



## Interaction of naphthalene acetic acid with divalent metal ions and the characterization of the complexes

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### Abstract

On literature review, the studies on carboxylic acids reveal that their coordination chemistry has been studied extensively. However, in view of the absence of reports on the coordination chemistry of substituted acetic acid moieties the present investigation has been undertaken. In the present work, naphthalene acetic acid (NAA) a base moiety was characterized by elemental analysis, Ultra Violet – Visible, FT-IR, and <sup>1</sup>H-NMR spectroscopy and magnetic susceptibility. Studies and the interaction of the base NAA with bivalent metal ions like cobalt (II), copper (II), nickel (II) and mercury(II) in non- aqueous media like ethanol was studied. The metal complexes of cobalt (II), copper (II), nickel (II) and mercury(II) ions with NAA are synthesized in solid state and their characterization was done by chemical analysis, electrical conductivity, elemental analysis and spectral measurement (UV-visible, FT-IR, and <sup>1</sup>H-NMR). Magnetic susceptibility values have also been measured for the complexes synthesized.

Key-Words: Naphthalene acetic acid, Bivalent metal ions.

### Introduction

Co-ordination chemistry is a rapidly developing field in chemistry. It has versatile applications in a number of ways. Many naturally occurring substances like chlorophyll (green pigment of plants) and haemoglobin (coloring matter in blood) are co-ordination compounds. The transition metals have a maximum number of vacant d-orbital and can gain considerable stability by accepting electrons from the ligands in the form of dative bonds with the resultant formation of a metal-ligand complex<sup>[6]</sup>. The tendency of the transition metals to form complex compounds is particularly marked with iron, cobalt and nickel. Indole-3 acetic acid, naphthalene acetic acid, and 2,4-dichlorophenoxyacetic acid are the well known plant growth regulating hormones, widely used in agricultural field<sup>[13, 14]</sup>. The derivatives of these hormones were also found to have plant growth regulating and other biological activities<sup>[13, 15, 16]</sup>.

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### Material and Methods

A short account of the analytical methods employed in this work is presented in this chapter. Molar conductance, magnetic susceptibility, infrared spectral measurement, ultra-violet spectral measurement, <sup>1</sup>H NMR spectral measurement, elemental analysis and chemical analysis were carried to characterize the synthesized compounds. Electrical Conductivity Measurement was measured the concentration around 10<sup>-3</sup>M. The infra-red spectra of the ligand NAA and the complexes were taken as KBr pellets in the conventional range of 4000-400 cm<sup>-1</sup>. The <sup>1</sup>H-NMR spectrum was recorded employing TMS as internal reference and DMSO-D<sub>6</sub> and CHCl<sub>3</sub>-D as solvent at 300 K. The <sup>1</sup>H-NMR spectral study has been carried out to elucidate the structure of the organic ligand NAA and its mercury (II) complex. The electronic spectra of the present ligand and the coloured metal complexes were measured in DMF or DMSO solutions using a double beam UV – visible spectrophotometer Lambda 35. The working range of the instrument was 350 – 1100nm. Magnetic measurements were carried out using a Gouy magnetic balance. The Gouy tube was calibrated using Hg[Co(SCN)<sub>4</sub>] and diamagnetic corrections were made with Pascal's constants. The effective magnetic moments were calculated using Curie's relation,  $\mu_{\text{eff}} = 2.84 [X_M^{\text{corr}} \cdot T]^{1/2}$  B.M, where, T is the absolute temperature at which measurements were done. The metal complexes of naphthalene acetic

acid were analyzed for the metal content after decomposing them in conc.  $\text{HNO}_3$ . Cobalt (II), nickel (II) and mercury (II) were determined gravimetrically. Copper (II) was determined by the iodometric titration method. The chemical composition of the ligand naphthalene acetic acid was determined by analyzing carbon and hydrogen content. Carbon and hydrogen were determined using Vario EL III analyzer.

#### Characterization of Naphthalene acetic acid

Commercially available and chemically pure naphthalene acetic acid purchased was used as ligand in the present study.

#### Experimental Techniques

#### Synthesis of metal complexes of Naphthalene acetic acid

##### Preparation of Cobalt (II) Complex

A weight of 2.38g of hydrated Cobalt (II) chloride (0.01 M) was taken in a 100 ml beaker and dissolved in 25ml of hot distilled water. An amount of 5.58 g of NAA (0.03 M) was taken in another 100ml beaker and dissolved in 25ml of hot ethanol. The clear solution of the ligand was added to the solution of the metal salt in small quantities with constant stirring for 10 minutes. A pink coloured solid complex separated from the solution. It was filtered, washed repeatedly with ethanol and finally with solvent ether. The complex was dried and stored in a vacuum desiccator.

The same procedure was adopted for the preparation of Cu(II), Ni(II) and Hg(II) complexes.

##### Estimation of Cobalt

Cobalt was estimated gravimetrically. Appropriate amounts of mercuric chloride and ammonium thiocyanate were added to the cobalt solution. A blue coloured solid complex  $\text{Hg}[\text{Co}(\text{SCN})_4]$  separated out. From the weight of the complex the weight of cobalt was calculated.

##### Estimation of copper

Copper was estimated iodometrically. To the copper solution ammonium hydroxide was added to form pale blue precipitate. It was dissolved in acetic acid and nearly 2g of solid potassium iodide was added and mixed well and then the liberated iodine was titrated against standardised sodium thiosulphate. 2ml of starch solution was added to get the end point. The end point was the disappearance of blue colour. From the titre value the amount of copper was calculated.

##### Estimation of Nickel

Nickel was estimated gravimetrically as follows. To the clear solution 5ml of 1:1 HCl was added and the solution was diluted to 100ml with distilled water. The solution was then heated to about  $80^\circ\text{C}$ . To the hot solution about 20 ml of 1% alcoholic solution of dimethyl glyoxime reagent was added in drops with

constant stirring. Immediately dilute ammonia solution was added drop wise directly to the solution to make it alkaline. A rosy red precipitate of nickel dimethyl glyoximate complex was obtained. The precipitate was digested on water bath for about an hour and the precipitate was allowed to settle. The precipitate was filtered, washed with hot water and dried in an air oven, cooled in desiccator and weighed. From the weight of the dimethyl glyoximate complex, the weight of nickel was calculated.

#### Results and Discussion

Naphthalene acetic acid (NAA), a new base derived from naphthalene contains two oxygen atoms as potential donor atoms. It is pertinent to study how the NAA binds with the metal atom, when it is interacted with a transition metal salt. Hence, the new base NAA was purchased and used as a ligand.

##### Analytical results

The results of elemental analysis on NAA and its complexes are furnished in Table 4.1. The percentage compositions of NAA and its complexes indicate their empirical formulae.

##### Characterization of NAA

The commercial product of NAA was purified and then characterized by melting point, elemental analysis, infrared, UV and  $^1\text{H-NMR}$  spectra. The results of elemental analysis of NAA are provided in (Table 4.1). The important vibrational frequencies are listed in (Table 4.2). The  $^1\text{H-NMR}$  resonance signals are listed in (Table 4.3). A perusal of the IR spectrum shows a medium strong absorption band at  $3434\text{ cm}^{-1}$  which attributed to the carboxylic OH group. The absorption band found at  $3060\text{ cm}^{-1}$  is ascribed to the aromatic C-H vibration and that at  $2916\text{ cm}^{-1}$  is due to aliphatic C-H absorption. The absorptions found in the range  $1696\text{--}1510\text{ cm}^{-1}$  may be due aromatic C=C stretch as well as the carboxylic carbonyl group.

The free ligand NAA multiplet signals found at  $\delta 7.966\text{--}7.246\text{ ppm}$  are ascribed to the aromatic protons and the signal seen at  $\delta 4.073\text{ ppm}$  is due to the protons of the  $\text{CH}_2$  group. The chemical shift due to the carboxylic proton (COOH) is expected to be observed at  $\delta 10\text{--}14\text{ ppm}$ . Since the spectrum was scanned in the  $\delta$  range  $-0.5$  to  $9.5\text{ ppm}$  this particular signal was not located.

##### Characterization of $\text{Co}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

This pink coloured complex having 1:2 metals: ligand stoichiometry may have cobalt in +2 oxidation state. During the interaction  $\text{Co}^{\text{II}}$  chloride with NAA the carboxylic acid might have ionized to give the carboxylate anion which combine with  $\text{Co}^{2+}$  to give the pink complex  $\text{Co}(\text{C}_{12}\text{H}_9\text{O}_2)_2$ . The elemental analysis (Table 4.1) and conductivity measured in  $\sim 10^{-3}\text{ M}$  DMF solution confirm the same.



The free acetate ion ( $\text{CH}_3\text{COO}^-$ ) shows absorption at 1560 and  $1416\text{cm}^{-1}$  which are attributed to  $\nu_a(\text{COO}^-)$  and  $\nu_s(\text{COO}^-)$  respectively. Considering the  $\text{NAA}^-$  ion, the band at  $1410\text{cm}^{-1}$  may be due to symmetric  $\nu_s(\text{COO}^-)$  vibrations. In the case of the cobalt (II) complex these respective absorption bands are observed at 1511 and  $1410\text{cm}^{-1}$ . The difference ( $\Delta$ ) between the asymmetric and symmetric bands of free  $\text{NAA}^-$  is at a much higher value of  $189\text{cm}^{-1}$  where as in the complex spectrum,  $\Delta$  is much lower at  $100\text{cm}^{-1}$  indicating bidentate chelation of the carboxylate ligand to  $\text{Co}^{\text{II}}$  ion.

A perusal of the electronic spectrum of the  $\text{Co}^{\text{II}}$  complex shows several bands due to d-d transition, charge transfer and ligand origin. A very weak absorption at  $524.7\text{nm}$  ( $19058\text{cm}^{-1}$ ) may be attributed to  ${}^4\text{T}_{2g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$  transition. Another weak absorption band found at  $651.2\text{nm}$  ( $15356\text{cm}^{-1}$ ) is attributed to  ${}^4\text{T}_{2g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$  transition. A strong band found at  $297\text{nm}$  ( $33670\text{cm}^{-1}$ ) may be due to charge transfer transition. The effective magnetic moment measured at room temperature for the  $\text{Co}^{2+}$  ion is 4.8 B.M. Hence on the basis of chemical analysis, infrared and electronic spectra and magnetic moment the cobalt (II) complex is proposed to have an octahedral stereochemistry.

#### Characterization of $\text{Ni}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

The green coloured nicked (II) complex on elemental analysis is found to have 1:2 stoichiometry. Very low electrical conductivity value indicate the complex to be a non- electrolyte. An examination of IR spectrum of this shows the appearance of  $\nu_a(\text{COO}^-)$  at  $1510\text{cm}^{-1}$  and that  $\nu_s(\text{COO}^-)$  at  $1410\text{cm}^{-1}$ . The difference between the symmetric and anti symmetric absorption is at  $100\text{cm}^{-1}$  which much less than the free ion ( $\text{NAA}^-$ ) value ( $189\text{cm}^{-1}$ ) this spectral feature indicates the bidentate chelation of the carboxylate ligand.

Also the far IR absorption band seen at  $540\text{cm}^{-1}$  may be due to  $\nu(\text{Co-O})$ . The IR spectrum also shows bands at  $3391(\text{r})$ ,  $1631$ ,  $801$ , and  $625\text{cm}^{-1}$  which are attributed to the presence of coordinated water molecules. The band found at  $540\text{cm}^{-1}$  is ascribed to the (Ni- O) stretching vibration.

The electronic spectrum of the  $\text{Ni}^{\text{II}}$  complex shows an absorption at  $24814\text{cm}^{-1}$  ( $403\text{nm}$ ) which is attributed to  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$  transition, another band at  $13405\text{CM}^{-1}$  ( $746\text{nm}$ ) due to  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$  and a third band at  $9814\text{CM}^{-1}$  ( $1019\text{nm}$ ) due to  ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$  transition. These absorption bands are indicative of an octahedral symmetry of  $\text{Ni}^{\text{II}}$  in the complex. The  $\mu_{\text{eff}}$  value measured at 2.84 B.M is also supportive of octahedral geometry.

Hence based on analytical conductivity and magnetic data and IR and UV-visible spectroscopy a bidentate chelation of the organic ligand and octahedral stereochemistry is proposed to this complex.

#### Characterization of $\text{Cu}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

On the basis of chemical and elemental analysis this complex is given 1:2 stoichiometry. The very electrical conductance measured for this complex reveals that the complex in non-ionic. The IR spectrum indicates the presence  $\nu_a(\text{COO}^-)$  and  $\nu_s(\text{COO}^-)$  vibrational frequencies at 1511 and  $1329\text{cm}^{-1}$  respectively. The difference between these two is much higher than expected for bidentate chelation mode of coordination of  $\text{NAA}^-$  ligand. Presence of water in the coordination sphere is indicated by IR bands at  $3466(\text{br})$ ,  $1599,79(\text{split})$  and  $625\text{cm}^{-1}$ . The band at  $511\text{cm}^{-1}$  also points to the binding of Cu and O atoms.

The green coloured  $\text{Cu}^{\text{II}}$  complex measures an electronic absorption band at  $11509\text{CM}^{-1}$  ( $868.86\text{nm}$ ) which is attributed to  ${}^2\text{E}_g \rightarrow {}^2\text{T}_{2g}(\text{F})$ . It also exhibits another band at  $32159\text{cm}^{-1}$  ( $310.95\text{nm}$ ) which may be due to charge transfer transition. The complex has a  $\mu_{\text{eff}}$  value of 1.45 B.M per Cu atom at room temperature. The very low value of  $\mu_{\text{eff}}$  may be due a coupling of the unpaired electrons one on each  $\text{Cu}^{\text{II}}$  ion.

Hence based upon the chemical and elemental analysis, conductivity, magnetic moment, IR and electronic absorption spectra, this complex is given a carboxylato bridged binuclear structure where there is antiferromagnetic interaction between two copper ions.

#### Characterization of $\text{Hg}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

Analysis shows that the mercury complex has 1:2 stoichiometry. The electrical conductivity measurement shows that it is a non-electrolyte. Investigation of its IR spectrum points to the fact that the carboxylato anion as a chelating bidentate ligand. The  $\nu_a(\text{COO}^-)$  appears at  $1511\text{cm}^{-1}$  and the  $\nu_s(\text{COO}^-)$  appears at  $1410\text{cm}^{-1}$ . The difference between the two is  $101\text{cm}^{-1}$  which is very low indicating bidentate chelation to the metal ion. Also the appearances of bands at  $3448$ ,  $1599$ ,  $801$  and  $625\text{cm}^{-1}$  are attributed to the presence of coordinated water molecules. Further, the far IR absorption at  $400\text{cm}^{-1}$  which is due to Hg-O stretching vibration may also indicate the coordination of the carboxylate anion and  $\text{H}_2\text{O}$  molecules. There is no magnetic and electronic spectral criterion for the bonding in  $d^{10}$  system. Hence, on the basis of other physico- chemical data a 6 - coordinated structure is proposed for this complex also.

#### Conclusion

This work embodies the results on the investigation of coordinating nature of 1- naphthalene acetic acid with

transition metals [Co(II), Ni(II), Cu(II), Hg(II)]. It details on transition metal ion complexes with reference to cobalt (II), nickel (II), copper (II) and mercury (II) ions, the biological importance of naphthalene acetic acid and a review on metal complexes of carboxylates and related compounds. It gives the details of the experimental techniques employed for the structural characterization of the complexes isolated. The experimental techniques include electrical conductivity and magnetic measurements, IR, UV-visible and  $^1\text{H}$  NMR spectroscopy, elemental and chemical analysis. This work outlines the discussion of the results obtained. On the physico-chemical data obtained the bonding atoms are fixed and the stereochemistry assigned for all the complexes. All the complexes have 1:2 stoichiometry. All of them have two coordinated water molecules each. In the  $\text{Co}^{\text{II}}$ ,  $\text{Ni}^{\text{II}}$  and  $\text{Hg}^{\text{II}}$  complexes, the carboxylate ligand acts as a chelating bidentate and in  $\text{Cu}^{\text{II}}$  complex, it is bridging bidentate. The very low magnetic moment determined in the case of  $\text{Cu}^{\text{II}}$  complex suggests a binuclear bridged structure. The metal ions in all the complexes studied are in an octahedral environment.

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Table 1: Analytical Results on IAA and its Metal Complexes

S/ No.	Compound	%C found (expected)	% H found (expected)	% M found (expected)
1	IAA (C <sub>10</sub> H <sub>9</sub> NO <sub>2</sub> )	76.49 (77.41)	5.32 (5.37)	-
2	Co(C <sub>10</sub> H <sub>8</sub> NO <sub>2</sub> ) <sub>2</sub> . 2H <sub>2</sub> O	62.20 (61.94)	4.62 (4.73)	12.74 (12.68)
3	Ni(C <sub>10</sub> H <sub>8</sub> NO <sub>2</sub> ) <sub>2</sub> . 2H <sub>2</sub> O	62.42 (61.81)	4.82 (4.72)	13.06 (12.86)
4	Cu(C <sub>10</sub> H <sub>8</sub> NO <sub>2</sub> ) <sub>2</sub> . 2H <sub>2</sub> O	61.46 (61.33)	4.96 (4.69)	13.88 (13.53)
5	Hg(C <sub>12</sub> H <sub>9</sub> O <sub>2</sub> ) . 2H <sub>2</sub> O	47.98 (47.48)	3.68 (3.63)	33.70 (33.07)

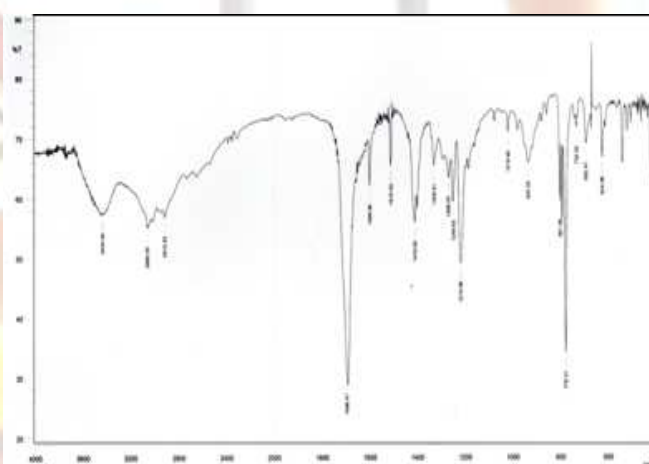


FIG.4.1 FT-IR SPECTRUM OF NAA

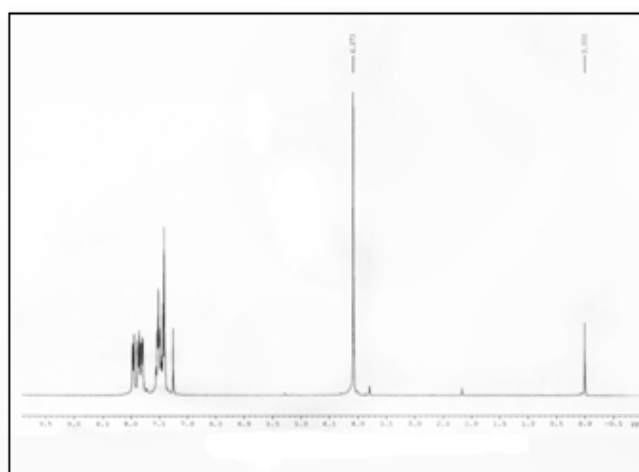


FIG. 4.2 H<sup>1</sup>-NMR spectrum of NAA



Table 2: Infrared Spectral Bands of NAA Complexes (cm<sup>-1</sup>)

Compound	$\nu_a$ (CO <sub>2</sub> <sup>-</sup> )	$\nu_s$ (CO <sub>2</sub> <sup>-</sup> )	$\Delta$	$\nu$ (MO)	$\nu$ (H <sub>2</sub> O)
Free NAA <sup>-</sup>	1599	1410	189	-	-
Co(NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	1510	1410	100	523	3407
Ni(NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	1510	1410	100	540	3391
Cu(NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	1511	1329	182	511	3466
Hg(NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	1511	1410	101	400	3448

Table 3: Proton NMR chemical shifts of NAA and its mercury complex (in ppm)

Compound	Aromatic Protons	CH <sub>2</sub> Protons
NAA	7.966-7.246	4.073
Hg(C <sub>12</sub> H <sub>9</sub> O <sub>2</sub> ) . 2H <sub>2</sub> O	8.037-7.160	4.039

Table 4: Magnetic and Electronic Spectral Data on NAA metal Complexes

Complex	$\mu_{eff}$ (BM) (Colour)	Absorption bands (cm <sup>-1</sup> )	Electronic transition
Co (NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	4.80 (Pink)	19058 15356 33670	<sup>4</sup> T <sub>2g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P) <sup>4</sup> T <sub>2g</sub> (F) → <sup>4</sup> A <sub>2g</sub> (F) Charge transfer.
Ni (NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	2.84 (Green)	24814 13405 9814	<sup>3</sup> A <sub>2g</sub> (F) → <sup>3</sup> T <sub>1g</sub> (P) <sup>3</sup> A <sub>2g</sub> (F) → <sup>3</sup> T <sub>1g</sub> (F) <sup>3</sup> A <sub>2g</sub> (F) → <sup>3</sup> T <sub>2g</sub> (F)
Cu (NAA <sup>-</sup> ) <sub>2</sub> . 2H <sub>2</sub> O	1.45 (Green)	11509 32159	<sup>2</sup> E <sub>g</sub> → <sup>2</sup> T <sub>2g</sub> (F) Charge transfer.

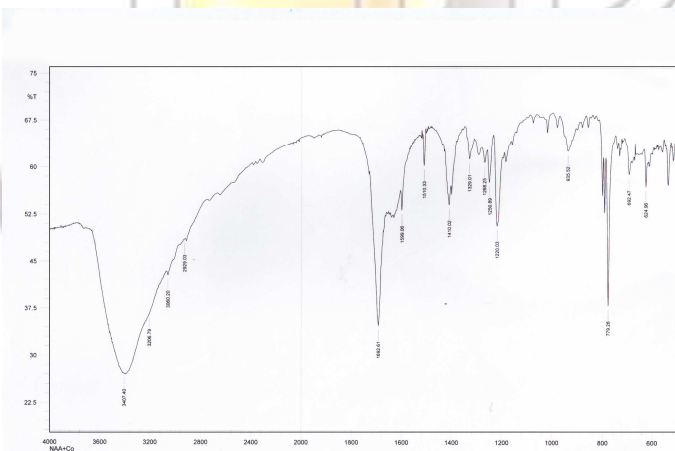


FIG.4.3 FT-IR SPECTRUM OF Co (C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O

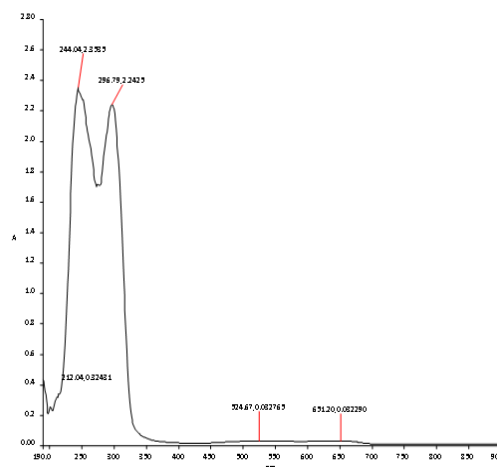
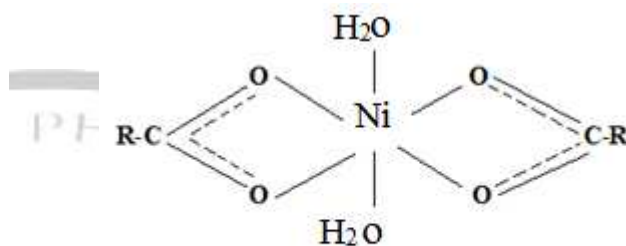
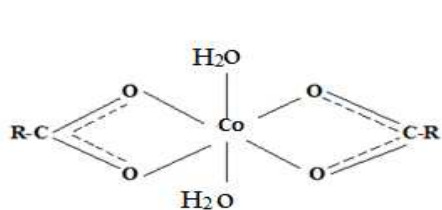


Fig. 4.4: UV Spectrum of Co (C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O



**Structure of  $\text{Co}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$**

The infrared absorptions observed at 3407, 1620, 800 and  $625\text{cm}^{-1}$  are attributed to the  $\nu(\text{OH})$ ,  $\delta(\text{HOH})$ ,

$\rho_{\text{r}}(\text{HOH})$  and  $\rho_{\text{w}}(\text{HOH})$  vibrations of coordinated water molecules respectively.

**Structure of  $\text{Ni}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$**

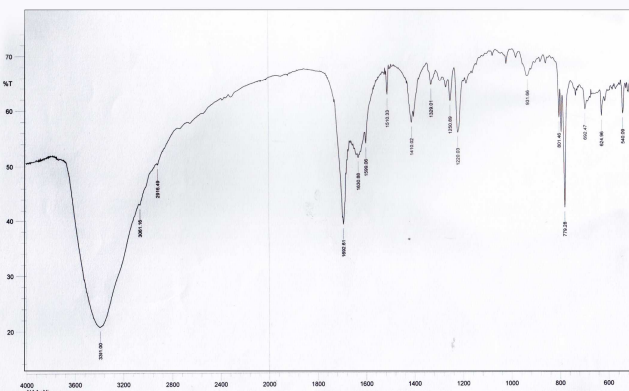


FIG.4.5 FT-IR SPECTRUM OF  $\text{Ni}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

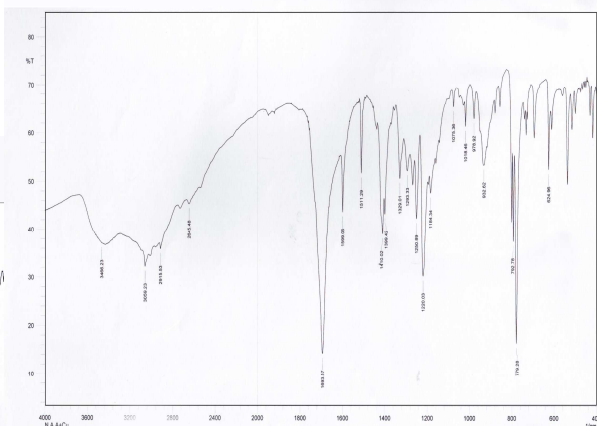


FIG.4.7 - FT-IR SPECTRUM OF  $\text{Cu}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

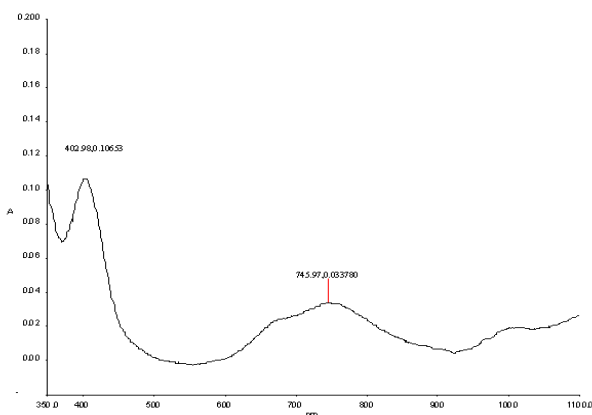


FIG.4.6 UV- SPECTRUM OF  $\text{Ni}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$

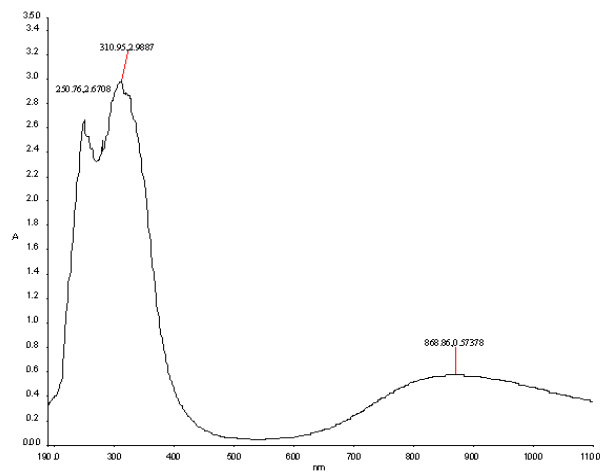
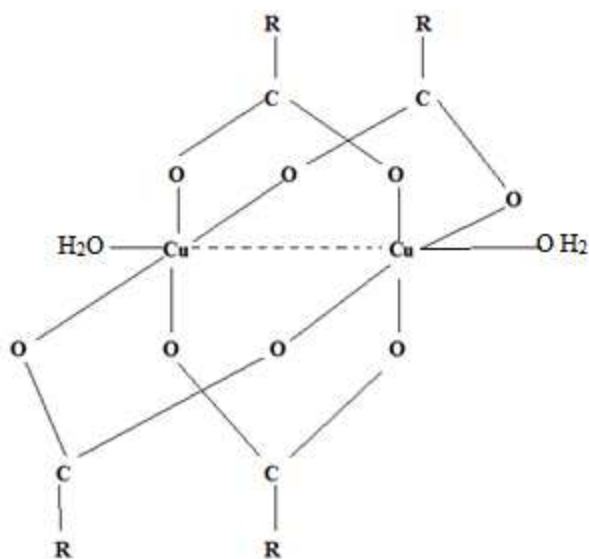


FIG.4.8 UV SPECTRUM OF  $\text{Cu}(\text{C}_{12}\text{H}_9\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$



Structure of Cu (C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O

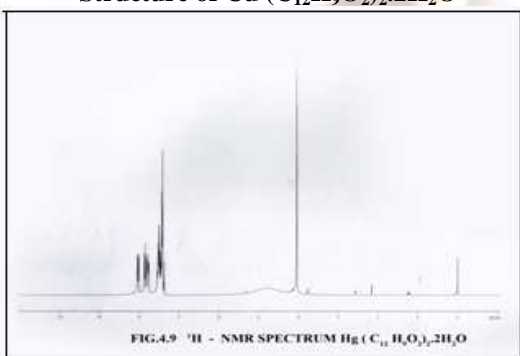


FIG.4.9 <sup>1</sup>H - NMR SPECTRUM Hg (C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O

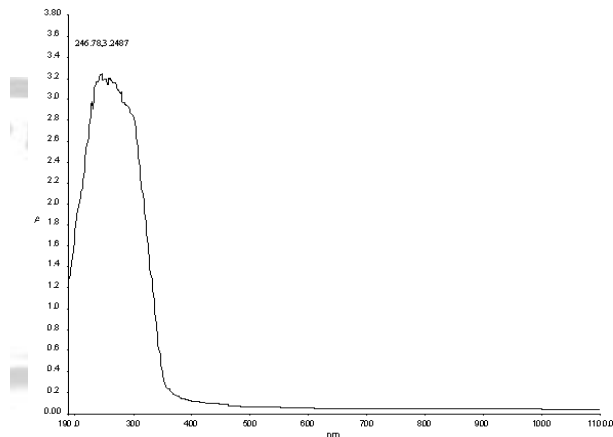
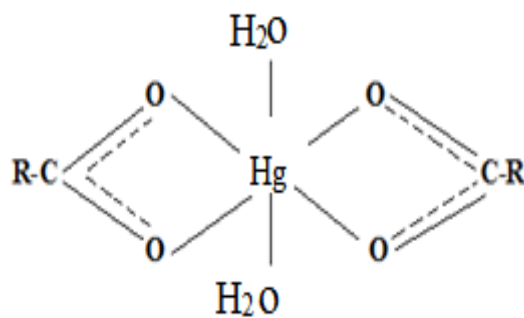


FIG.4.10 UV SPECTRUM OF Hg(C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O



Structure of Hg (C<sub>12</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>.2H<sub>2</sub>O