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Synthesis, Characterization and Thermogravimetric Analysis of Cobalt (III) and Nickel (II) Complexes with N-(3-Nitrobenzylidene)-1-Naphthylamine

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Abstract: The Schiff base ligand (3-nitrobenzylidene)-1-naphthylamine was prepared by the condensation between 3-nitrobenzaldehyde and 1-naphthylamine. The Ni and Co complexes of the corresponding ligand were prepared and was characterized by different methods like Thermogravimetric analysis, CHN analysis , IR and UV spectra From CHN analysis and IR spectral data the structure of Ni complex is found to be tetrahedral and that of Cobalt complex is octahedral structure.

Keywords: Schiff base, aldehyde, amine, XRD, TG

I. INTRODUCTION

Coordination chemistry is the study of compounds that have a central atom (often metallic) surrounded by molecules or anions, known as ligand. The ligands are attracted to the central atom by dative bonds, also known as coordinate bonds.

The coordination chemistry was pioneered by Nobel Prize winner Alfred Werner (1866-1919). He received the Nobel Prize in 1913 for his coorrdination theory of transition metal amine complexes. At the start of the 20th century, inorganic chemistry was not a prominent field until Werner studied the metal-amine complexes such as $[Co(NH_3)_6Cl_3]$. precipitate with silver ion.

Metal-ligand bonds are typically thought of lewis acid-base interactions. The metal atom acts as an electron pair donors(Lewis base). The nature of the bond between metal and ligand is stronger than intermolecular forces because they form directional bonds between the metal ion and the ligand, but ae weaker than covalent bonds and ionic bonds Schiff base is a class of organic compounds which are also known as azomethine, anilino or imines. Schiff bases forms an interesting class of compounds which found various applications in the field of medicinal, pharmaceutical, dye and plastic industries as well as in liquid crystalline technology. It has been reported that several compounds containing isatin moiety possess antibacterial, antifungal, anticonvulsant, anti-inflammatory and anti HIV activities. Sciff bases was first reported by Hugo Schiff. Studies show that the presence of lone pair of electron on triagonally hybridized nitrogen atom of C=N group is of fundamental and biological importance. The formation of chelate ring is essential for the production of stable complexes with the ligands containing azomethine groups. Schiff bases are the special types of organic compounds, named after the work done by a German chemist Hugo-Schiff , he will condensed carbonyl compounds with primary amines to get azomethine group (>CH = N-) as follows:

$>C = O + H_2N - R \quad > CH = N - R + H_2O$ —

II. MATERIALS AND METHODS

A. Materials

All reagents used for the synthesis of ligand and complexes are of commercial grade and they are directly used without further purification. 3-nitrobenzaldehyde is used as aldehyde and 1-naphthylamine is used as the amine and the solvent used is methanol.

B. Instruments

Instruments used in this investigation are given below:

1) Schimadzu IR prestige-20 spectrometer
2) Schimadzu UV-2450 A Spectrometer
3) Systronies conductivity meter 304
4) Gouy type magnetic balance
5) CHN elemental analyser
6) Thermobalance.

The purity of the compounds were checked by Thin Layer Chromatography (0.5mmthickness) using silica gel-G and spots were visualized by exposing the dry plates to iodine vapours.

C. Methods

1) *Synthesis Of N-(3-Nitrobenzylidene)-1-Naphthylamine:* 3-nitrobenzaldehyde (0.151g, 0.001M) dissolved in 20 ml methanol and 1-naphthylamine in 20 ml methanol was mixed well. The resulting mixture was refluxed for about four hours. On cooling, yellow coloured substance were separated from the solution. It was filtered and dried.

2) *Synthesis Of Metal Complexes*

a) *Synthesis of Cobalt Complex:* Cobalt nitrate has been used as a synthetic intermediate for the preparation of Co(III) complex. The methanolic solution of ligand (0.001) was just heated to dissolve the ligand and to this, methanolic solution of the metal (0.0005) was added so that the ration will be 1:2 and the mixture is refluxed for four hours. The P^H is maintained between 6-7. Then the volume was reduced to half its initial volume. After concentration the solution was cooled and the complex formed is separated out. It is filtered, washed with methanol and dried in vacuum.

b) *Synthesis of Nickel Complex:* Nickel Chloride has been used as a synthetic intermediate for the preparation of Ni(II) complex. The methanolic solution of the ligand (0.001M) was just heated to dissolve the ligand and to this, methanolic solution of the metal (0.0005M) was added so that the ratio will be 1:2 and the mixture is refluxed for four hours. The pH is maintained between 6-7. Then the volume was reduced to half its initial volume. After concentration, the solution was cooled and the complex formed is separated out. It is filtered, washed with methanol and dried in vaccum.

Table2.3: Physical properties of ligand and complexes

COMPOUND	COLOUR	SOLUBILITY	YIELD (%)
Ligand	Yellow	DMSO	80%
Ni Complex	Yellow	DMSO	78%
Co complex	Dark Yellow	DMSO	75%

D. Infrared Spectra

IR spectroscopy is a spectroscopic technique used to identify chemical compounds and to investigate sample composition. A comparison of IR stretching frequency of the ligand and its metal complexes gives an idea about the mode of binging in complexes. On complexation with metal ions, the characteristic IR frequencies of the coordinating groups were influenced by the force constant of the metal ligand bond resulting in shifting of the group frequencies. The shift were useful in identifying the coordination sites. The IR spectra of the solid samples were recorded in Schimadzu IR prestige -20 spectrometer in the range of 4000-400 cm^{-1}. Potassium bromide disc method was employed for sample preparation.

E. Electronic Absorption Spectroscopy

Electronic absorption spectroscopy is used to study the stereochemistry of the complexes. By using d-d transitions of the metal ions and their absorption spectra, it is possible to determine the ligand field splitting of the d orbitals of the metal ions.

Metal-ligand interaction can be studied from the UV-Visible spectra of free ligand and its metal complexes.The UV-Visible spectra of the samples in DMSO solution were recorded in Schimadzu UV-2450 A spectrometer in the range of 200-800 nm

F. Molar Conductance

Molar conductance of transition metal complexes were determined in DMF and N,N‘‘-dimethyl formamide at room temperature using a systolic conductivity Meter 304. The cell constant of the conductivity cell was 1 cm^{-1}. The concentration of the solution was around 1x 10^{-3} M. The molar conductance is measured by the equation,

$$M = 1000\ k/c$$

Where c = concentration of the solution in mol/L
k = conductivity (specific conductance)

G. *CHN Analysis*

CHN analysis was done in CHN elemental analyzer at the karyavattom campus.

H. *Magnetic Susceptibility*

Magnetic susceptibility measurements of the metal complexes were studied at room temperature (3000K) by using Magway MSB Mk1 magnetic susceptibility balance. Diamagnetic corrections were computed using Pascal's constant by adding the diamagnetic contribution of various atoms and structural units. Gram susceptibility was calculated using the formula,

$$X_g = (\alpha + \beta F) / W$$

Where α = Air Displacement Constant
β = Tube Constant
F = change in weight in milligram
W = weight of sample in gram

The effective magnetic moment μ_{eff} was calculated using the formula, $\mu_{eff} = 2.84\sqrt{X_m T}$
where X_m = molar susceptibility corrected for diamagnetism and T = Temperature, 293 K

I. *Thermogravimetry*

Thermogravimetry is the technique in which a sample is continuously weighed as it is heated at a predetermined and a preferably at a linear rate. The resulting weight change vs temperature curve so obtained provides information regarding the thermal stability and composition of the original material, the composition and thermal stability of the intermediate compounds and the composition of residue.

The instrument used is a thermobalance. Both manual and automatic balance are used, but latter type is preferred.

J. *Estimation Of Nickel*

The nickel is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime and then adding a slight excess of aqueous ammonia solution.

$$NiSO_4 + 2C_4H_8O_2N_2 \longrightarrow Ni(C_4H_7O_2N_2)_2 + H_2SO_4$$

The complex solution was first digested with HCl – H_2SO_4 mixture and is made upto 25 mL and 10 mL is pipetted out into a 100 ml beaker. Then it is diluted to 50 mL The solution is heated to 80^{0}C and 10 mL of 1% dimethyl glyoxime in rectified spirit is added immediately followed by dilute ammonia solution drop wise directly to the solution. The precipitate is heated for 20 – 30 minutes and the supernatant liquid is tested for complete precipitation. The precipitate is allowed tocool and then filtered through a previously weighed sintered crucible (IG_4).

The precipitate is washed with cold water and dried at 110-120^{0}C. It is allowed to cool in desicator and weighed.

K. *Estimation Of Cobalt*

Cobalt was estimated by means of gravimetric method after the decomposition of the complex with concentrated nitric acid. The resulting solution was made up to 100ml. 20ml of the solution was pipetted out into a beaker. Added with content stirring 4.8ml of the mercury(II)chloride solution followed by 5.2ml of the ammonium thiocyanate reagent. Scratching the sides of the beakers with the stirring rod was avoided. A dark blue precipitate formed after stirring for 1-3 minutes; the stirring was continued for a further 2-3 minutes and allowed stand for 2 hours at room temperature. Collected the precipitate in a weighed sintered glass or in a crucible, used the filtrate to arrest the transfer of any residual precipitate in the beaker. Washed the precipitate with 2-3ml of a dilute solution of the precipitating reagent and finally with 5ml ice cold water. Dried at 100$^{\cdot}$C and weighed as $Co[Hg(SCN)_4]$.

III. RESULT AND DISCUSSION

Schiff base derived from 3-Nitrobenzaldehyde and & 1-naphthylamine has been examined as ligand for Nickel in +2 oxidation state and Cobalt in +3 oxidation state.

A. *General Properties*

Both Ni(II) and Co(III) complexes are yellow and dark yellow in colour respectively and are stable in air. Both the complexes are insoluble in water and readily soluble in methanol, ethanol, Dimethyl Sulphoxide.

Ligand

Cobalt Complex

Nickel Complex

B. Analysis

Metal content in the complexes were determined by standard method after decomposing the complexes with hydrochloric acid-sulphuric acid mixture. The metal complexes of Schiff base ligand were prepared by the stoichiometric reaction of the corresponding metal and ligand in 1:2 ratio. The gravimetric analysis of Nickel complex and Cobalt complex are found to be successful and from these, the metal content in the complexes are determined.

C. Molar Conductance

Molar conductance of 10^{-3} M solutions of the metal complexes at 25^0C were measured in DMF and N,N‘‘-dimethyl formamide. The molar conductance values of Ni(II) and Co(III) complexes under investigation are found to be 77 $\Omega^{-1}cm^2mol^{-1}$ and 155.3 $\Omega^{-1}cm^2mol^{-1}$ respectively.

The molar conductance value shows that the Ni(II) and Co(III) complex was electrolytic in nature. Because there is no charged species in the complex to neutralize the charge of the central metal ion.

D. Magnetic Measurements

Magnetic susceptibility of the complexes were determined using Magway MSB Mk1 magnetic susceptibility balance. The measurements were made at room temperature. Table shows the effective magnetic moments calculated from the magnetic susceptibility which is corrected for diamagnetic corrections. Some indications about the structure, geometry and coordination of the complexes can be obtained from magnetic moment values.

The Ni(II) and Co(III) complexes are paramagnetic in nature.

The magnetic moment values of Ni(II) and Co(III) complexes are found to be 3.81 BM and 5.81BM. From this, it is clear that the Ni(II) complex has tetrahedral and Co(III) complex has octahedral in structure.

Complex	Colour	Yield(%)	Molecular weight	Magnetic moment(BM)	Molar conductance $(\Omega^{-1}cm^{2}mol^{-1})$
Ni(II) complex	Yellow	78%	389.0034	3.81	20
Co(III) Complex	Dark yellow	75%	683.55	5.81	22.8

Table 3.4(a)

E. *CHN Analysis*

Complex	C%		H%		N%	
	Experimental	calculated	Experimental	Calculated	Experimental	Calculated
Ni complex	49.55	52	4.57	4.63	7.70	7.20
Co complex	57.05	59.68	5.03	4.68	9.46	8.19

Table 3.5(a)

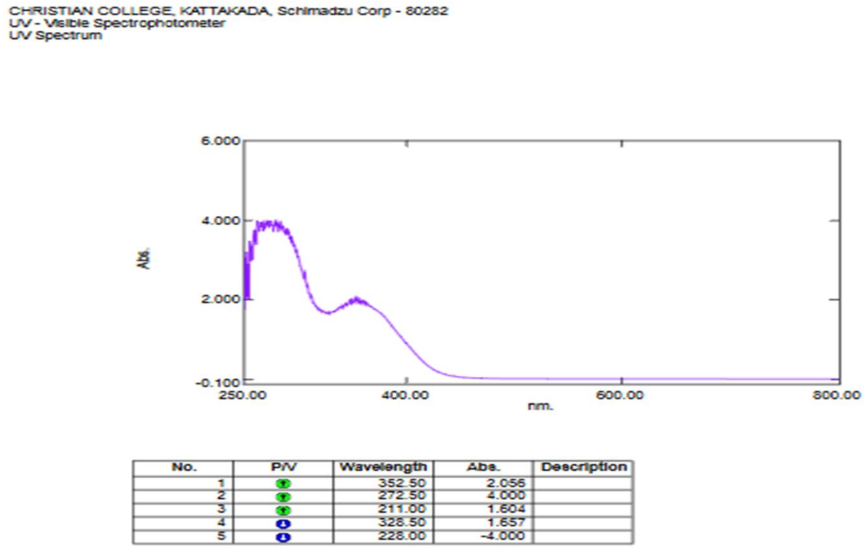
The experimental percentage values of carbon, hydrogen and nitrogen in the two complexes found out by CHN analysis were similar to the percentage calculated from the theoretical aspects. So from CHN analysis, the structure and denticity of the ligand and complex can be confirmed.

Here, the CHN analysis values are in good agreement with the calculated percentage of carbon, hydrogen and nitrogen in the complex. So by using CHN analysis, it is easy to confirm that the ligand is monodentate.The structures of the complexes can also be confirmed from CHN analysis value. The structure of Ni complex is found to be tetrahedral and that of cobalt complex is octahedral

F. *Electronic Spectra*

The electronic spectra are often helpful in the evaluation of result furnished by other methods of analysis. The electronic spectral bands of the ligand and complexes was recorded over the range 200-800nm in DMSO

1) *Electronic Spectrum of Ligand*

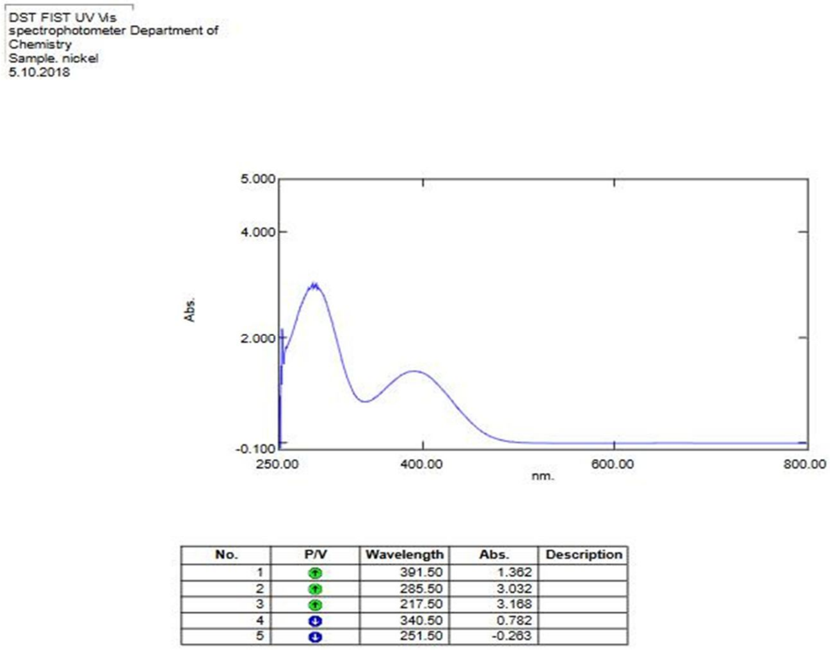

CHRISTIAN COLLEGE, KATTAKADA, Schimadzu Corp - 80282
UV - Visible Spectrophotometer
UV Spectrum

No.	P/V	Wavelength	Abs.	Description
1	↑	352.50	2.056	
2	↑	272.50	4.000	
3	↑	211.00	1.604	
4	↓	328.50	1.657	
5	↓	228.00	-4.000	

Ultraviolet spectra of the ligands recorded in DMSO showed strong bands around 272.5 nm and at 352.5 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

Electronic spectra of complexes.

2) Nickel Complex

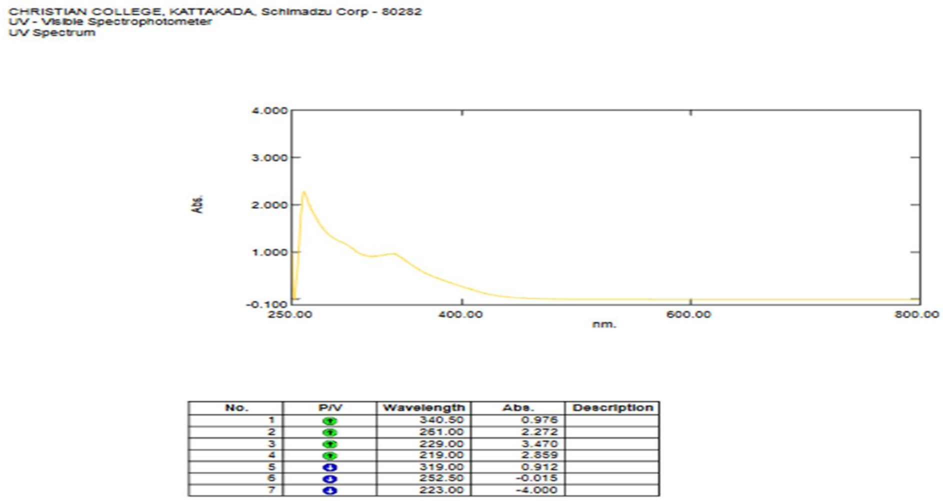


DST FIST UV Vis
spectrophotometer Department of
Chemistry
Sample. nickel
5.10.2018

No.	P/V	Wavelength	Abs.	Description
1	↑	391.50	1.362	
2	↑	285.50	3.032	
3	↑	217.50	3.168	
4	↓	340.50	0.782	
5	↓	251.50	-0.263	

Ultraviolet spectra of the Nickel complex recorded in DMSO showed strong bands around 285.5 nm and at 340.5 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

3) Cobalt Complex



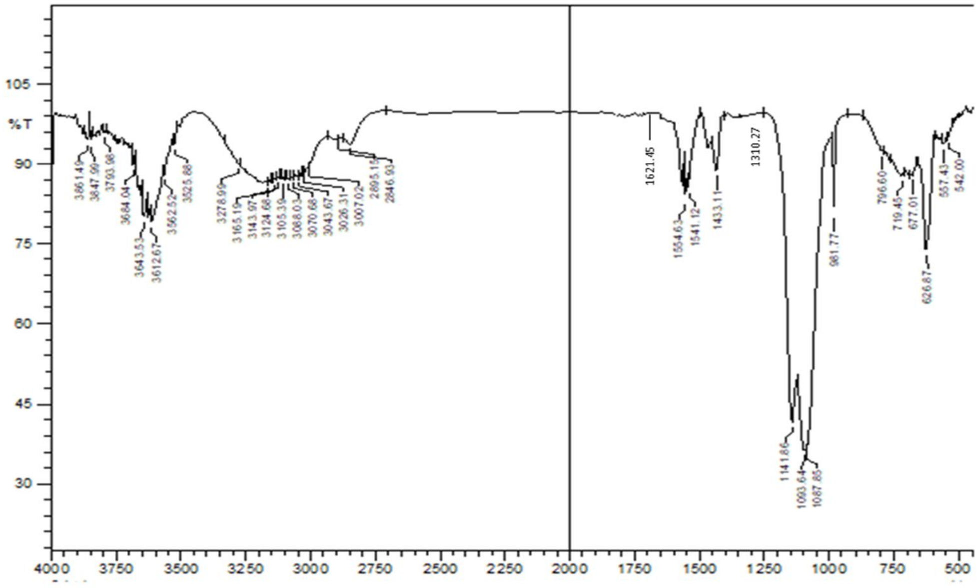
CHRISTIAN COLLEGE, KATTAKADA, Schimadzu Corp - 80282
UV - Visible Spectrophotometer
UV Spectrum

No.	P/V	Wavelength	Abs.	Description
1	↑	340.50	0.976	
2	↑	261.00	2.272	
3	↑	229.00	3.470	
4	↑	219.00	2.859	
5	↓	319.00	0.912	
6	↓	252.50	-0.015	
7	↓	223.00	-4.000	

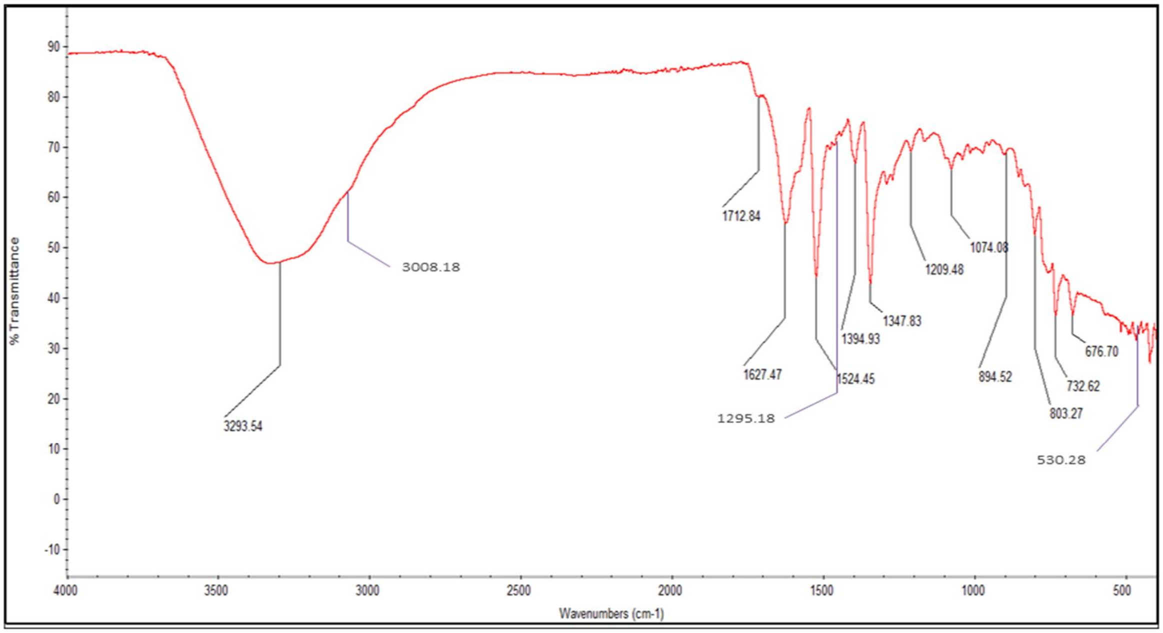
Ultraviolet spectra of the Cobalt complex recorded in DMSO showed strong bands around 261 nm and at 340 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of Π—›Π* and n—›Π* transition respectively.

4) *Infrared Spectrum*: The IR spectral data of the Schiff base ligand and its metal complexes are presented in table. The spectra of the complexes were compared with that of the free ligand to determine the coordination sites which involve in chelation

a) *IR Spectrum of Ligand*



b) *IR Spectrum Of Nickel Complex*

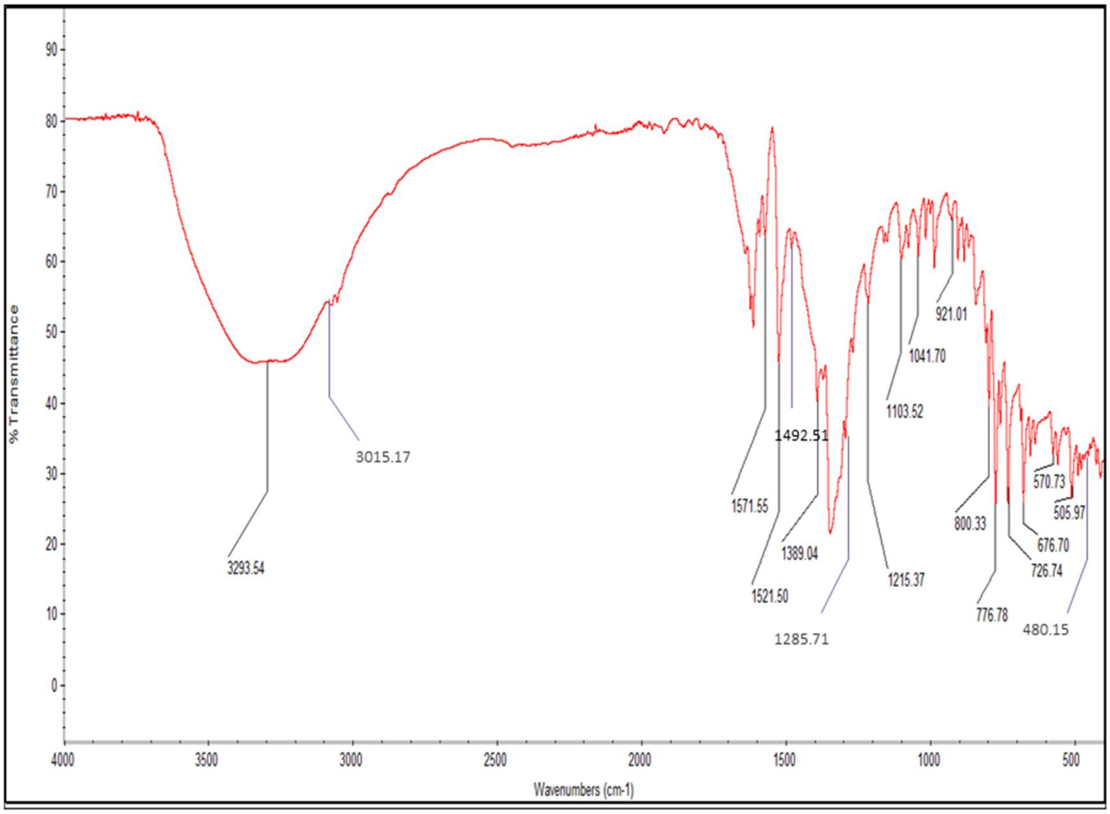


Ligand	Ni complex	Assignment
-	3298.54	Coordinated water molecule
3026.31	3008	=CH
1621.45	1627.47	C=N
1433.11	1394.93	C=C
1541.12 1310	1524.45 1295.18	N=O(Sym.Bending) N=O(Asym.Bending)
719.45	732.62	Monosubstituded
-	530.28	Ni-N

Table 3.7b

In the IR spectrum of the ligand, a medium strength band is observed at 3026.31 cm^{-1} which corresponds to =CH group. In the complex, it is shifted to a lower frequency of 3008cm^{-1}. The intense band near 1621.45 cm^{-1} can be attributed to υ (C=N) of the ligand. It is shifted to a higher frequency of 1627.47 cm^{-1} which indicates the presence of coordination. Two peaks at 1541.12 cm^{-1} and 1310cm^{-1} corresponds to the symmetric and asymmetric bending of NO_2 group. In Ni complex, a strong band is obtained in 3298.54 cm^{-1} which indicates the presence of coordinated water molecule. The peak at 530.28 cm^{-1} can be attributed to Ni-N bond which is absent in the free ligand. This peak at 530.28 cm^{-1} confirms the coordination in Ni complex.

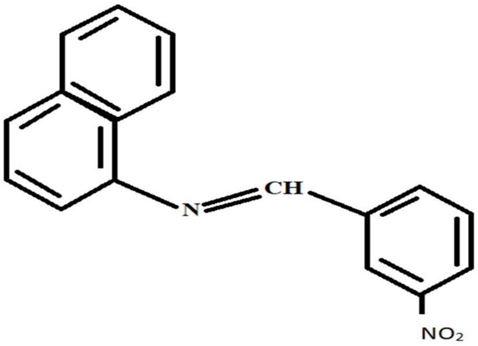
c) Spectrum Of Cobalt Complex



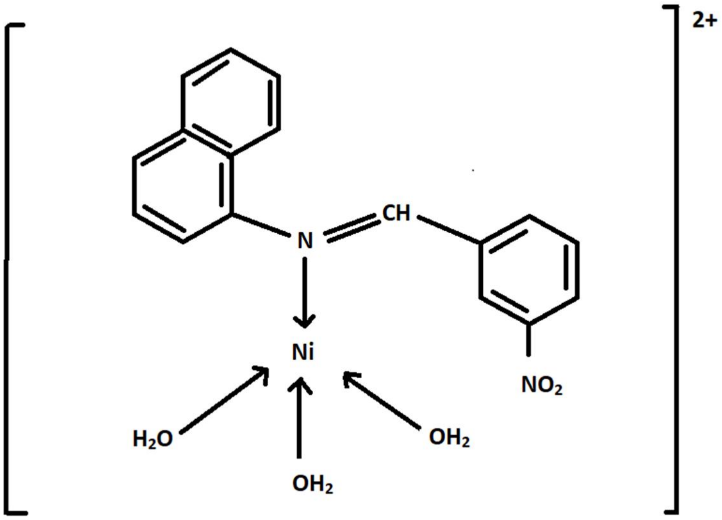
Ligand	Co complex	Assignment
-	3293.54	Coordinated water molecule
3026.31	3015	=CH
1621.45	1617.50	C=N
1433.11	1389.04	C=C
1541.12 1310	1492.51 1285.71	N=O(Sym.Bending) N=O(Asym.Bending)
719.45	726.74	Monosubstituded
-	480.15	Co-N

Table 3.7c

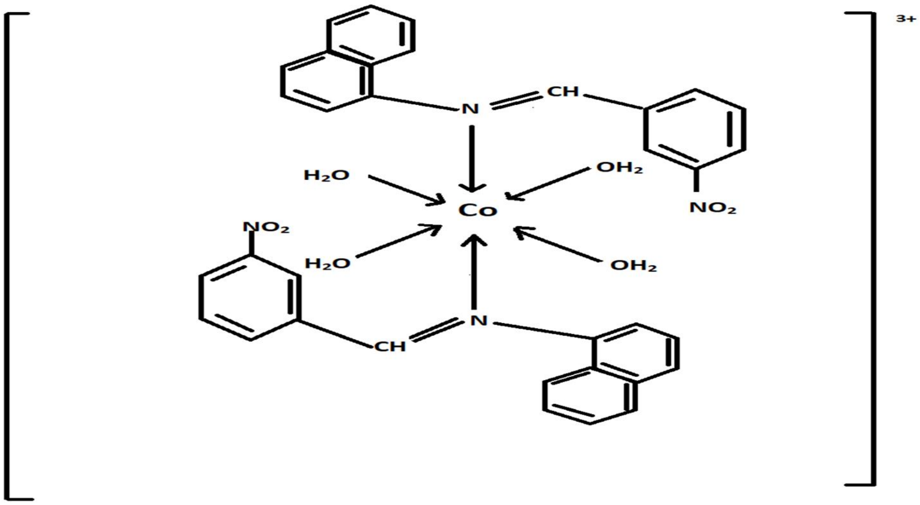
In the IR spectrum of the ligand, a medium strength band is observed at 3026.31 cm^{-1} which corresponds to =CH group. In the complex, it is shifted to a lower frequency of 3015cm^{-1}. The intense band near 1621.45 cm^{-1} can be attributed to υ (C=N) of the ligand. It is shifted to a lower frequency of 1617.50 cm^{-1} which indicates the presence of coordination. Two peaks at 1541.12 cm^{-1} and 1310cm^{-1} corresponds to the symmetric and asymmetric bending of NO_2 group. In Co complex, a strong band is obtained in 3293.54 cm^{-1} which indicates the presence of coordinated water molecule. The peak at 480.15 cm^{-1} can be attributed to Co-N bond which is absent in the free ligand. This peak at 480.15 cm^{-1}confirms the coordination in Co complex.



Structure Of Ligand



Structure Of NI Complex

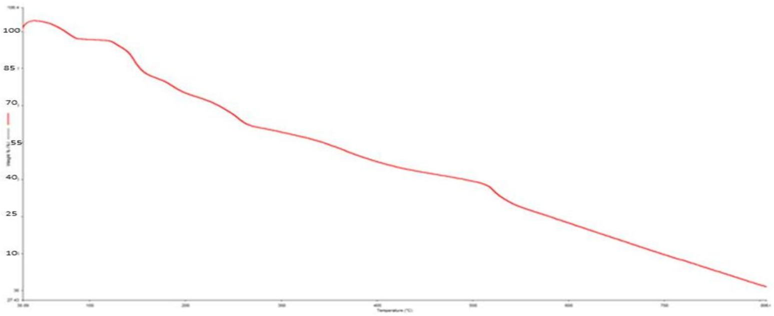


Structure of Co Complex

G. Thermogravimetric Analysis

Thermogravimetric analysis of complexes was conducted. TG curve of the complexes are shown below

1) TG Analysis of Cobalt Complex

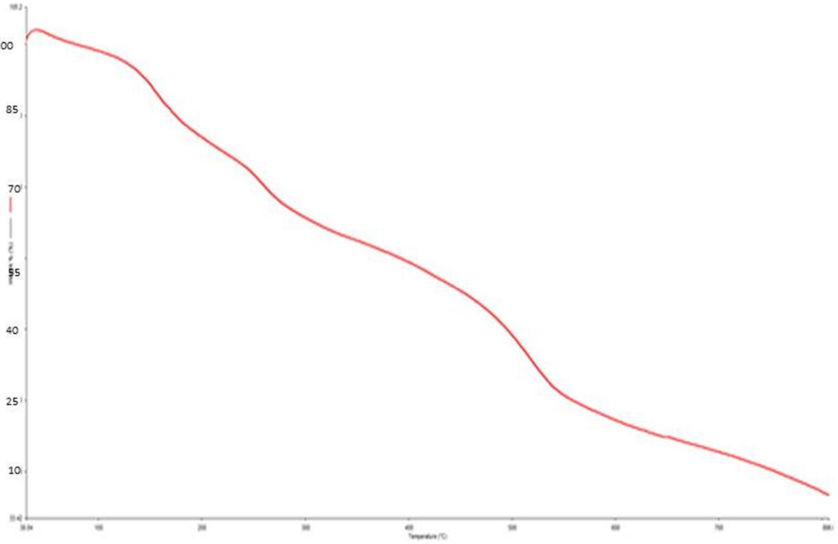


Temperature range ^{0}C	Mass Loss In %		Assignment
	Experimental	Calculated	
30-90	5.21	5.26	Removal of two molecules of water
120-150	5.18	5.20	Removal of two molecules of water
160-240	12.98	13.5	Removal of two molecules of NO_2
270-670	67.08	67.29	Removal of two molecules of $C_{17}H_{12}N$
	9.55	8.69	Leaving of metal residue

Table3.8.(a)

The thermogravimetric analysis (TG) curve obtained during the decomposition of Co-complex is shown in the figure. This demonstrate that the decomposition occur at several step. The weight loss(5.21%) curve between a temperature range of 30-90^{0}C represent the elimination of two water molecule. The Weight loss (5.18%) at temperature range 120-150^{0}C represent removal of another two molecules of water. At the temperature range 160-240^{0}C the weight loss is 12.98% represent the removal of two molecules of NO_2. At the temperature range 270-670^{0}C the weight loss is 67.08% represent the removal of two molecules of $C_{17}H_{12}N$.

2) TG Analysis of Nickel Complex



Temperature range ^{0}C	Mass loss in %		Assignment
	Experimental	Calculated	
120-180	13.48	13.88	Removal of three molecules of water
200-260	10.51	11.82	Removal of NO_2 molecule
280-670	59.03	59.13	Removal of $C_{17}H_{12}N$
	16.98	15.17	Leaving of metal residue

Table3.8b

The thermogravimetric analysis (TG) curve obtained during the decomposition of Ni-complex is shown in the figure. This demonstrate that the decomposition occur at several step. The weight loss(13.48%) curve between a temperature range of 120-180^{0}C represent the elimination of three water molecule. The Weight loss (10.51%) at temperature range 200-260^{0}C represent removal of a molecule of NO_2. At the temperature range 280-670^{0}C the weight loss is 59.03% represent the removal of a molecules of $C_{17}H_{12}N$.

IV. SUMMARY AND CONCLUSION

The Schiff base ligand (3-nitrobenzylidine)-1-naphthyl amine has been synthesized by the condensation of 3-nitrobenzaldehye and 1-naphthyl amine. It is yellow in colour and having 80% yield and soluble in dimethyl sulphoxide. The spectral data of the ligand concluded that the ligand is monodentate.

The Nickel complex of this ligand is yellow in colour and having 78% yield and soluble in DMSO. The molar conductance value of this complex was found to be 20 $\Omega^{-1}cm^{2}mol^{-1}$. This value indicates that the complex is electrolytic in nature. The magnetic moment value of this complex is 3.81 BM which suggests that the complex has tetrahedral structure. The appearance of new bands at 530.28 cm^{-1} in the IR spectrum is assigned to Ni-N stretching vibration. The spectral data of the ligand concluded that the ligand is monodentate. From TG analysis it is confirmed that three water molecules,one NO_2 and a $C_{17}H_{12}N$ molecules were eliminated at different temperature range.

The Co complex of this ligand is dark yellow in colour and having 75% yield and soluble in DMSO. The molar conductance value of this complex were found to be 22.8 $\Omega^{-1}cm^{2}mol^{-1}$. This value indicates that the complex is electrolytic in nature. The magnetic moment value of this complex is 5.81 BM which suggests that the complex has octahedral structure. The appearance of new band at 480.15 cm^{-1} in the IR spectrum are assigned to Co-N stretching vibrations. From TG analysis it is confirmed that four water molecules, two NO_2 and two $C_{17}H_{12}N$ molecules were eliminated at different temperature range.

The UV spectra of ligand and complexes shows characteristic absorption at the range of 285 nm and at 340 nm region which confirms the presence of benzenoid and azomethine linkages, which are characteristic of $\Pi \longrightarrow \Pi^*$ and $n \longrightarrow \Pi^*$ transition respectively.

The experimental percentage values of carbon, hydrogen and nitrogen in the two complexes found out by CHN analysis were similar to the percentage calculated from the theoretical aspects. So from CHN analysis, the structure and denticity of the ligand and complex can be confirmed.

V. ACKNOWLEDGEMENT

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