



# Synthesis & Characteristics of schiff base, transition metal complexes

DAYA VEER and MUKESH BABOO

Department of Chemistry, Hindu College, Moradabad- 244 001, India.

Corresponding Author E-mail : dayaveergangwar@gmail.com

## ABSTRACT

The schiff-base in metal complexes N, N' Propylene bis-2-amino benzamide was prepared & characterized its complexes with transition metal Ti, Mo, Mn, V, & Co were prepared & characterised by elemental analysis molar conductivity magnetic moment - T.G.A., IR & electronic spectra.

## Introduction :

Schiff bases are an important class of ligands in coordination chemistry and their complexing ability containing different donor atoms is widely reported<sup>1,2</sup>. The chemistry of Transition metal complexes containing schiff bases continues to be of interest on account of their interesting structural features and also because of their biological importance<sup>3,4</sup>.

The coordination complexes of the schiff bases have been widely investigated due to their manifestation of novel structural features, unusual magnetic properties and relevance to biological process<sup>5-7</sup>.

The polydentate schiff bases are well known to coordinate with various metal ions to form metallic complexes with theoretical and practical application of different type<sup>8</sup>. These ligand systems have gained considerable importance because of their utility as model compounds in bioinorganic studies as well as their potential as

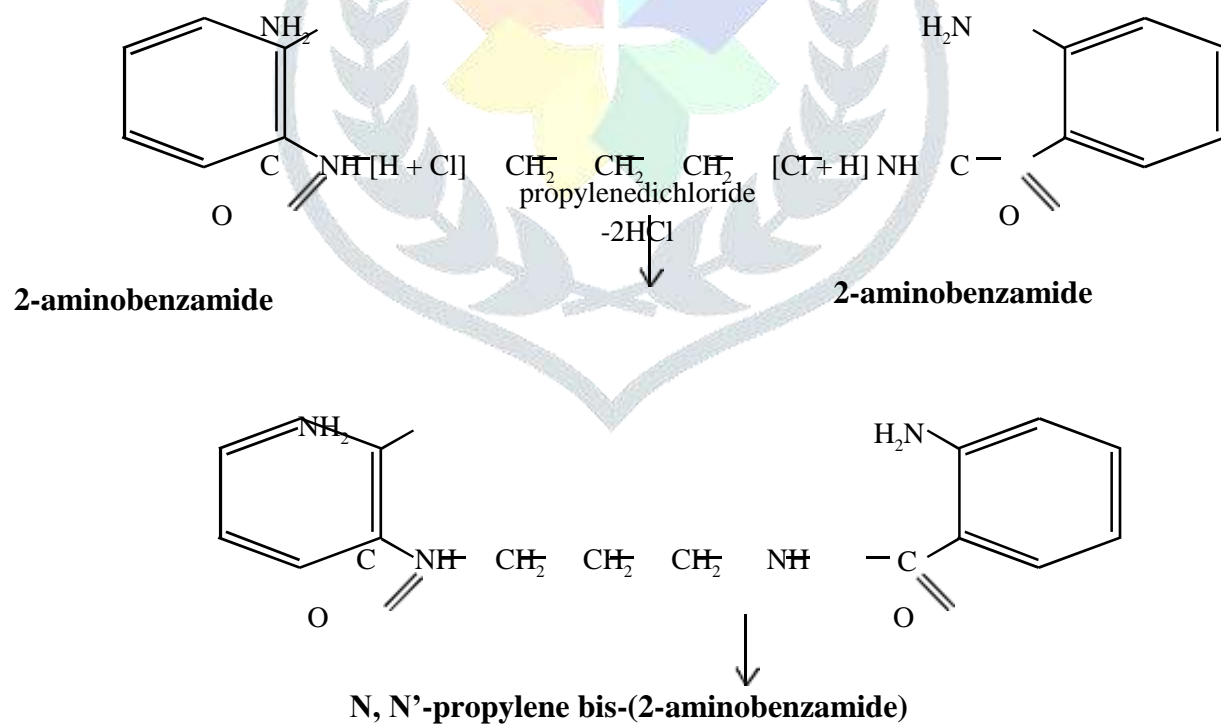
catalysts in many reactions<sup>9</sup>. They find application in the determination of transition metal ions<sup>10-13</sup> in the study of acid base equilibrium acidity constants value<sup>13</sup> & for analytical purposes<sup>15</sup>.

### Experimental :

All the chemicals and reagents used for this research work were of highest purity or A.R. Grade. The chemicals used were 2-aminobenzamide and ethylene dichloride. The solvents used were methanol, ethanol and DMF. Ethanol was double distilled in the lab before use whereas other solvents were used as procured. The metal used were Ti, Mo, Mn, V & Co, Ti(III) chloride was prepared in the lab by the reported method while other the metal facts were used as procured.

### Preparation of the ligand :

1 mole of propylene dichloride & 2 mole of 2-aminobenzamide were dissolved in methanol and refluxed for 3 hr. The solid product so obtained was filtered, washed with methanol and dried in vacuum desiccator.



Name of Molecular formula of schiff base	M.W.	Colour	M.P.	Elemental Analysis Calc.		
				%C	%H	%N
N,N'-propylene bis-(2-amino-benzamide)	312	Yellow	202 <sup>o</sup>	65.38 (65.33)	6.41 (6.37)	17.95 (17.91)
(C <sub>17</sub> H <sub>20</sub> N <sub>4</sub> O <sub>2</sub> )						

### **Preparation of the complex :**

The complex of Ti(III) chloride were prepared globe bag while all other metal complexes were prepared under normal condition. N, N' - ethylene-bis-(2-aminobenzamide) was dissolved in methanol, the pH of the solution was adjusted to 6.0 by adding methanolic solution of NaOH. The metal salt was dissolved in methanol and gradually added to the solution of the ligand with constant stirring. The microcrystalline complexes formed were suction filtered and washed with hot methanol several times. The products were dried in vacuo over anhydrous calcium chloride.

### **Characterization of Ligand and Metal Complexes**

The colour of the ligand and its metal complexes were noted. The m.p. of the ligand and its complexes were determined by open capillary method and are uncorrected. The ligand and its complexes were subjected to elemental analysis for C, H and N whereas metal was estimated gravimetrically in the lab. The electronic spectra of the complexes were recorded with the help of Beckman DU. The IR spectra were recorded at CDRI, Lucknow in KBr phase.

### **Result and Discussion**

The Ti(III) complexes was sensitive to Air, while all other metal complex were non-hygroscopic & static in Air. The complexes were found to be Insoluble in water & common organic solvents, but, soluble in DMF, DMSO & Acetoxitive. The analytical data suggested 1 : 1 metal : ligand stoichiometry for the complexes. The molar conductance values of the complexes suggested 1 : 3 electrolytic nature for trivalent metal ion.

### **Magnetic moment & electronic spectra**

All the complexes were paramagnetic in natural except cobalt complex, which was diamagnetic in nature.

The magnetic moment Ti(III) complex was 1.71 B.M.. It is indicated paramagnetic nature of the complex & also suggested that Ti (III) has not been

oxidised to Ti (IV) ruring or after complexation.

The electronic spectrum of the complex exhibited a single band at  $19440\text{ cm}^{-1}$  derived from  ${}^2T_{2g} \rightarrow {}^2E_g$  transition for an octahedral geometry. This band is unsymmetrical in shape and is indeed made up of two closely shaped bands. The second band appears as a hump and is due to presence of Jahn-Teller distortion.

The IR spectra of the ligand shows three bands at  $3465$ ,  $3360$  and  $3280\text{ cm}^{-1}$  which may be assigned to N-H stretching frequencies which has shifted by  $30$ ,  $40$  and  $50\text{ cm}^{-1}$  in the IR spectrum of the complex. These shift suggest coordination through both amine N-atom of the ligand with the metal.

The other important band at  $1540\text{ cm}^{-1}$  may be due to amide (II) absorption [ $\nu(\text{C-N}) + \delta(\text{N-H})$ ]. This band has shifted by  $45\text{ cm}^{-1}$  the complex, indicating coordination through amide N-atom.

The amide carbonyl absorption  $\nu(\text{C=O})$  at  $1645\text{ cm}^{-1}$  remained unaffected in the complex and indicates non-coordination of carbonyl oxygen.

These coordination sites are further supported by the appearance of new band in far IR region of the complex at  $470\text{ cm}^{-1}$  assignable to  $\nu(\text{M-N})$  frequency.

The IR spectrum of the complex shows two new bands at  $3510$  ( $\nu\text{O-H}$ ) and  $845\text{ cm}^{-1}$  (rocking mode) of coordinated water molecule.

The magnetic susceptibility of the complex was determined by Gouy- balance using  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  as calibrant and diamagnetic correction for the ligand was made. The spin only value of the magnetic moment was calculated from the observed magnetic susceptibility which comes out to be  $1.79\text{ B.M.}$  This value is normal and indicates the absence of any Mo-Mo interaction.

The electronic spectral bands of the present oxomolybdenum (V) complex show similar absorption peaks. A moderately intense band observed in the region of  $25000\text{ cm}^{-1}$  attributed to  $O(\pi) \rightarrow (Mo)$ . The band due to transition  ${}^2B_1 \rightarrow {}^2A_1(dx^2 - dy^2 \rightarrow dxz)$  is probably

masked by the above bands. The complex exhibits two more bands. A moderate intensity band at  $19310\text{ cm}^{-1}$  and a weak broad band spread over the region  $12910\text{ cm}^{-1}$  assigned to  ${}^2B_{1g} \rightarrow {}^2B_{2g} (dxy \rightarrow dx^2 - y^2)$  and  ${}^2B_{2g} \rightarrow 2E_g (dxy \rightarrow dxz, dyz)$  respectively. The unpaired electron is in the dx orbital. The spectral bands at  $12910\text{ cm}^{-1}$  indicates octahedral environment for the complex.

The room temperature molar conductance value in DMF at  $10^{-3}\text{ M}$  dilution suggested 1 : 3 electrolytic nature of the complex. Magnetic moment of complex is 5.40 B.M. revealing the high spin nature of the complex corresponding to four unpaired electrons.

The electronic spectrum of Mn(II) complex showed an intense and sharp charge transfer band at  $19800\text{ cm}^{-1}$  and a shoulder at  $18230\text{ cm}^{-1}$  and may be assigned to the  ${}^5E_g \rightarrow {}^5T_{2g}$  transition, characteristics of octahedral geometry.

The value of magnetic moment of the complex is 2.92 B.M. which is in accord with the presence of two unpaired electrons and suggested paramagnetic nature for the complex.

The electronic spectrum of V(III) complex exhibits band at  $16125\text{ cm}^{-1}$  with a shoulder at  $20410\text{ cm}^{-1}$ . The low energy band has been assigned to  ${}^2A_{1g} \rightarrow {}^3A_{2g}$  while the high energy band may be due to  ${}^2A_{1g} \rightarrow {}^3T_{2g} (P)$  transition. These bands are characteristic of octahedral geometry.

The study of magnetic properties of complex indicated diamagnetic nature of the metal chelate, which is expected for low spin d6 ion like Co(III). The electronic spectrum of the complex exhibits three bands at  $15250, 21210$  and  $23530\text{ cm}^{-1}$ . These bands have been assigned to  ${}^1A_{1g} \rightarrow {}^3T_{1g}, {}^1A_{1g} \rightarrow {}^1T_{1g}$  and  ${}^1A_{1g} \rightarrow {}^1T_{2g}$  transitions respectively for other Co(III) complexes and are characteristic of octahedral geometry.



**Molar Conductance :**

The molar conductance at  $10^{-3}$  M dilution & room temperature suggested 1 : 2 electrolytic nature for Ti(III) oxo-molybdenum (VI), Mn(III), V(III) & Cr(III) complexes.

**IR Spectra :**

The IR spectra of the ligand shows three bands at 3465, 3360 and 3280  $\text{cm}^{-1}$  which may be assigned to N-H stretching frequencies which has shifted by 30, 40 and 50  $\text{cm}^{-1}$  in the IR spectrum of the complex. These shift suggest coordination through both amine N-atom of the ligand with the metal.

The other important band at 1540  $\text{cm}^{-1}$  may be due to amide (II) absorption [ $\nu(\text{C-N}) + \delta(\text{N-H})$ ]. This band has shifted by 45  $\text{cm}^{-1}$  the complex, indicating coordination through amide N-atom.

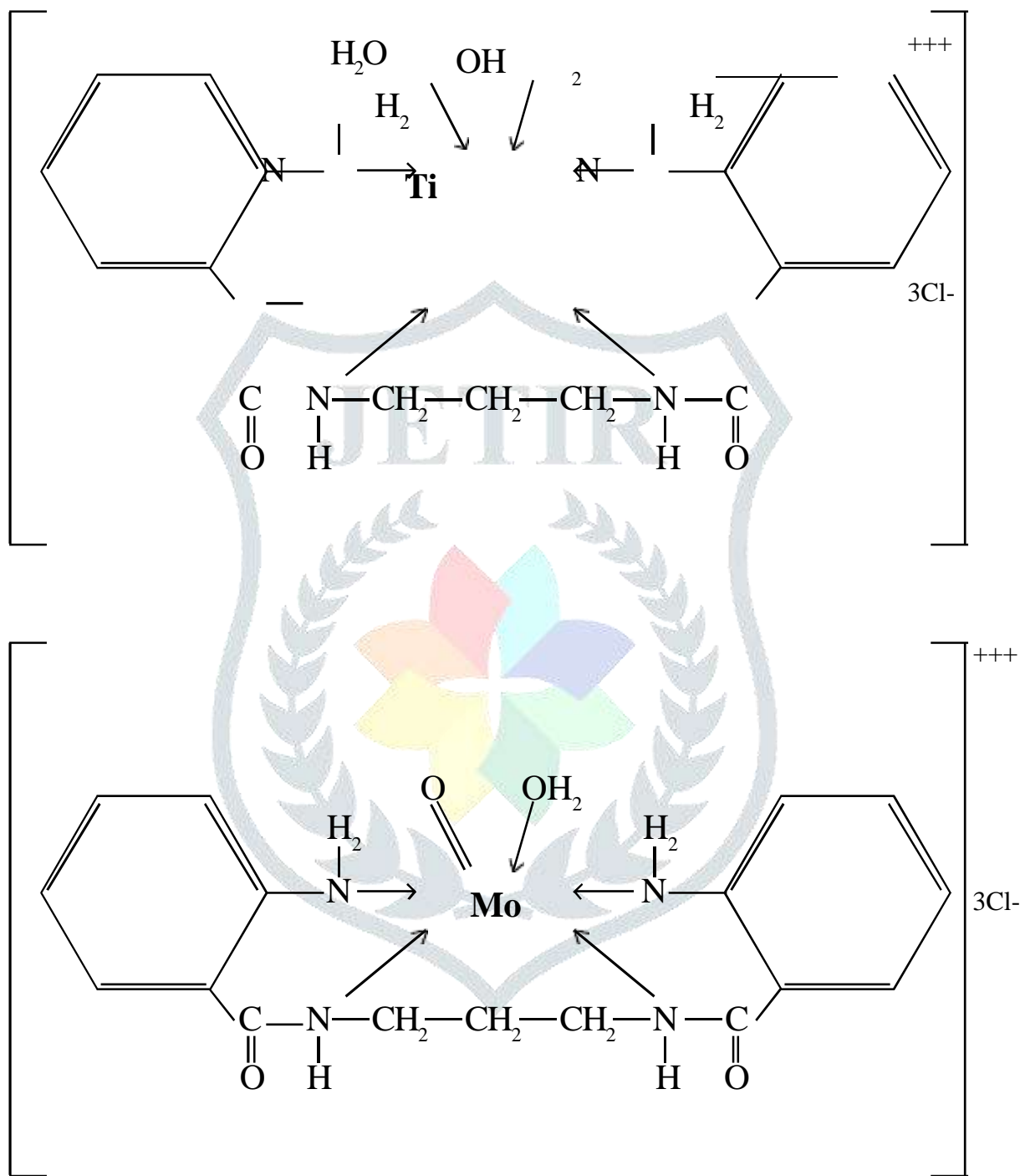
The amide carbonyl absorption  $\nu(\text{C=O})$  at 1645  $\text{cm}^{-1}$  remained unaffected in the complex and indicates non-coordination of carbonyl oxygen.

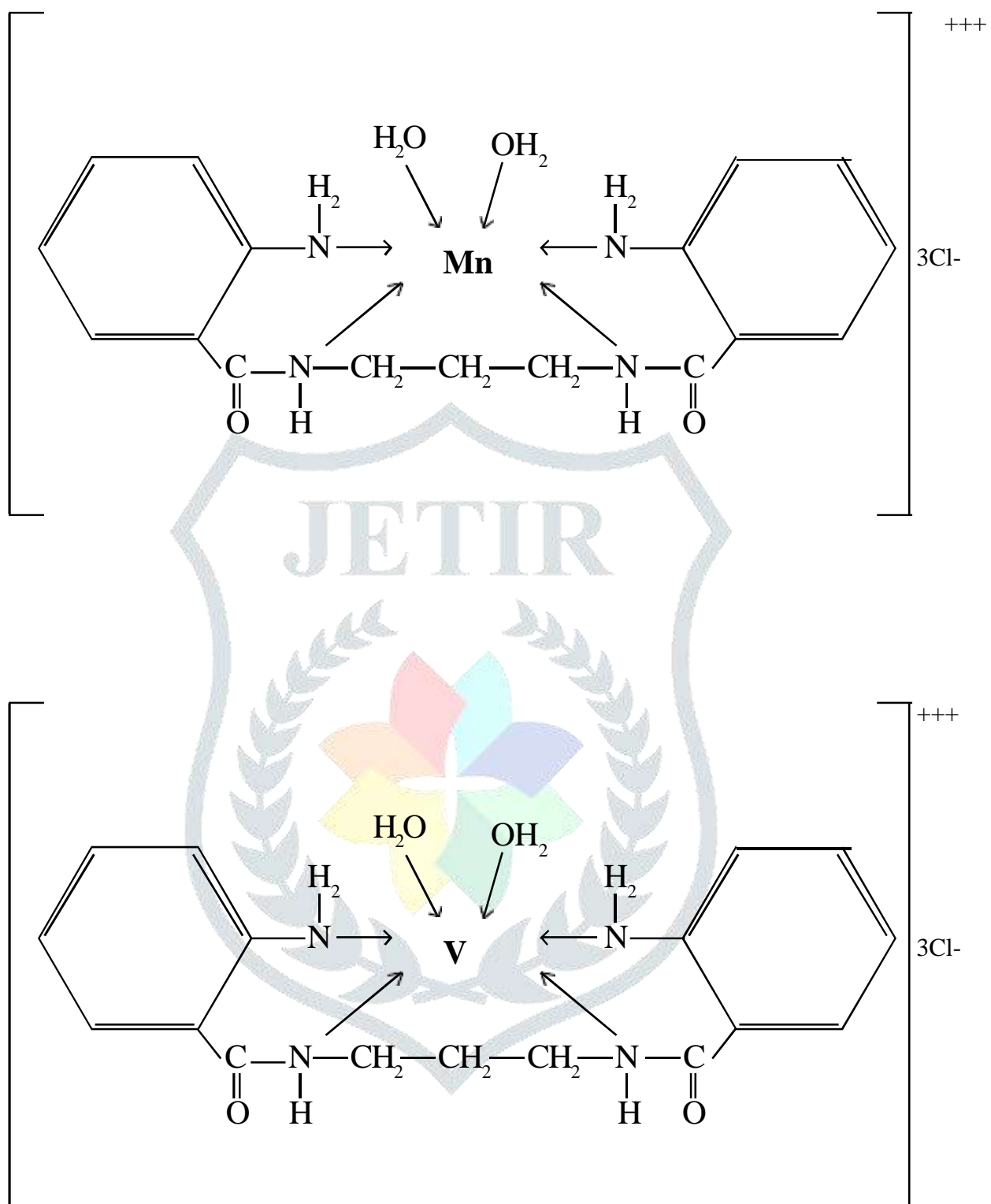
These coordination sites are further supported by the appearance of new band in far IR region of the complex at 470  $\text{cm}^{-1}$  assignable to  $\nu(\text{M-N})$  frequency.

The IR spectrum of the complex shows two new bands at 3510 ( $\nu\text{O-H}$ ) and 845  $\text{cm}^{-1}$  (rocking mode) of coordinated water molecule.

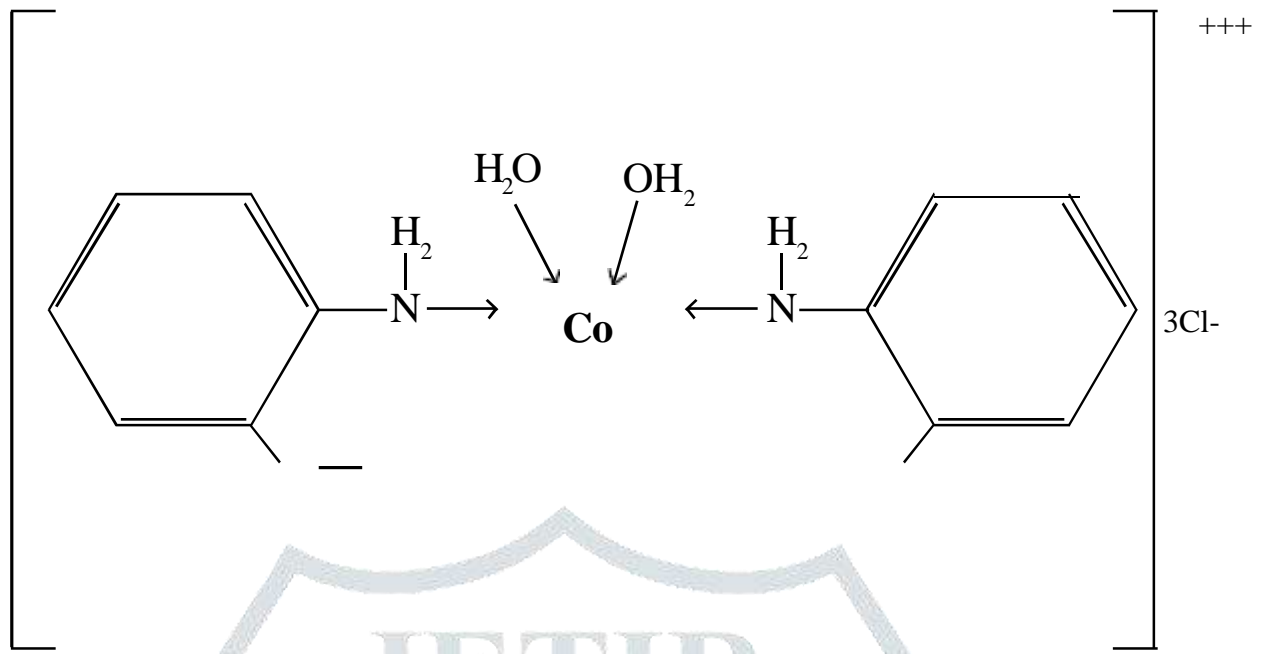
In the case of oxomolybdenum (VI) complexes a new band appeared at 930  $\text{cm}^{-1}$  in the IR spectrum of the complex which may be due to  $\nu(\text{Mo=O})$  1801.

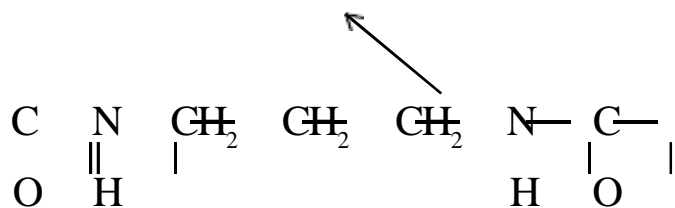
The analytical data of the complex suggested 1 : 1 M : L ratio for all the complex. The ligand has behave, as quadridentate coordinating through two amide Nitrogen atoms & two Amine Nitrogen atoms.











### Conclusion :

On the basis of studies performed octahedral geometry has been proposed for all the synthesised complexes with positive distortion in the case of oxo- dioxo, molybdenum complexes due to Mo = O.

### Reference

1. Isac Sobana Raj, C.M. Sofia and M. Antilin Princela, Synthesis and Characterization of Bioactive Transition Metal Complexes from Cardanol, *Asian J of Research in Chemistry*, 7(8), 2014, 711-716
2. Blessy. C, Isac Sobana Raj C, and Allen Gnana Raj G, Synthesis characterization and Biological Activities of Co(II), Ni(II) & Cu(II) Complexes with DFMPM and glycine, *Der Pharma Chemica*, 8(18), 2016, 364-373
3. Bismi S Prakash, Synthesis and Characterization of Bio Active Transition Metal Complexes of Cu (II) Co (II) and Ni (II) using Natural Sources, *Asian Journal of Research in Chemistry*, 8(12), 2015, 726- 732
4. Isac Sobanaraj.C and Anusha, Synthesis and Characterization of Transition Metal Complexes of Cu(II), Co(II) and Ni(II) using Cardanol and 4-aminoantipyridine, *Journal of chemical and pharmaceutical research*, 7(11), 2015, 485-493
5. C. Isac Sobana Raj, G. Allen Gnana Raj and M. Antilin Princela, Synthesis and Characterization of Bioactive Transition Metal Complexes of Zr(IV) and Th(IV) using di-±-formylmethoxy bis (3-penta decenyl phenyl) methane (DFMPM) derived from Cardanol, *AJCPR*, 3(1), 2015, 208–214

6. Hermansen Ralph D. and Lau Steren E., Adhesive of epoxy resin, amine-terminated resin and conductive filler, *US. Patent 5929141 A*, 8/9011, 2004, 153 International Journal of Scientific and Research Publications, Volume 7, Issue 10, October 2017 19 ISSN 2250-3153 www.ijsrp.org
7. Bismi S. Prakash, Isac Sobana Raj. C, M. Stalin Joseph, Synthesis and Characterization of Bioactive Transition Metal Complexes of Zr(IV) and Th(IV) using natural sources, *Int. J. Res. Chem. Environ. Vol. 6(3),2016, (30-39)*
8. Bismi S Prakash, Isac Sobana Raj. C, Allen Gnana Raj. G, Synthesis, Characterization and Anti Cancerous Studies of Schiff Base Complexes of Co(II), Cu(II) And Ni(II) Using DFMPM and L-Histidine, *Int. Journal of Engineering Research and Application, 7(7, [ Part -9])2017,38-49.*
9. Faridul Islam, Md. Amran Hossain, Nur Mostaq Shah, Hridika Talukder Barua, Md. Alamgir Kabir, Mohammad Jamshed Khan, and Romel Mullick, Synthesis, Characterization, and Antimicrobial Activity Studies of Ni(II) Complex with Pyridine as a Ligand, *Journal of Chemistry (Hindawi), 2015(2015).*
10. Maged S. A. (2018). Synthesis and Characterization of Coordination Polymers of 1, 3-Di (4-Pyridyl)-Propane And 2-Aminobenzothiazole with Mn(II), Co(II), Cu(II) and Ni(II) Ions. *Journal of Chemical and Pharmaceutical Research. 10(2), 77-83.*
11. a) F.G. Montelongo, V.G. Diaz and C.R.T. Gonzalez *Microchem. J. 27: 194 (1982).*  
b) F. Kai, Y. Sakanashi, S. Satoh and S. Uchikawa, *Anal, Lett., 16(A13) : 1013 (1983).*
12. M.I. Toral, P. Richter, N. Lara, M.T. Escudero and C. Soto *Anal, Lett., 33:93 (2000).*
13. D.K. Singh and R. Kumar, *Orient. J. Chem. 27(1) : 277-281 (2011).*
14. V.G. Diaz C.R.T. Gonzalez and F.G. Montelongo, *Anales de Quimica, 79 : 83 (1983).*
15. J.J. Arias, F. Jimenez, A.I. Jimenez and F.G. Montelongo, *Anales. De Quimica, 79 : 248 (1983).*

16. N.K. Singh and Kushwaha, Indian Journal of Chemistry, 43A : 1454-1458 (2004).
17. Islam M.S. and Alam M.A., J. Indian Chem. Soc., 76 : 2 (1999).
18. Kolawola G.A., Synth React to Inorg Met Org Chem 23 : 907 (1993).
19. Patel I.A., Thaker B.T. & Thaker P.B., Indian J. Chem., 37A : 429 (1998).
20. Lever ABP., Inorganic Electroni spectroscopy (Elsevier Amsterdam) pp. 275-77 (1984).
21. Rahul Kumar Rastogi, Poonam Garg and Shamim Ahmad, Asian Journal of Chemistry, 21(8) : 6144-6148 (2009).
22. Mohammad Azim and Shamim Ahmad, Oriental Journal of Chemistry, 27(2) : 673-677 (2011).
23. T. Daniel Thangadural & K. Natarajan, Indian Journal of Chemistry, 41A : 741-745 (2002).
24. P. Viswanathamurthia, R. Karvembub, V. Thahraneeswaranb and K. Natarajan\*, J. Chem. Soc. 117(3) : pp-235-238 (2005).
25. Swamy SJ & Bhaskar K. Indian J. Chem., 38-A:961 (1999).
26. Clark RH & Wagner EC, J. Org. Chemistry, 9:55 (1944).
27. Swamy SJ & Kumar BK, Indian J. Chem., 35A : 489 (1996).
28. Swamy SJ Kumar BK & Dharmapuri Y, Indian J. Chem., 34A : 811 (1995).
29. S.J. Swamy, A. Dharma Reddy & K. Bhaskar, Indian Journal of Chemistry, 40 : 1166-1171 (2001).
30. Sobhanadevi G & Indrasenan P. Inorg. Chin. Acta, 133 : 157 (1987).
31. Prabhakaran CP & Nair BG, Trans. Met. Chem., 8 : 368 (1983).