FTIR spectrum shows that bidentate coordination of metal ions with the ligand is due to O, N the electron donating site of azomethine group. From the analytical techniques, structure and geometry of the complexes were predicted.

Keywords: Schiff base, 3-methoxysalicylaldehyde, 3-aminopyridine, Metal Complexes, Molar conductance, ^1H NMR.

1. Introduction

The synthesis of coordination compounds using organic ligand is one of the most developing, expanding and successful branches of science. Schiff bases have played a vital role in the development of synthetic organic chemistry. The compounds have been exhibit a wide range of biological activities. Schiff base was derived from the condensation of 3-methoxysalicylaldehyde and 3-aminopyridine (MSAP) and its metal complexes of Co(II), Ni(II) Cu(II) and Zn(II) were synthesized^[1-5].

2. Experimental Details

2.1 Synthesis of Schiff base

The Schiff base ligand was prepared by the condensation of 3-methoxysalicylaldehyde and 3-aminopyridine ethanol was used as solvent. The reaction was carried out about 3 hrs, and the yellow color solid product was formed^[6]. The obtained yield was filtered, washed with diethyl ether in several times and recrystallized from ethanol. The product of the ligand is 78%.

2.2 Synthesis of metal complexes

In a hot ethanolic solution of the schiff base ligand and corresponding metal salts were added in (2:1) ratio. The reaction mixture was refluxed for about 5hrs^[7]. The solid product was obtained and it was filtered, washed with ethanol and dried in a anhydrous CaCl_2 .

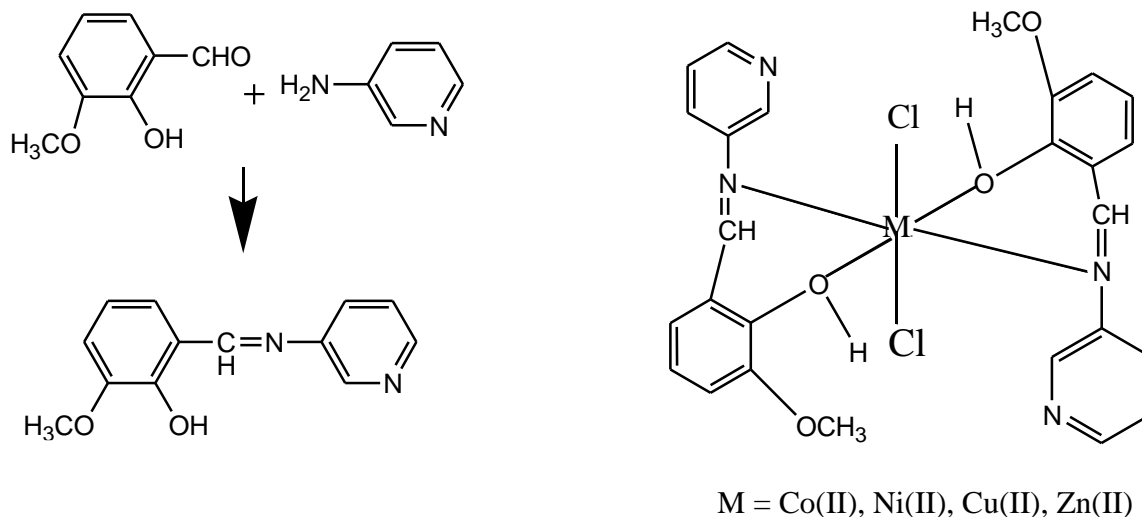


Fig.1 Proposed structure of ligand and metal complexes

3. Results and Discussion

3.1 FTIR Spectral Study

The spectra of all complexes showed sharp band at 1622cm^{-1} due to the presence of azomethine moiety coordinated to the metal ions while decreases its frequency. In all metal complexes a broad band at 3422cm^{-1} attributed due to phenolic OH and also it was coordinated through metal ions. The bands between $400\text{-}470\text{cm}^{-1}$ and $520\text{-}550\text{cm}^{-1}$ were due to M-N and M-O respectively.

Table-1 FTIR Spectral data for ligand and metal complexes

Compound	ν_{OH}	$\nu_{\text{C=N}}$	$\nu_{\text{M-N}}$	$\nu_{\text{M-O}}$
MSAP-L	3422	1622	-	-
MSAP-CoCl ₂	3410	1617	452	539
MSAP-NiCl ₂	3415	1617	468	546
MSAP-CuCl ₂	3419	1612	427	543
MSAP-ZnCl ₂	3407	1616	416	549

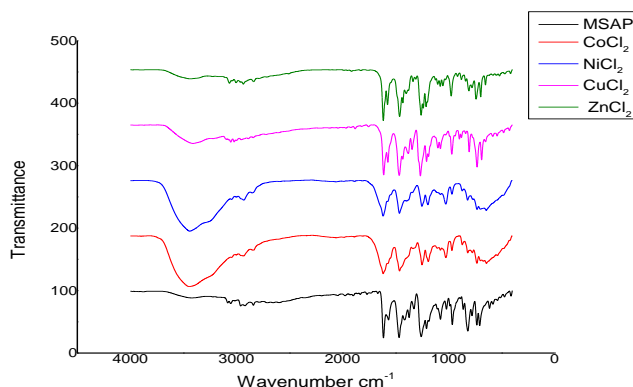


Fig.2 FTIR Correlation Spectrum

3.2 UV-Visible Spectral Study

The electronic spectrum of Co (II) complex shows 412 nm is due to ${}^4T_{1g} \rightarrow {}^4A_{2g}$ (F) transition suggests that octahedral geometry. The Ni (II) complex exhibits at 431 nm which is assigned to ${}^3A_{2g} \rightarrow {}^3T_{1g}$ (F) transition. This confirms the octahedral geometry. The Cu (II) complex display at 437 nm attributed to ${}^2E_g \rightarrow {}^2T_{2g}$ transition also suggests octahedral geometry.

Table-2 UV-Visible Spectral data for metal complexes

Compound	UV-Visible (nm)	UV-Visible (cm^{-1})	Possible Transitions	Geometry
CoCl ₂	412	24,271	${}^4T_{1g} \rightarrow {}^4A_{2g}$ (F)	octahedral
NiCl ₂	431	23,201	${}^3A_{2g} \rightarrow {}^3T_{1g}$ (F)	octahedral
CuCl ₂	437	22,883	${}^2E_g \rightarrow {}^2T_{2g}$	octahedral

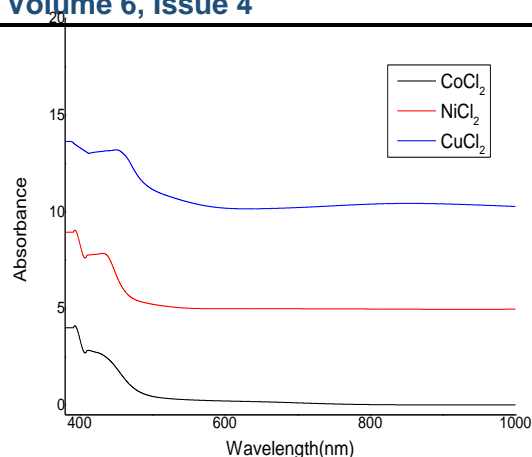


Fig.3 Correlation Spectrum of UV-Visible

3.3 ^1H NMR Spectral Study

The sharp singlet observed at about δ 12.67 ppm is assigned to phenolic $-\text{OH}$ proton. The azomethine proton observed at δ 8.99 ppm in the spectrum of the ligand. The aromatic protons appeared between δ 6.8-8.6 ppm respectively. The peak at δ 3.82 ppm is assigned to $-\text{CH}_3$ group of methoxy substituent on the benzene ring ^[8].

Compound	$-\text{OCH}_3$	$-\text{CH}=\text{N}$	$-\text{OH}$	Aromatic Protons
MSAP-L	3.82	8.99	12.67	6.8-8.6

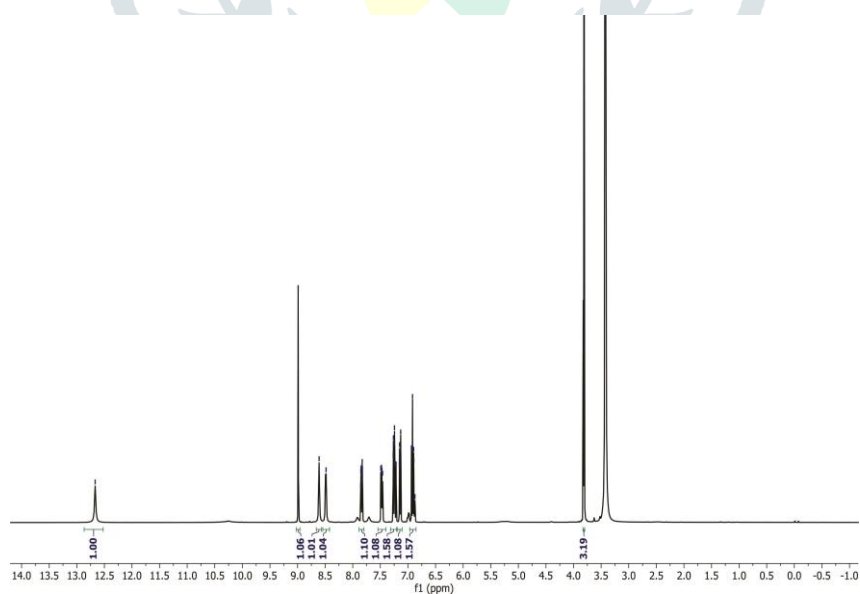
Table-3 ^1H NMR Spectral data for ligandFig.4 ^1H NMR Spectrum of ligand

Table. 4 Elemental Analysis, Colour, Molar Conductance, and Magnetic Susceptibility of the Ligand and Metal Complexes

Compound	Mol. Formula	Mol. Weight	Elemental analysis % Calculated / (Founded)					Colour
			C	H	N	M	Cl	
MSAP-L	C ₁₃ H ₁₂ N ₂ O ₂	228	68.41 (67.86)	5.30 (5.32)	12.27 (12.15)	-	-	Red
MSAP-CoCl ₂	C ₂₆ H ₂₄ Cl ₂ CoN ₄ O ₄	586	53.26 (53.15)	4.13 (3.96)	9.56 (9.33)	10.05 (9.86)	12.09 (11.89)	Orange
MSAP-NiCl ₂	C ₂₆ H ₂₄ Cl ₂ NiN ₄ O ₄	586	53.28 (52.92)	4.13 (3.95)	9.56 (9.31)	10.01 (9.79)	12.10 (11.94)	Pale yellow
MSAP-CuCl ₂	C ₂₆ H ₂₄ Cl ₂ CuN ₄ O ₄	590	52.84 (52.45)	4.09 (3.89)	9.48 (9.22)	10.75 (10.56)	12.00 (11.83)	Green
MSAP-ZnCl ₂	C ₂₆ H ₂₄ Cl ₂ ZnN ₄ O ₄	592	52.68 (51.98)	4.08 (3.78)	9.45 (9.26)	11.03 (10.84)	11.96 (11.72)	Yellow

4. Conclusion

The coordination ability of the newly synthesized Schiff base has been proved on complexation reaction with Co(II), Ni(II), Cu(II) and Zn(II) ions. FTIR, UV-Vis, ¹H NMR and magnetic measurements of the ligand and its complexes confirm the suggested coordination of the ligand through azomethine linkage. Based on these facts, an octahedral structure has been proposed for all complexes.

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