

Synthesis & Spectral studies of some Co-ordination compounds of Cobalt(II) & Nickel (II) with Schiff's base ligand derived from Carbonyl and amino Hydroxamic acid

Dr. Rita Kumari

Government (+2) High School, Dwarikanagar, Mushahari, Muzaffarpur, Bihar.

Abstract : A Novel series of Co-ordination compounds of Cobalt(II) & Nickel(II) of the type ML_2B_2 (where M = Co(II) & Ni(II), B = Bases like water, ammonia, Pyridine, Quinoline etc, and L = R² (3-methoxy-4-hydroxy benzidine imino benzhydroxamic acid) have been synthesised by general procedure. All such co-ordination compounds are found stable under normal conditions & possess characteristic colour. Their structure have been elucidated on the basis of analytical, infrared, electronic spectral data and magnetic susceptibility value. Molar conductance data of the co-ordination compounds are indicative of their non-ionic nature. The molecular weight determination of the complexes also indicate their monomeric nature infrared spectra shows that ligand acts as bidentate co-ordinating to the metal ion through two imine nitrogen in all complexes. The complexes are found to be paramagnetic with octahedral arrangement of ligand atoms around the metal ions.

Keywords : 3-methoxy-4-hydroxy benzaldehyde, imine nitrogen, 2(3-methoxy-4-hydroxy benzidine) imino benzhydroxamic acid, 2-aminobenz hydroxamic acid.

I. INTRODUCTION

Schiff's bases arise from aminohydroxamic acids & their derivatives and carbonyl compounds have been extensively studied as a consequence of their biological importance which is related with their ability to form metal ion complexes. These are important due to their pharmacological toxicological, pathological and analytical properties^{1,2,3}. After deprotonation, they act as bidentate ligand & co-ordinate to metal ions through azomethine nitrogen forming octahedral complexes.

In our present work, the ligand 2(3-methoxy-4-hydroxy benzylidene) imino benz hydroxamic acid have been synthesised by the condensation of 2-amino benzhydroxamic acid & 3-methoxy-4-hydroxy benzaldehyde. This ligand behaving as Schiff base forms complexes with metal ions like Co(II) & Ni(II). The co-ordination sites of the metal ion are well satisfied by the ligand as well as water & nitrogen donor molecules to form different complexes.

II. EXPERIMENTAL

All the chemicals used in the present work were of E-merch extrapure or BDH (AR) quality. All the complexes have been prepared under similar conditions. The compounds were analysed for different constituents by the standard procedure⁴. Magnetic moments were determined by Gouy method. The analytical data is recorded in Table-1&2 Relevant bonds in electronic spectra and room temperature magnetic moment values & colour are recorded in Table - 3, 4, 5 & 6. A typical preparation of ligand & complexes described here as :

Table-1

Analytical data of [Co(L)₂B₂]

Complexes	Metal	C	H	N
1. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	8.76 (8.85)	54.08 (54.14)	4.55 (4.51)	8.45 (8.42)
2. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	8.80 (8.88)	54.22 (54.30)	4.85 (4.82)	12.70 (12.67)
3. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	6.98 (7.05)	63.14 (63.24)	4.35 (4.31)	10.10 (10.06)
4. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	6.56 (6.64)	56.80 (64.94)	4.54 (4.51)	9.50 (9.47)
5. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	7.40 (7.48)	60.85 (60.99)	4.60 (4.57)	10.70 (10.67)
6. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α-picoline)	7.15 (7.22)	61.74 (61.84)	4.92 (4.90)	10.32 (10.30)
7. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β-picoline)	7.14 (7.22)	61.76 (61.84)	4.95 (4.90)	10.35 (10.30)
8. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ-picoline)	7.16 (7.22)	61.72 (61.84)	4.93 (4.90)	10.31 (10.30)

Table-2

Analytical data of [Ni(L)₂B₂] Complexes Found (calculated) → %

Complexes	Metal	C	H	N
1. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	8.75 (8.83)	54.08 (54.15)	4.54 (4.51)	8.45 (8.42)
2. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	8.74 (8.85)	54.30 (54.32)	4.86 (4.82)	12.72 (12.67)
3. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	6.96 (7.05)	63.20 (63.24)	4.35 (4.31)	10.12 (10.06)
4. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	6.56 (6.62)	64.81 (64.95)	4.56 (4.50)	9.52 (9.47)
5. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	7.38 (7.46)	60.90 (61.01)	4.62 (4.57)	10.74 (10.67)
6. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α-picoline)	7.10 (7.20)	61.74 (61.86)	4.96 (4.90)	10.35 (10.31)
7. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β-picoline)	7.06 (7.20)	61.77 (61.86)	4.94 (4.90)	10.38 (10.31)
8. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ-picoline)	7.12 (7.20)	61.70 (61.86)	4.93 (4.90)	10.32 (10.31)

Preparation of 2(3-methoxy-4-hydroxy benzilidene) iminobenz hydroxamic acid (LH)

An ethanolic solution of 3-methoxy-4-hydroxy benzaldehyde (3.04 gm, 0.2 mol) was mixed with 2-amino benz. hydroxamic acid (0.2 mol) in same medium and the resulting solution was refluxed at water bath for about two hours at room temp. The resulting solution produced faint orange colour solid on cooling in the freezer.

The m.p. was obtained to be 54^oC.

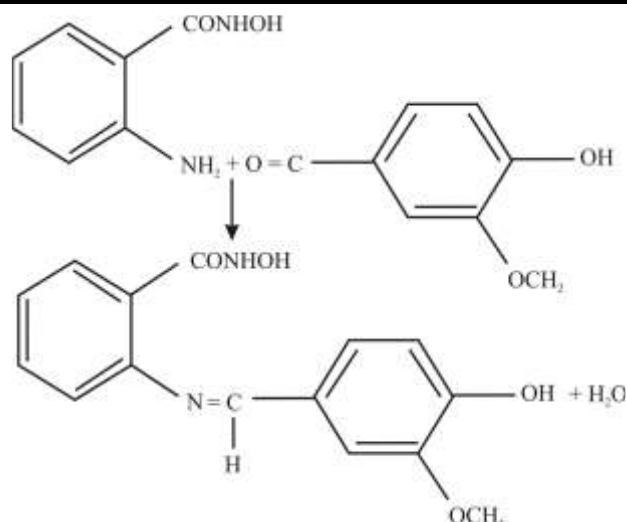


Fig.-1

Preparation of Complexes

The ethanolic solution of ligand was added to the alcoholic solution of Co(II) or Ni(II) Salt (in 2:1 ratio) with regular and gradual shaking and stirring in each case with water or nitrogen containing bases. The resulting solution was then refluxed for an hour on water bath at room temperature. The colour of the solution was gradually changed and crystals of different colours separated out by allowing the solution to stand over night. The product was separated by filtration, washed with small amount of acetone and then dried over KOH in degicator.

III. RESULTS AND DISCUSSION

IR Spectra : The IR spectra of complexes & ligands were recorded in nujol mull on Perkin Elemer 577 spectrophotometer.

A perusal of IR spectra of the ligand and the complexes shows that there are certain bands of the ligand which are appreciably affected after the complex formation and these groups are the co-ordination sites at the ligand to form complexes with the metal ions.

IR Spectra of ligand & complexes

	Groups	Frequencies in ligand	Frequencies in complexes
1.	ν_{OH}	3540 -3550	3540-3550
2.	$\nu_{(OCH_3)}$	2800	2800
3.	$\nu_{C=N}$	1660-1640	1620-1610
4.	ν_{N-OH}	1080-1070	1110-1120

The IR spectral bands due to the vibrations of ν_{OH} & $\nu_{(OCH_3)}$ groups are the same in the ligand and in the complexes which rules out the co-ordination of these groups in the bond formation.

The IR spectral bands of $\nu_{C=N}$ & ν_{H-OH} groups are found to be changed in the ligand and in the complexes. There is a lowering in frequency of ν_{CN} group by 40-30 cm^{-1} indicating co-ordination of the nitrogen atom of azomethine group $\nu_{C=N}$ in the bond formation. The frequency of $\nu_{(N-OH)}$ group is raised upto by 35-25 cm^{-1} in the complexes suggesting the diprotonation of N-OH hydrogen and co-ordination of hydroxamino nitrogen atom in the bond formation with the metal ions.

The co-ordination of nitrogen atom of azomethine $>C=N$ group & hydroximino nitrogen atom is further confirmed due to the appearance of a weak & sharp band in the region of $440-455\text{ cm}^{-1}$ which was assigned to be due to ν_{M-N} vibrations.

The frequencies obtained in the range of $700-800\text{ cm}^{-1}$ in aquo the complexes may be attributed as due to rocking mode of co-ordinated water. The pyridine complexes show some extra band in finger print & far infra red region. The pyridine ring breathing vibration located around $1000-1040\text{ cm}^{-1}$ in the complexes has been taken to be the characteristics vibrations of co-ordinated pyridine molecule. IR band around $590-600\text{ cm}^{-1}$ in the complexes are also characteristic of presence of pyridine molecule in the complexes. In case of phenyl isocyanide complexes, a band around 2090 cm^{-1} is obtained indicating the participation of nitrogen atom in the complex formation.

Magnetic Susceptibility : Magnetic susceptibilities of the complexes were determined by Gony's method by using $[\text{Hg}\{\text{CO}(\text{SCN})_4\}]$ as a calibrant.

The value of magnetic moment obtained from magnetic susceptibility measurement for the CoL_2B_2 complexes in the range of $4.90-5.20\text{ BM}$ & for the complexes NiL_2B_2 in the range of $2.9-3.4\text{ BM}$ indicate the octahedra geometry for the complexes.

Electronic spectra⁵ :

The electronic absorption spectra of the complexes were recorded with Hitachi-320 spectrophotometer. The electronic spectra for cobalt complexes CoL_2B_2 shows three spectra bands at $8360-8600\text{ cm}^{-1}$, $16800-17400\text{ cm}^{-1}$ and $20750-21500\text{ cm}^{-1}$ corresponding to the transitions ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$, ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$, & ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{P})$, respectively.

The electronic spectra of Nickel complexes of the type NiL_2B_2 shows three bands at 9210 cm^{-1} , 16100 cm^{-1} and 24690 cm^{-1} corresponding to ${}^3\text{A}_{2g}(\text{P}) \rightarrow {}^3\text{T}_{2g}(\text{P})$, ${}^3\text{A}_{2g}(\text{P}) \rightarrow {}^3\text{T}_{1g}(\text{P})$, & ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$, respectively.

Table-3

The magnetic moment data of $[\text{Co}(\text{L})_2\text{B}_2]$ complexes

Complexes	Colour	Meff (B.M.)	Magnetic nature
1. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{H}_2\text{O})_2]$	Faint brown	4.95	Paramagnetic
2. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{NH}_3)_2]$	Light Pink	4.95	Paramagnetic
3. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_6\text{H}_5\text{NC})_2]$ (Ph-isocyanide)	Yellowish brown	4.90	Paramagnetic
4. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_9\text{H}_7\text{N})_2]$ (Quinoline)	Pinkish brown	4.94	Paramagnetic
5. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_5\text{H}_5\text{N})_2]$ (Pyridine)	Pinkish brown	4.86	Paramagnetic
6. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_5\text{H}_4\text{NCH}_3)_2]$ (α -picoline)	Yellowish brown	4.90	Paramagnetic
7. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_5\text{H}_4\text{NCH}_3)_2]$ (β -picoline)	Yellowish brown	4.88	Paramagnetic
8. $[\text{Co}(\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4)_2(\text{C}_5\text{H}_4\text{NCH}_3)_2]$ (γ -picoline)	Yellowish brown	4.85	Paramagnetic

Table-4
Electronic spectral data of [Co(L)₂B₂] complexes

Complexes	$\nu_1(\text{cm}^{-1})$	ϵ	$\nu_2(\text{cm}^{-1})$	ϵ	$\nu_3(\text{cm}^{-1})$	ϵ	C.T. band
1. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	8650	2.1	17800	0.0	21600	4.1	33000
2. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	8690	1.4	17400	0.4	20800	3.7	34000
3. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	8750	1.6	16300	1.2	20600	2.9	33500
4. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	8690	1.7	17200	0.8	20900	2.8	33200
5. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	8490	1.5	16800	1.0	21200	3.0	33100
6. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α -picoline)	8650	1.3	16900	1.1	20500	2.6	33400
7. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β -picoline)	8700	1.8	17500	1.2	20700	2.4	32500
8. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ -picoline)	8560	1.9	17500	0.7	20200	2.7	33300

Table-5
The magnetic moment data of [Ni(L)₂B₂] complexes

Complexes	Colour	M _{eff} (B.M.)	Magnetic nature
1. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	Light green	2.90	Paramagnetic
2. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	Greenish white	2.92	Paramagnetic
3. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	Faint green	2.96	Paramagnetic
4. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	Yellowish green	2.90	Paramagnetic
5. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	Dirty green	2.92	Paramagnetic
6. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α -picoline)	Greenish white	2.94	Paramagnetic
7. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β -picoline)	Greenish white	2.95	Paramagnetic
8. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ -picoline)	Greenish white	2.93	Paramagnetic

Table-6
Electronic spectral data of [Ni(L)₂B₂] complexes

Complexes	$\nu_1(\text{cm}^{-1})$	ϵ	$\nu_2(\text{cm}^{-1})$	ϵ	$\nu_3(\text{cm}^{-1})$	ϵ
1. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	9200	1.2	16200	2.98	24600	4.2
2. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	9210	1.1	16100	3.2	24500	4.1
3. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	9220	1.3	16150	3.0	24550	3.98
4. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	9190	1.4	16250	2.96	24650	4.3
5. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	9180	1.3	16200	2.85	24520	4.2
6. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂]	9160	1.2	16100	2.80	24540	4.1
7. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂]	9170	1.3	16250	2.98	24580	3.96
8. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂]	9190	1.2	16100	2.94	24560	3.94

Table-7

I.R. Spectral data of the ligand 2(3-methoxy-4-hydroxy benzylidene) imino benz hydroxamic acid, LH₂

I.R. Band Positions (cm ⁻¹)	Nature of bands	Probable assignment
1. 3540-50	Strong + sharp	ν(OH)
2. 2990-3010	Strong + sharp	ν(C-H) aromatic
3. 3130-3140	Strong + sharp	Hydrogen bonded ν(OH) of hydroxamic acid
4. 2800-2780	Strong + sharp	ν(OCH ₃)
5. 1660-1640	Strong + sharp	ν(C=N)
6. 1580-1570	Strong + sharp	ν(C-C) aromatic
7. 1360-1335	Strong + Medium	ν(OH) bonding
8. 1260	Strong + Medium	ν(C-N) aromatic
9. 1080-1070	Strong + Strong	ν(N-OH)

Table-8

I.R. Spectral data of the [Co(L)₂B₂] complexes

Complexes	ν(C=N)	ν(C=N)	ν(OCH ₃)	ν(C-N)	ν(M-N)	ν(N-O)	ν(M-O)	ν(OH) + ν(NH)
Ligand LH	3500	1660	2800	975		1085		3150
1. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	3500	1610	2810	970	450	1110	580	3250
2. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	3550	1620	2790	965	440	1100		3240
3. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	3540	1615	2780	965	445	110		3230
4. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	3520	1610	2783	970	435	1120		3220
5. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	3540	1620	2800	970	445	1090		3240
6. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α-picoline)	3520	1615	2870	965	450	1100		3220
7. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β-picoline)	3500	1615	2790	970	455	1120		3230
8. [Co(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ-picoline)	3540	1620	2780	965	445	1110		3240

Table-9

I.R. Spectral data of the [Ni(L)₂B₂] complexes

Complexes	ν(OH) (cm ⁻¹)	ν(OH+NH) (cm ⁻¹)	ν(OCH ₃) (cm ⁻¹)	ν(C=N) (cm ⁻¹)	ν(C-N) (cm ⁻¹)	ν(M-N) 1085	ν(M-N) (cm ⁻¹)	ν(H-O) (cm ⁻¹)
1. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (H ₂ O) ₂]	3550	3140	2790	1590	965	1120	460	580
2. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (NH ₃) ₂]	3500	3130	2775	1600	960	1110	440	
3. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₆ H ₅ NC) ₂] (Ph-isocyanide)	3540	3140	2790	1610	970	1115	445	
4. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₉ H ₇ N) ₂] (Quinoline)	3520	3135	2780	1620	970	1105	450	
5. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₅ N) ₂] (Pyridine)	3530	3135	2785	1600	965	1110	440	
6. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (α-picoline)	3540	3140	2790	1590	960	1120	450	
7. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (β-picoline)	3540	3140	2780	1610	970	1100	440	
8. [Ni(C ₁₅ H ₁₃ N ₂ O ₄) ₂ (C ₅ H ₄ NCH ₃) ₂] (γ-picoline)	3500	3130	2790	1600	965	1110	445	

Thus on the basis of elemental analysis, measurement of magnetic moment and interpretation of spectral data, the octahedral geometry for the complexes of Co(II) & Ni(II) ions with the ligands has been proposed as shown below :

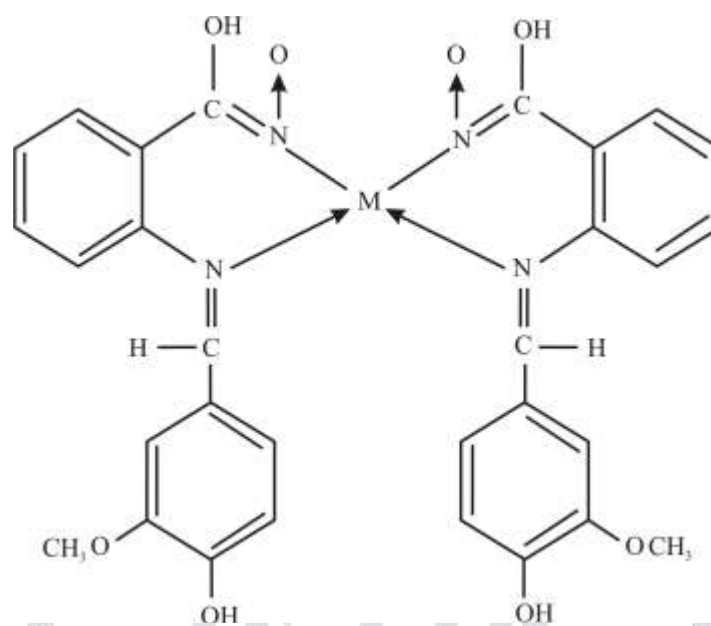


Fig. 2

M = Co(II) & Ni(II)

B = Water, ammonia, pyridine etc.

IV. ACKNOWLEDGEMENT

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