

Synthesis, Spectral Characterization and Microbial Activity of Ni (II) Complex with DBAP Schiff's Base Ligand

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ABSTRACT

Ni (II) complexes were synthesized from the Schiff Base Ligand. The Schiff base ligand [DBAP] (L) is prepared when 3,4- dimethoxy benzaldehyde reacts with 3- amino pyridine. Elemental Analysis, FTIR, UV analysis, HRMS, Magnetic Susceptibility and Molar Conductance data have concluded information regarding the nature of bonding and geometry of the transition metal complex as well as Schiff base Ligand. The Ligand (L) acts as bidentate and co-ordinate through Nitrogen atom of imine group (azomethine group) & Nitrogen atom of the Pyridine ring. Bacterias like E.coli and Bacillus Subtilis (by well disc and fusion method) were used to study the biological activity of the ligand and the Ni(II)complexes. Experimental data revealed that the metal chelates are more active than the free Schiff's base ligand.

Introduction

Schiff Base Metal Complexes [SBMC] have been studied extensively for years due to synthetic flexibilities, selectivity as well as sensitivity towards the metal ions. The presence of donor atoms in the ligand will play an important role in the formation of stable chelate ring and this situation facilitates the complexation process [1]. Transition Metal with the ligands have shown wide spectrum of biological and pharmaceutical activities such as antimicrobial, antibacterial, antifungal, anti-inflammatory, anticonvulsant, antitubercular, antiviral, anti-oxidative effects and inhibition of tumor growth [2-4].

Since Nickel compounds are present in the active sites of urease and are used extensively in the design and building of new magnetic materials, the study of Ni-compounds is of considerable interest in various facets of chemistry. [5-7]

Mechanistic Pathway for Preparation of DBAP Ligand (L): -

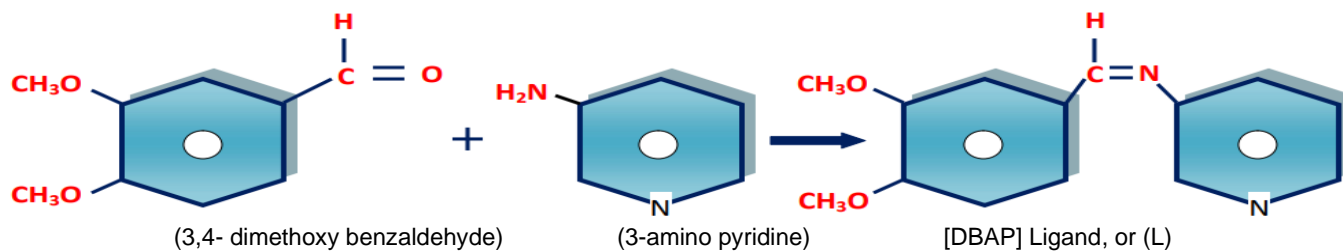


Fig 1: - Preparation of Ligand (L)

Experimental: -

3-amino Pyridine (Rohm), 3,4-dimethoxy benzaldehyde (Rohm, Mumbai) [Laboratory reagent] and remaining all chemicals were procured from various commercial sources and used as such without purification. Standard procedure has been adopted for the present experimental work [8-9].

Synthesis of Schiff Base Ligand: -

0.01 mole of 3,4- dimethoxy benzaldehyde was dissolved in minimum amount of ethanol and the solution was warmed for four hours. To this ethanolic solution of 3-amino pyridine (0.01 mole) was added and refluxed the solution for an hour. Ligand prepared was in liquid state [1 & 2]. The colour of the Ligand is of wine colour & yield is 65%. The mechanistic pathway preparation of the ligand has been depicted in Fig.1.

Synthesis of Metal Complexes: -

The Ligand, (L) (0.005 moles) is dissolved in ethanol and ethanolic solution of corresponding salts (0.05 moles), MX_2 [where M= Metal, X= Cl/Br/I] were mixed together and refluxed with constant stirring for 3-4 hours at refluxing temperature. On cooling coloured solids were precipitated. The products were filtered, washed with cold ethanol and dried in air.

Instrument Used: -

Micro-analytical methods on Gembtt Germany UNICUBE Elemental Analyze were used to determine the percentage composition of Carbon (C), Hydrogen (H), Nitrogen (N) of ligand and complexes. FTIR spectral data of the ligand and complexes were recorded using KBr pellets in the range of $4000-400\text{ cm}^{-1}$, on Perkin Elmer-Spectrum 2. The UV visible spectral data of the Schiff base ligand and the metal complexes of Schiff base ligand were accomplished in DMSO by spectrophotometer. Gouy balance model gave the magnetic measurements of the complex using $Hg[CO(NCS)_4]$ as standard. The conductivity measurements were carried out in DMSO using Electronic Digital conductivity meter 0.01 m KCl solution is used for calibration.

Microbial Activity: -

E-Coli, Bacillus Subtilis were used to study the antibacterial activities of the Ni(II) complexes. Ni-metal complexes dissolved in DMSO at a concentration of 1mg/mL was prepared. Trypticase Soy Agar media (Sigma-Aldrich) were used as basal media for testing bacteria. These Trypticase Soy Agar media were inoculated with 0.2 ml of the 24 hours liquid cultures comprising microorganisms. Sample dish were placed delicately on pre-inoculated agar plates, and Escherichia coli (E. coli) and Bacillus Subtilis were incubated aerobically at $37\text{ }^\circ\text{C}$ for 24 hours. Inhibitory activity was estimated (in mm) as the diameter of the remarked inhibition zones. From the Table (5), the different inhibition of the investigated complex results has been explained. The inhibition values of different complexes are as: $[Ni(DBAP)_2Cl_2]$ (Significant)[S], $[Ni(DBAP)_2Br_2]$ (Moderate)[M], $[Ni(DBAP)_2I_2]$ (Moderate)[M] with the Antibiotics like Amikacin (18), Ceftriaxone (16), Gentamycin (resist), Levofloxacin (12). The inhibition factor can be seen by the photo depicted in Fig.2. Biological studies of Ni (II) complexes showed better microbial activity as compared to ligand.

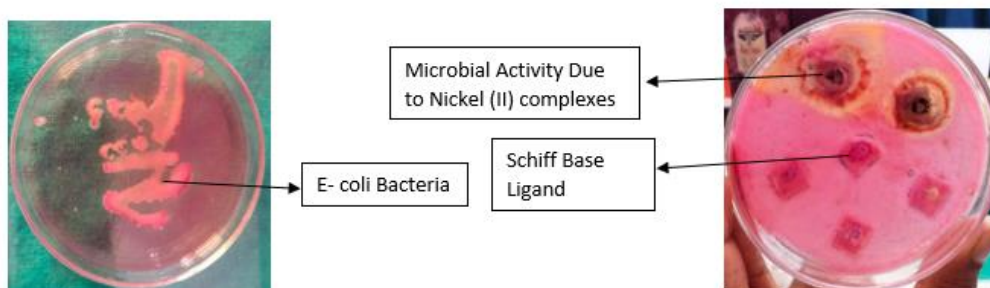


Fig. 2:- Antibacterial Activity against Pathogenic Bacteria with DBAP Schiff Base Ligand and Nickel(II) Complexes

Results & Discussion: -

Table 1 depicts the physical and analytical data of Schiff base ligand and the metal complexes. The data shows that the Ligand [L] forms 1:2 [M: L] complex with Ni (II). The processed complexes are found to have the formula $[Ni(L)_2Cl_2]$, $[Ni(L)_2Br_2]$ and $[Ni(L)_2I_2]$, where L is Schiff base Ligand. The Ligand binds with the metal in a bidentate manner. The C, H, N, O analysis of Schiff base ligand and its complexes are found in satisfactory agreement with the likely predicted values. [Table-2]

FTIR Spectra & Mode of Binding: -

FTIR spectra of Schiff base ligand signifies a very strong absorption band at 1677 cm^{-1} [10] which is due to ν (C=N) of imine. For the spectrum of analyzed Complexes, this absorption band has been shifted to lower region by $67-76\text{ cm}^{-1}$

¹, [11-12] indicating the co-ordination of the Schiff base ligand through Nitrogen atom present in the 3rd position to pyridine ring. i.e. (imine nitrogen.) advocating coordination via the azomethine group in all the complexes (M-N).

The another characteristic FTIR spectrum shows an intense band at 1586 cm^{-1} which is a characteristic of ν - pyridine ring. The observed band gets shifted to lower region by $(70-76\text{ cm}^{-1})$, indicates co-ordination of ligand through N-atom of the pyridine ring.

In addition to the above, the FTIR spectra of metal chelates show absorption band at $440-430\text{ cm}^{-1}$ are attributed due to ν (M-N) stretching. These bands are absent in the spectrum of the Schiff base ligand. (Table 3) (Fig. 3-6)

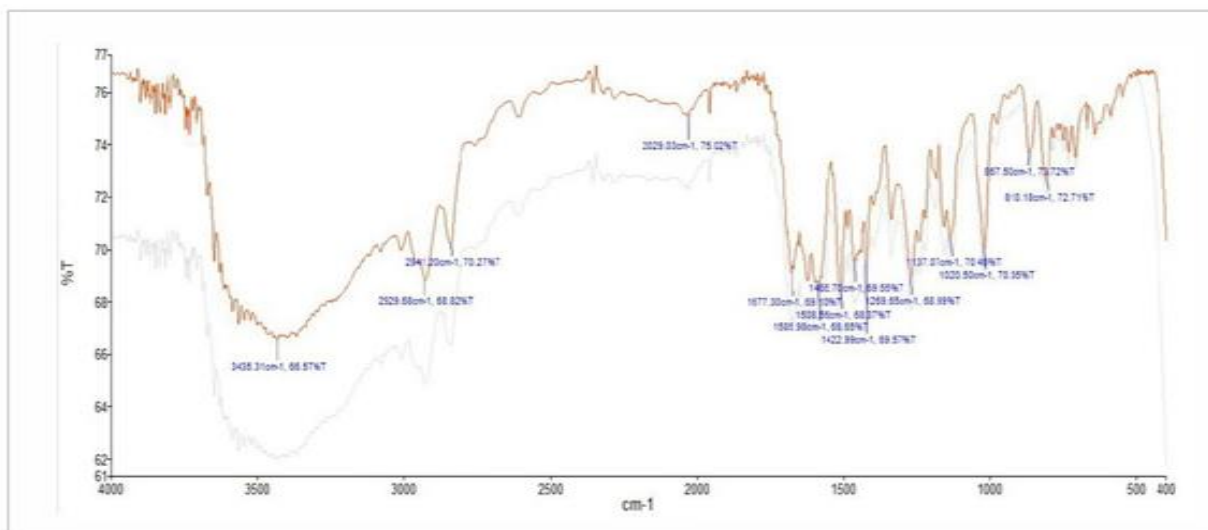


Fig 3 :- FTIR Spectral Study of Ligand(DBAP)

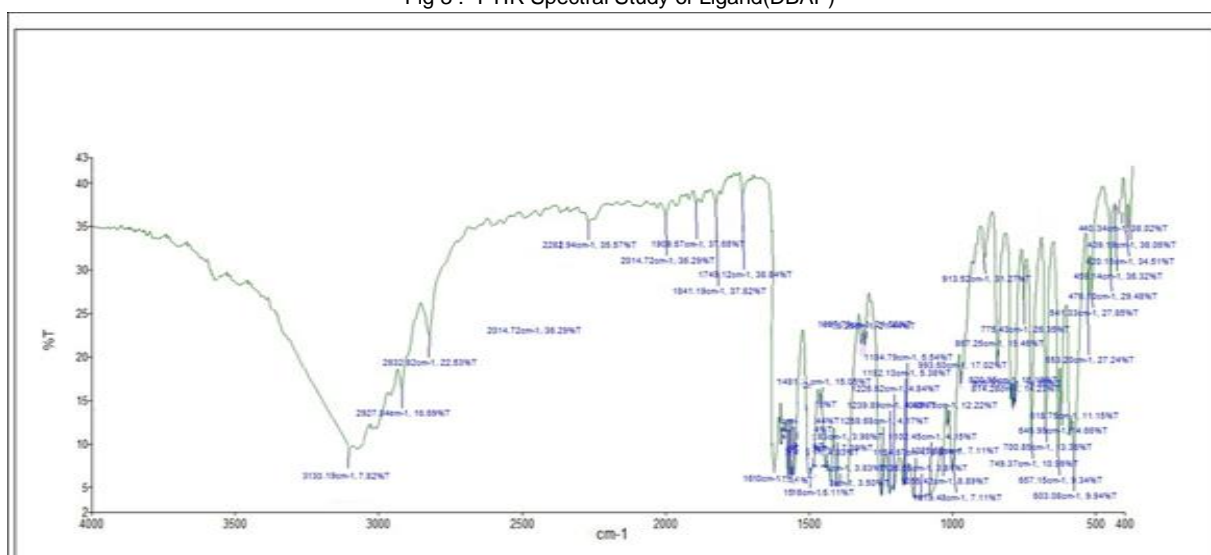


Fig 4:- FTIR Spectral Study of [Ni(DBAP)₂Cl₂]

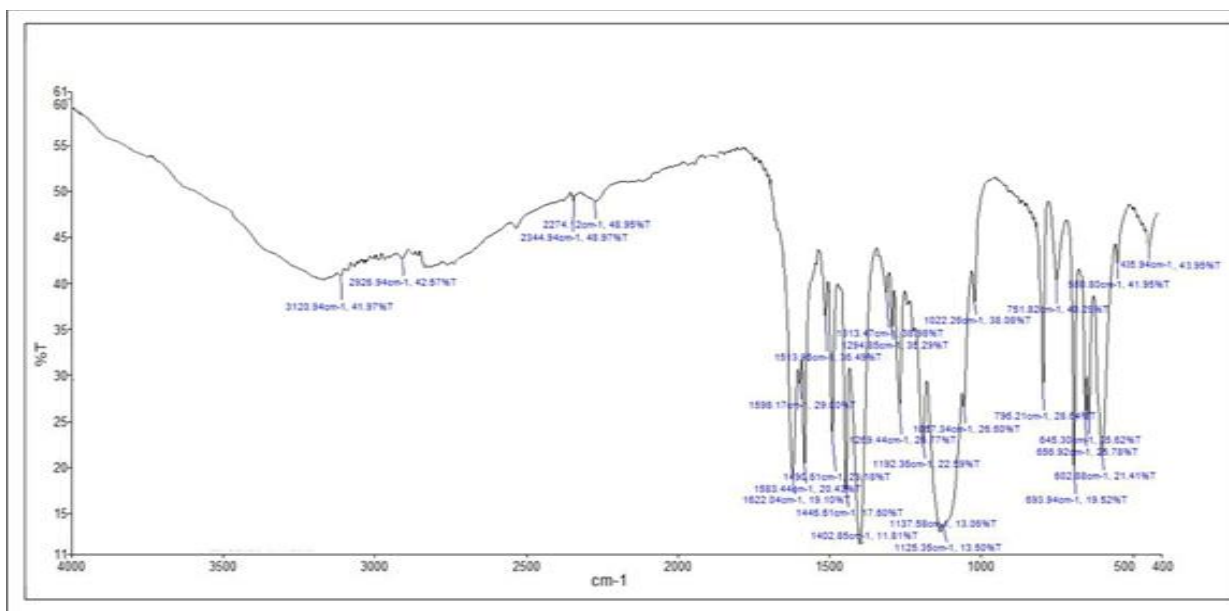


Fig 5:- FTIR Spectral Study of [Ni(DBAP)₂Br₂]

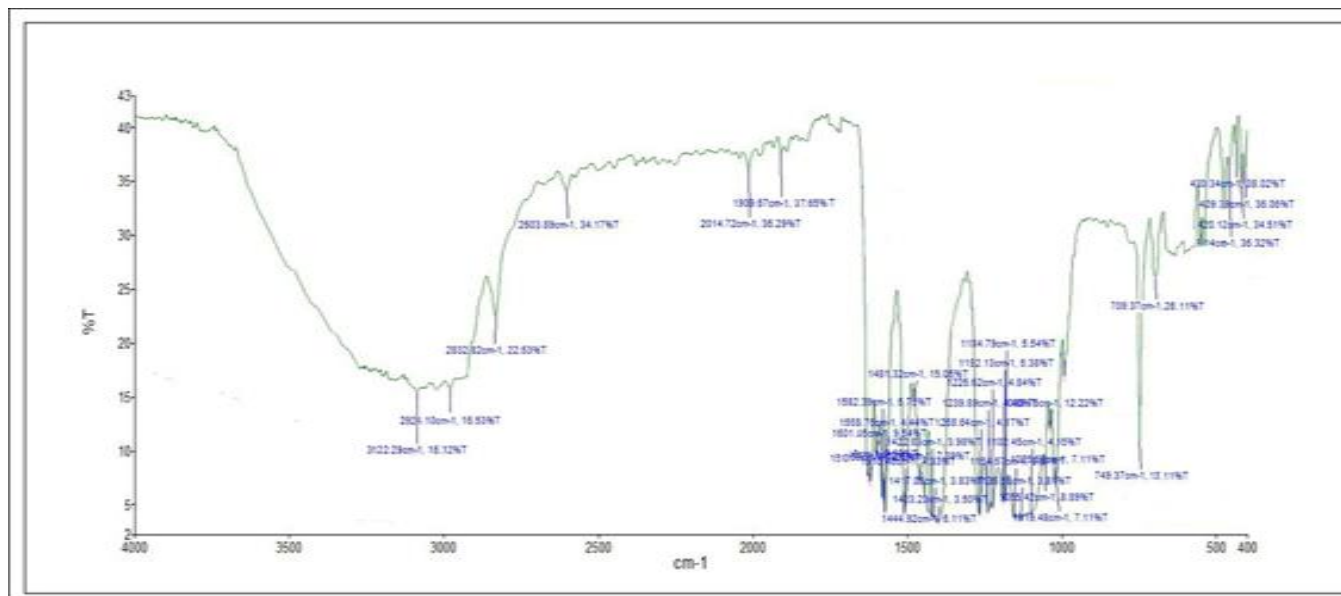
Fig 6:- FTIR Spectral Study of $[\text{Ni}(\text{DBAP})_2]_2$ **Magnetic Moment and Electronic Absorption Spectra: -**

Table 4 explains the different electronic absorption data and magnetic moment values of Schiff base ligand and its chelates of transition metals. Frequency 25126cm^{-1} and 42017cm^{-1} attributes to $n-\pi^*$ and $\pi-\pi^*$ transition respectively. Strong absorption band is shown by the electronic spectra of Schiff base ligand (L) at these frequencies.

The Ni (II) complexes in $[\text{Ni}(\text{L})_2\text{Cl}_2]$ exhibits peaks at 10600 , 15700 , 24800cm^{-1} attributed to $3A_{2g}(\text{F}) \rightarrow 3T_{2g}(\text{F})$,

$3A_{2g}(\text{F}) \rightarrow 3T_{1g}(\text{F})$ and $3A_{2g}(\text{F}) \rightarrow 3T_{1g}(\text{P})$ [2] transitions respectively. [Table- 4]

Generally, Ni (II) complexes in $[\text{Ni}(\text{L})_2\text{Br}_2]$, & $[\text{Ni}(\text{L})_2\text{I}_2]$ exhibits peak at 10300 , 15300 , 24700cm^{-1} & 10700 , 15900 , 25200cm^{-1} respectively mentioned to transition [Table-4]. The magnetic moment value of Ni (II) complex is found to be 2.95, 2.90, 2.92 and falls within the range of 2.8-3.5 B.M for complexes proposing octahedral geometry, with sp^3d^2 hybridization. [13-23]

Table 1: - Physical and Analytical Data of Schiff Base Ligand[L] & its Complexes

Compound	Empirical Formula	Mol. Wt.	Colour	Yield %	Molar Conductance Mhocm ² mol ⁻¹
Schiff Base Ligand [L]	$\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$	242	Wine	65	----
$[\text{Ni}(\text{L})_2\text{Cl}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{Cl}_2]$	613.7	Green	58	23
$[\text{Ni}(\text{L})_2\text{Br}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{Br}_2]$	702.5	Brick Red	52	24
$[\text{Ni}(\text{L})_2\text{I}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{I}_2]$	796.5	Brown	47	21

Table 2: - Elemental % Composition of Elements Present in the Complex

Compound	Empirical Formula	Elemental % Composition Found (Calculated %)					
		Metal	C	H	N	O	Halogen
Schiff Base Ligand [L]	$\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2$	69.42 (69.40)	5.78 (5.69)	11.57 (10.98)	10.54 (12.97)
$[\text{Ni}(\text{L})_2\text{Cl}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{Cl}_2]$	9.54 (9.56)	54.69 (54.75)	4.52 (4.56)	9.08 (9.12)	10.39 (10.42)	11.54 (11.56)
$[\text{Ni}(\text{L})_2\text{Br}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{Br}_2]$	8.32 (8.35)	47.81 (47.82)	3.96 (3.98)	7.96 (7.97)	9.08 (9.11)	22.72 (22.74)
$[\text{Ni}(\text{L})_2\text{I}_2]$	$[\text{Ni}(\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_2)_2\text{I}_2]$	7.34 (7.35)	42.16 (42.19)	3.50 (3.52)	7.01 (7.02)	8.03 (8.04)	31.84 (31.85)

Table 3: FTIR- Spectral Data of Metal Complexes of $[\text{Ni}(\text{L})_2\text{X}_2]$ cm⁻¹

Compound	$\nu-(\text{C}=\text{N})$	$\nu-(\text{Pyridine})$	$\nu-(\text{OCH}_3)$	$\nu-(\text{CH})$ (Aromatic Benzene)	$\nu-(\text{M}-\text{N})$
Ligand, L	1677 (s)	1586 (s)	1269.6 (s)	2929 (b)	-
$[\text{Ni}(\text{L})_2\text{Cl}_2]$	1610 (s)	1516 (b)	1268.5 (w)	2927 (w)	440 (b)
$[\text{Ni}(\text{L})_2\text{Br}_2]$	1622 (s)	1513 (s)	1269.4 (s)	2926 (s)	435 (b)
$[\text{Ni}(\text{L})_2\text{I}_2]$	1601 (s)	1510 (s)	1268.7 (w)	2924 (w)	430 (s)

s= sharp, w= weak, b= broad; Where, X= Cl⁻, Br⁻, I⁻

Table 4: -Different Electronic Absorption Spectral Data and Magnetic Moment of Schiff Base Ligand and Its Metal Complexes

Compound	Absorption ν in cm^{-1} or (nm)	Transition	Magnetic Moment μ (B.M)	Geometry & Hybridization
Ligand, L	25126 cm^{-1} (398 nm)	$n \rightarrow \pi^*$	-----	-----
	42017 cm^{-1} (238 nm)	$\pi \rightarrow \pi^*$	-----	-----
[Ni(L) ₂ Cl ₂]	10600 cm^{-1} (943.4 nm)	$3A_{2g}(F) \rightarrow 3T_{2g}(F)$	2.95	Octahedral (sp^3d^2)
	15700 cm^{-1} (636.9 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(F)$		
	24800 cm^{-1} (403.2 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(P)$		
[Ni(L) ₂ Br ₂]	10300 cm^{-1} (970.87 nm)	$3A_{2g}(F) \rightarrow 3T_{2g}(F)$	2.90	Octahedral (sp^3d^2)
	15300 cm^{-1} (653.59 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(F)$		
	24700 cm^{-1} (404.86 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(P)$		
[Ni(L) ₂ I ₂]	10700 cm^{-1} (934.58 nm)	$3A_{2g}(F) \rightarrow 3T_{2g}(F)$	2.92	Octahedral (sp^3d^2)
	15900 cm^{-1} (628.93 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(F)$		
	25200 cm^{-1} (396.83 nm)	$3A_{2g}(F) \rightarrow 3T_{1g}(P)$		

**Table 5: - Microbial Study of the Complexes with reference to Ligand and Some Antibiotics
10<weak (W); >10 Moderate (M);>16 Significant (S)**

Compound	Escherichia Coli (E. coli.) (Gram negative)	Curd (Bacillus Subtilis) (Gram Positive)
Ligand	Resist (6) [W]	Resist [W]
[Ni(L) ₂ Cl ₂]	16 [S]	14 [M]
[Ni(L) ₂ Br ₂]	12 [M]	10 [M]
[Ni(L) ₂ I ₂]	10 [M]	11 [M]
Amikacin	18 [S]	17 [S]
Ceftriaxone	16 [S]	15 [M]
Gentamycin	Resist (6) [W]	Resist (7) [W]
Levofloxacin	12 [M]	11 [M]

HRMS: -

The mass spectra of DBAP ligand are illustrated in the Fig.-7 The peak at m/z 243.11 signifies molecular ion peak $[M+H]^+$ for DBAP Ligand. In addition, other peaks for the ligand are observed at m/z - 244.11, 276.13, 275.13, 181.08 and 139.07 with significant intensity and they correspond to fragment peaks.

The metal complexes viz. $[\text{Ni}(\text{DBAP})_2\text{Cl}_2]$, $[\text{Ni}(\text{DBAP})_2\text{Br}_2]$, and $[\text{Ni}(\text{DBAP})_2\text{I}_2]$ delivers respective molecular ions peak at m/z 614.59 $[M+H]^+$, 703.49 $[M+H]^+$,

and 820.48 $[M+Na]^+$ (Fig. 8-10) that strongly support the expected molecular formula of the complex.

The other fragments of $[\text{Ni}(\text{DBAP})_2\text{Cl}_2]$ are m/z - 677.06, 514.94, and 348.83. The other fragments of $[\text{Ni}(\text{DBAP})_2\text{Br}_2]$ are m/z - 522.99, 376.92 (Fig.9) The other fragments of $[\text{Ni}(\text{DBAP})_2\text{I}_2]$ are m/z - 547.13, 428.03.

All these data of the molecular ion and fragments peak nicely co-relates the proposed molecular geometry of the compound.

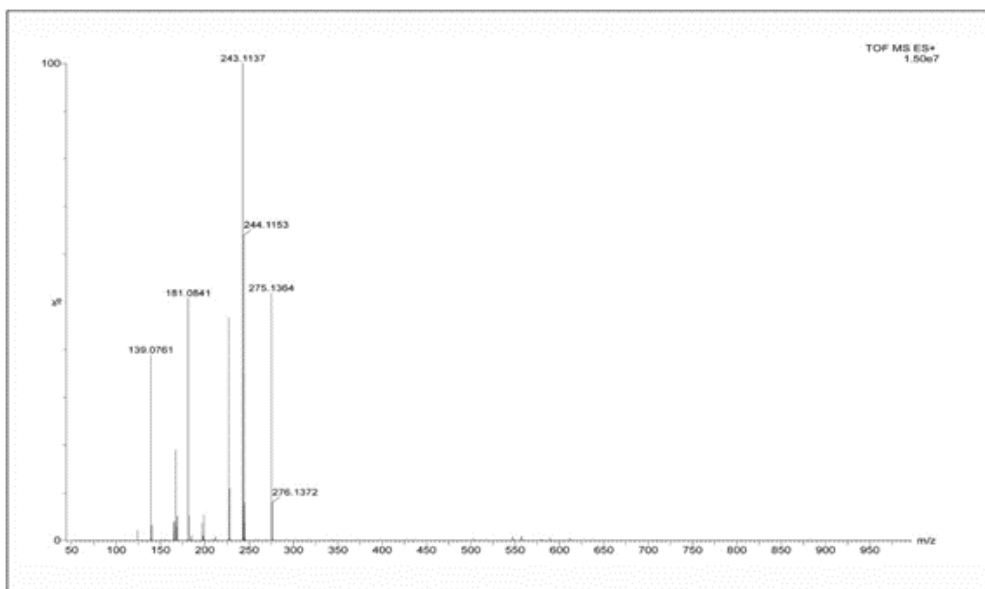


Fig 7:- HRMS of Ligand (DBAP)

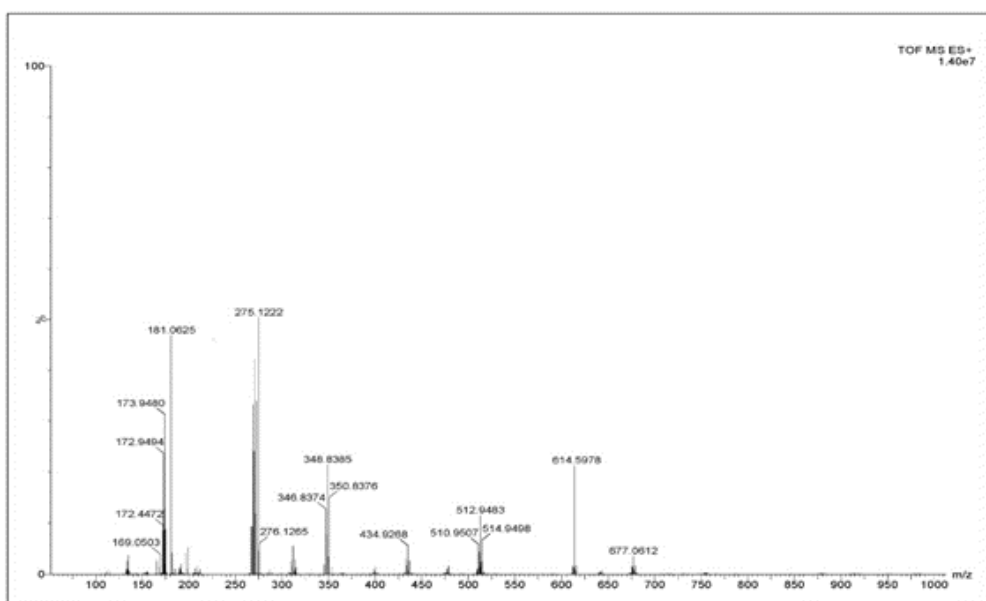


Fig 8:- HRMS of [Ni(DBAP)₂Cl₂]

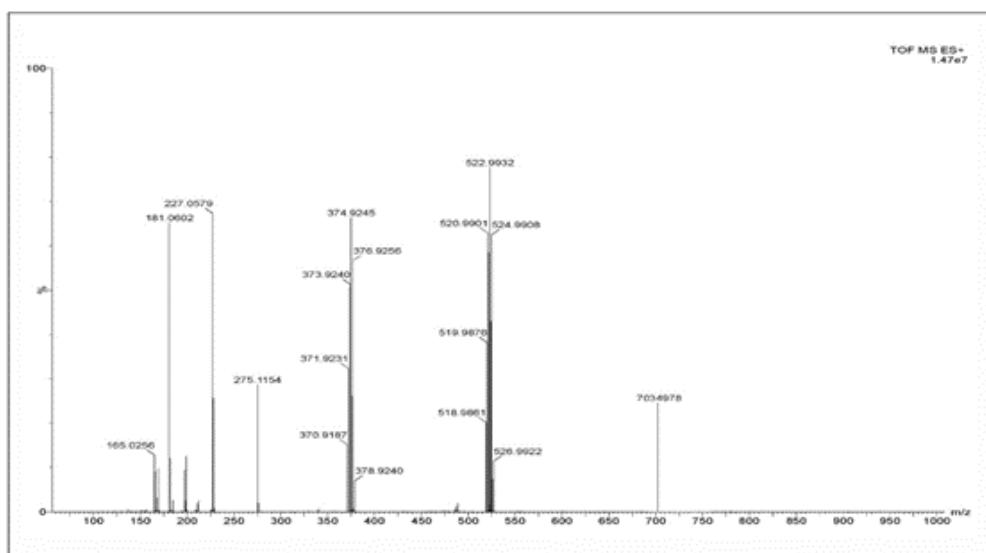
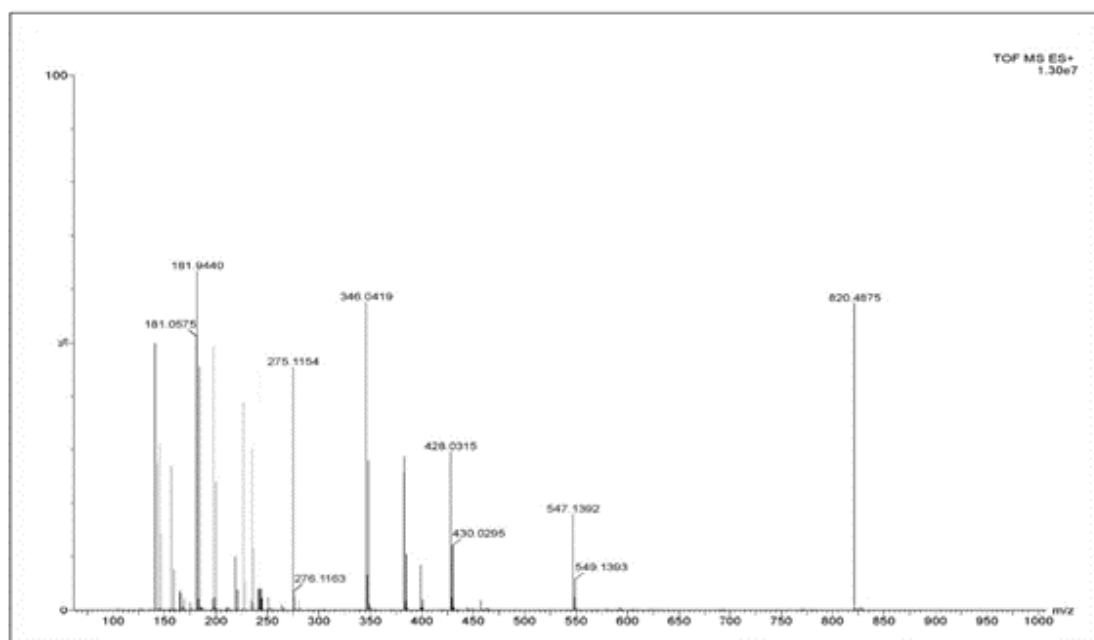
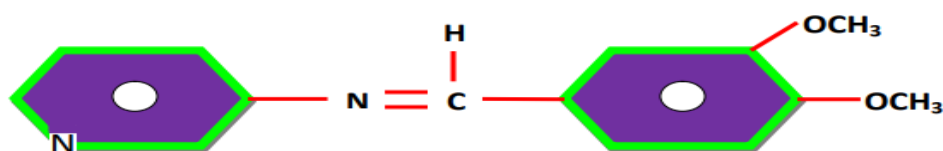


Fig 9:- HRMS of [Ni(DBAP)₂Br₂]

Fig 10:- HRMS of $[\text{Ni}(\text{DBAP})_2]_2$

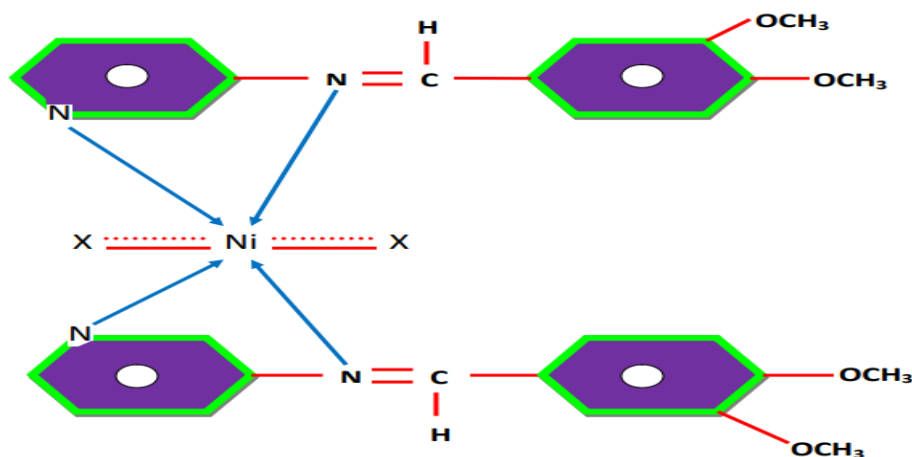
Proposed Structure of Ligand: - From the above Experimental data, Fig. Table, the proposed structure of Ligand has been depicted in Fig 11.

Fig 11: - Proposed Structure of Ligand [L], $[\text{N}-(3,4\text{-dimethoxybenzylidene})\text{-3-amino Pyridine}]$

Structure of Ligand [Fig. 11] reveals that it has four donor sites i.e., two are on one side and other two are on other side i.e., pyridine Nitrogen, and azomethine Nitrogen having property of potential donor while other two oxygen atoms of methoxy group have comparatively low capacity on the arrangement of donor atom. It is clear that two donor sites are available in one condition meaning ligand may co-ordinate

through pyridine Nitrogen and azomethine (imine) Nitrogen, so ligand behave as bidentate.

FTIR, HRMS, , Elemental analysis, U-V electronic spectra revealed that the ligand behaves as bidentate coordinating through imine group and pyridine Nitrogen having proposed octahedral geometry [Fig.12] with sp^3d^2 hybridization as shown below.

Fig 12: - Proposed Structure of $[\text{Ni}(\text{L})_2 \text{X}_2]$ [Where $\text{X} = \text{Cl}^-$, Br^- , I^-]
Shape: - Octahedral, Hybridization: - sp^3d^2

Conclusion: -

Metal complexes of Ni (II) and DBAP ligand have been characterized structurally. The analytical data shows that the metal ligand stoichiometry in complex is 1:2. All complexes are non-electrolyte in DMSO. The ligand (L) here is bidentate which is governed by the spectral data. Ligand (L) coordinates

through N-atom of the imine group and N-atom of the pyridine ring. Ni(II) complexes have octahedral geometry which is advocated by the analytical data, conductance, magnetic and electronic spectral data. Ni(II) complexes had better activity when compared to the ligand which was proved by biological studies and the results were very closer to the antibiotics used.

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