Characterization and synthesis of new mixed ligands complexes and antibacterial activity of 2-(naphthalene-lylamino)-2- phenylacetonitrile and 1,10-phenanthroline with VO(II), Fe(II), Mn(II), Cr(III), Co(II) metal ions

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Abstract

A new group of VO(II), Co(II), Fe(II), Mn(II), and Cr(III), complexes produced from 2-(naphthalene-lylamino)-2-phenylacetonitrile (L_1) as well as 1,10phenanthroline (L_2) are characterized by C.H.N.S., FT-IR, flame atomic absorption, 1H-NMR, UV-Vis spectra, molar conductivity as well as magnetic susceptibility measurement. The complexes had been recommended as octahedral except for [VOL₁L₂] SO₄.0.5H₂O which was discovered square pyramidal.

The accompanying metallic particle structures have been put in place alongside their suggested formulae platform around the accompanying: These edifices had been put in place by building a solution fusion of the above mentioned specks in outright ethanol with blending to give the common formulae: $[ML_1L_2Cln]$ $Cl.yH_2O$ where M: Cr^{+3} , Mn^{+2} , Fe^{+2} , and also Co^{+2} , n=1 or 2, 1, y=0.5, 1 and 2.

Keywords: 2-(naphthalen-l-ylamino)-2-phenylacetonitrile, Transition metal ions, Phenanthroline, Mixed Ligands, Benzaldehyde.

Introduction

1, 10-Phenanthroline is a white solid compound which is soluble in some organic solvents. It forms complexes with most metal ions. Among the metal-ion compounds, those of 1, 10-phenanthroline have pulled in line. 1, 10phenanthroline as chelating nitrogen giver ligand is among the most productive miscreant for moving metal particles with which it frames stable edifices in arrangement 1 . The nearness of sweet-smelling as well as heteroaromatic gatherings into the structure of nitrogen givers gives these ligands extra properties. For example, poly nitrogen contributors containing sweet-smelling and additionally heteroaromatic bunches conjugate the ligational capacity with the photophysical properties run of the mill of these gatherings and in like manner, they have been generally utilized as chemosensory for metal particles in arrangement, since their coordination may influence the properties of the photosensitive gathering offering ascend to an optical reaction^{2,3}. It is additionally reported that some ternary edifices of 1, 10-phenanthroline (phen) e.g. [Cu (phen)] have an antitumor action where it restrained DNA or RNA

polymerase exercises^{1,4}. Microbial contaminations regularly deliver torment and aggravation ⁵.

Chemotherapeutic, pain relieving and calming medications are endorsed all the while in ordinary practice ⁶. The compound having every one of the three exercises is not regular. Calming, pain relieving and antipyretic exercises are known for some pyridine and phenol subsidiaries ⁷. These mixes can square ligament pulverization amid the provocative procedure and therefore are promising class of mitigating mixes ⁸.

Material and Methods

Materials: The inorganic salts used were VOSO₄.H₂O, MnCl₂.4H₂O, CrCl₃.6H₂O, CoCl₂.6H₂O and FeCl₂.4H₂O and were obtained from Sigma-Aldrich. Benzaldehyde and 1-Naphthylamine were obtained from Sigma-Aldrich. Glacial acetic acid and ethanol were obtained from Carl Roth.

Preparation of 2-(naphthalen-l-ylamino)-2phenylacetonitrile (L₁): 2-(naphthalene-l-ylamino)-2phenylacetonitrile (L₁) was put in place with the accompanying KCN (0.13 gm, 2mmol) in (5mL) of distilled drinking water and cooled under 5°C. Benzaldehyde (0.212 gm, 2mmol) in 20mL of ninety five % C₂H₅OH was added. The mix was mixed at temperature underneath 5°C. Glacial acetic acid (0.12 gm, 0.002mol) was provided with constant blending holding heat beneath 5°C that was trailed by the development of crucial amine (1Naphthylamine) (0.286 gm 0.002mol) in 10mL of ninety five % C₂H₅OH as well as 5mL of Glacial acid cooled beneath 5°C with ceaseless blending in ventilated hood.

Amid the development, the heat was kept in place at 15° C. The blend was mixed for two hrs and was maintained at room temperature for twenty-four hrs. The acquired extended (Maroon) fine needles, ended up being flooded with weakened hydrochloric acid (0.2M) to evacuate some abundance of cyanide. The combination was recrystallized with ninety-five % C₂H₅OH. The yield fee was (80.9 %).

Preparation of Complexes (C_1 - C_5): A fix of 0.258 gm, 0.001 mol of 2-(naphthalene-l-ylamino)-2-phenylacetonitrile (L_1) in 15mL of complete ethanol plus (0.198gm, 1mmol) on the ligand (L_2) in 10mL of complete ethanol was added dropwise to 1mmol of VOSO₄.H₂O and also certain metallic

chlorides (0.181gm, 0.266 gm, 0.197 gm, 0.198 gm as well as 0.238 gm) for VOSO₄.H₂O, MnCl₂.4H₂O, CrCl₃.6H₂O, CoCl₂.6H₂O and FeCl₂.4H₂O respectively dissolved in (25mL) total C₂H₅OH with stirring below anhydrous factors making use of Na₂SO₄ (anhydrous). The ensuing mixtures had been refluxed at 75-80 °C for 4 hrs. The remedies had been left to be stirred immediately at room temperature. The precipitates had been recrystallized with total ethanol, collected and kept in desiccators. (Yield % of complexes C1to C5 was identical to 83.9, 75.5, 82.3, 78.5 as well as 84.6 respectively.

Results and Discussion

Some physical properties and elemental analysis of the two ligands (2-(naphthalen-l-ylamino)-2- phenylacetonitrile and 1, 10-iphenanthroline) and the prepared metal-ion complexes are shown in table 1.

FT-IR Spectra of L₁ and L₂: The FT-IR data of aminonitrile ligand (L₁) as well as 1, 10 phenanthroline (L₂) as well as their metal ion compounds have been revealed in table 2. Figures one and two were illustrating the FT-IR spectra of the 2 ligands. The bands given to stretching vibration of aromatic and aliphatic (C-H) of the 2 ligands have been seen at 2981 as well as 3064 cm⁻¹ for L₁ and also 2860 plus 3058 cm⁻¹ for L₂^{8,9}. The band associated with (C-N) extending vibration of the no cost ligand (L₁) was seen at 2169 cm^{-1 10, 11}. The band associated with (N-H) stretching vibration came out at 3330 cm^{-1 12-14}. The band given to (N-H) deformation came out at 1650 cm^{-1 15, 16}.

FT-_iIR Spectra of Complexes $(C_1 - C_5)$: FT-IR spectra of all of the prepared metal ion complexes have shown distinct rings of whichever lattice H₂O came out at (923-985)cm⁻¹ given to ρw (H₂O) of several complexes ^{17, 18} or perhaps coordinated water came out at 3451, 3532 cm⁻¹ given to v(OH) of most of the well prepared complexes coming from C1 to C5. The band that had been seen at 3340 cm⁻¹ was given to the N- H extending vibration. N-H team of L₁ was shifted within the spectra of most complexes C1-C5 to 3367, 3336, 3379, 3382 and 3382 cm⁻¹ respectively. This gave a sign that the ligand was coordinated with the metallic ions with the nitrogen atom on the amino group. The band at (2169) cm⁻¹ that had been given to the extending vibration of $v(C \equiv N)$ team of (L₁) was shifted within the spectra on the complexes C_1 , C_3 and C_5 to greater wavelengths 2225, 2185 as well as 2187 cm⁻¹ respectively as well as to lower frequencies within the spectra of complexes (C4 and C2) 2150 and 2152 cm⁻¹ respectively that describe the linkage of (C-N) set from nitrogen atom.

The spectra of complexes have shown an increasing change in (C-N) extending vibration towards greater frequencies and lower frequencies as a result of coordination with metallic ion via the lone set of electrons of a nitrogen atom. The band at (1650) cm⁻¹ that had been given to the (N-H) bending vibration of (N-H) on the ligand (L₁) was shifted within the spectra of most complexes C1 and C5 to 1623, 1633, 1627, 1631 as well as 1625 cm⁻¹ respectively, that provided additional indication that the ligand was synchronized with metallic ions through the nitrogen atom of amino group and therefore supports the complexes development.

The powerful band was performing at 1137 cm⁻¹ given to benzene and also pyridine band stretching vibration in the cost-free 1, 10-phenanthroline. This band shows some change to higher or lower frequencies by 7–52 cm⁻¹ as shown in table 2 that had been indicating the coordination on the pyridine nitrogen atoms to the metal ions in a bidentate manner¹⁹⁻²¹. New bands found within the spectra on the equipped metal ion complexes lie in the range from 478-587 cm⁻¹ which are not contained in the spectrum of the ligand attributed v (M-N) vibration.

Molar Conductance: The molar conductance values of the metal ion complexes C_1 , C_2 , C_3 , C_4 and C_5 lied in the range of 26-40 Ω^{-1} .cm⁻².mol⁻¹, these values were indicated that these metal ion complexes are weak electrolytes and that for complexes C_2 , C_3 , C_4 and C_5 one chloride anion was coordinated to the metal ion, leaving the other chloride anion outside the coordination sphere ²². The values of molar conductance of the complexes are given in table_i3.

UV Visible of the prepared ingredients: The UV-Vis absorption information of the ligands and their metal ion complexes have been captured around C₂H₅OH at 25°C (10^{-4} M) illustrated in table 3. The spectra of L₁ as well as C4 had been illustrated in figures 4 and 5. The spectra of the L_2 and L_1 had been displayed at high intensity bands happened to show up in the area 43668, 38461 cm⁻¹ respectively, that have been given to $\pi \rightarrow \pi^*$ on the conjugated system ²³. Low-intensity bands came out in the near UV Visible area 28168, 37174 cm⁻¹ respectively, had been given to $n \rightarrow \pi^*$ change, the intensity as well as positions of these bands count on the framework of particles and also the dynamics of solvent used ²⁴. The electronic spectra of the metallic ion complexes have shown new companies, the intensities and site; roles of these rings are determined by the ligand area consequences, the stereochemistry of complexes plus electron setup of the metallic ions ²⁵.

¹**H** -NMR Spectra: The ligand (2-(naphthalene-1-ylamino)-2- phenylacetonitrile) was indicated by ¹H-NMR spectroscopic methods, in addition to all of the prepared metal ion complexes (C1-C5) utilizing dimethyl sulphoxide (d⁶) as illustrated in table 4 and also figures 6 and 7. The ¹H -NMR spectrum of (L₁) confirmed three peaks; the first peak has been seen at δ 4.35 ppm that had been given to the (N-H), the next peak seen at δ 5.94 ppm that was given towards the (-CH-C≡N) proton while the last one appeared at δ 6.71-7.98 ppm corresponding towards the protons of (Ar-H). The ¹H -NMR spectrum of the 1,10-phenanthroline ligand (L₂) was confirmed a peak that had been seen at δ 7.58-8.83 ppm due to the protons of the aromatic ring²³. The ¹H -NMR spectra of the prepared compounds (C1-C5) had been like to ligands L_2 and L_1 , the one distinction had been that the signal of (=N-H) on the ligand L_1 was shifted within these complexes by 0.08, 0.18, 0.22, 0.23 and 0.30 ppm respectively as well as the signal of (-CH-C=N) was shifted in these metallic ion complexes by 0.13, 0.27, 0.02, 0.11 and 0.03 ppm respectively, these formations gave a sign for metal ion complexes formation. Several techniques including conductivity, FTIR, UV–Vis and ¹H-NMR Spectra were used to elucidate the probable structure of the C1-C5 complexes produced as shown in figures 8 and 9.

In vitroi antibacterial _iactivity: Agar (well diffusion method) was utilized to discover the activity ²⁶. Borer of 0.6 mm diameter was utilized, the focus of all elements was 10⁻³ M using dimethyl sulphoxide like a solvent and was utilized as a command gram negative while tetracycline was utilized as a command gram positive. The inhibition zones have been assessed in mm and also when compared with the inhibition zone of broad-spectrum antibiotic. The solvent showed absolutely no activity against the examined bacteria, while several of the synthesis compounds happened to show very excellent outcomes. Table 4 displayed the inhibition zones of the solvent and additionally the synthesis compounds.

A number of compounds C5 and C3 have confirmed pastime against the gram negative bacterium (*Escherichia Coli*), that cause illness for example; enterotoxigenic stresses are able to generate a toxin within the gut, ensuing usually in diarrhea ²⁷. The Complexes (C1, C4, C3, along with C5) have confirmed pastime against the Gram bacterium (Pseudomonas). This bacterium was popular because of its opposition to nearly all the evolved antibiotics and was recognized to function as the main reason for many health problems and infections ²⁸.

The tetracycline confirmed pastime against the grampositive bacterium (*Staphylococcus aureus*), the inhibition zone was 15.4 mm. The complexes C2, C5 and C3 have been confirmed pastime against the gram positive bacterium (*Staphylococcus aureus*), this particular bacterium triggers food poisoning and was discovered on the skin ²⁹. The complexes C3, C5 and C4 have been confirmed pastime against the gram-positive bacterium (*Bacillus subtilis*). A lot of variables have been reported to manage the natural tasks of metallic ion complexes: Configuration of metallic ions, the cost on the complex, metal type, kind of ligand, the move series plus geometry of metallic ion complexes.³⁰

Comp.	Formula M.wt. (gm/mol)	Yield%	Color	M.P °C	C % Cal. (Found)	H % Cal. (Found)	N % Cal. (Found)	M % Cal. (Found)	Chlorine% (Found)
L ₁	C18H14N2 (258.00)	80.9	Maroon	155- 157	83.72 (82.33)	5.,42 (5.17)	10.88 (10.07)		
L ₂	C12H8N2.H ₂ O (198.00)		White- Crystal	100- 102	(72.72)	(5.05)	(14.14)		
C ₁	[VOL ₁ L ₂]SO ₄ .0.5H ₂ O (628.17)	83.9	Olive Green	206 Dec.	57.88 (57.31)	4.10 (3.97)	9.11 (8.91)	9.22 (8.11)	
C ₂	[CrL ₁ L ₂ Cl ₂)]Cl. H ₂ O (632.61)	82.3	Light Green	280- 283	57.21 (56.90)	4.10 (3.54)	7.88 (8.85)	9.23 (8.21)	17.66 (16.83)
C ₃	[MnL ₁ L ₂ Cl(H ₂ O)]Cl (600.11)	75.5	Brown	325 Dec.	60.64 (59.98)	4.92 (4.33)	9.58 (9.33)	9.67 (9.16)	12.44 (11.83)
C4	[FeL ₁ L ₂ Cl(H ₂ O)]Cl. 2H ₂ O (636.95)	78.5	Light- Yellow	277- 280	55.12 (56.52)	5.50 (4.70)	8.84 (8.79)	9.77 (8.76)	12.50 (11.14)
C ₅	[CoL ₁ L ₂ Cl(H ₂ 0)]Cl.0.5H ₂ O (631.11)	84.6	Purple	300 Dec.	59.24 (58.71)	4.48 (4.40)	10.72 (9.13)	10.88 (9.13)	12.87 (11.58)

 Table 1

 Elemental analysis and some physical properties of the ligands (L1 and L2) and their prepared metal ion complexes

Dec. =Decomposition

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Table 2
Characteristic infrared absorption rings of the ligands (L ₂ and L ₁) and their metal ion complexes

Comp.	Molecular Formula	v(N-H) cm ⁻¹	υ(C- H) arom. cm ⁻¹	υ(C≡N) cm ⁻¹	δ(N-H) cm ⁻¹	v(C=N) + (C=C) cm ⁻¹	υ(M-N) cm ⁻¹	Others
L_l	C18H14N2	3340 w.sh	3064 v.sh	2169 v.s.sh	1650 w.sh	1582 s.sh		vH ₂ O=3475
L_2	C12H8N2.H ₂ O		3058			1616,1587,1558, 1504,1446,1137 m.sh		vH ₂ O=3359 - 3384
C_1	[VOL ₁ L ₂]SO ₄ .0.5H ₂ O	3367 w.sh	2974 w.sh	2225 s.sh	1623 m.br	1542,1515,1450, 1431 w.sh	587 m.sh	V=O = 964 $vH_2O = 3496$ vSO4 = 1054 ,1114
C_2	$[CrL_1L_2Cl_2)]Cl.H_2O$	3379 w.sh	2964 w.sh	2150 s.sh	1633 m.sh	1562,1506,1454 m.sh	574 w.sh	$vH_2O=3463$
C_3	$[MnL_1L_2Cl(H_2O)]Cl$	3336 s.br	298 5 w	2185 v.sh	1627 s.sh	1544,1512,1416, 1423 w.sh	578 W.br	$vH_2O = 3488$
C_4	$[FeL_1L_2Cl(H_2O)]Cl.2H_2O$	3382 w.sh	2991 w.sh	2152 v.sh	1631 m.sh	1566,1508,1411 s.br	568 w.br	$vH_2O=3448$
<i>C</i> ₅	$[CoL_1L_2Cl(H_2O)]Cl.$ $0.5H_2O$	3382 w.sh	3060 w.br	2187 v.sh	1625 w.sh	1575,1514,1492, 1425 m.sh	478 w.sh	$vH_2O=3485$

where: s=strong, m=medium, w=weak, v=very, br=broad, sh=sharp

Table 3

UV-Vis spectra, spectra parameter, molar conductance, magnetic susceptibility of the prepared complexes

		-			1 V		
Comp.	Molecular Formula	Wave length (nm)λ	Wave no. cm ⁻¹	Assignment	Molar Cond. (Ω ⁻¹ cm ² mol ⁻¹)	Magnetic susceptibilit y μ eff (B.M)	Suggested Geometry
L_{l}	C18H14N2	355 260	28169 38461	$n \rightarrow \pi \pi \rightarrow \pi^*$			
L_2	C12H8N2.H2O	269 229	37174 43668	$n \rightarrow \pi \pi \rightarrow \pi^*$			
C_1	[VOL ₁ L ₂]SO ₄ .0.5H ₂ O	956 602 429	10460 16611 43668	$2B2g \rightarrow 2Eg$ $2B2g \rightarrow 2B1g$ $2B2g \rightarrow 2A1g$	35.0	2.02 (1.73)	Square pyramid
C_2	$[CrL_1L_2Cl_2)]Cl.H_2O$	978 893 523	10224 11198 19120	$4A2g \rightarrow 4T2g$ $4A2g \rightarrow 4T1g(F)$ $4A2g \rightarrow 4T1g(P)$	40.3	3.92 (3.87)	Octahedral
<i>C</i> ₃	$[MnL_1L_2Cl(H_2O)]Cl$	913 902 325	10952 11086 30769	$6A1g \rightarrow 4T1g$ $6A1g \rightarrow 4Eg(D)$ C.T	26.8	6.05 (5.91)	Octahedral
<i>C</i> ₄	$[FeL_1L_2Cl(H_2O)]Cl.2$ H_2O	602 269 225	16611 37174 43478	$6Alg \rightarrow 4T2g (G)$ $6Alg \rightarrow 4Alg$ +4 Eg	30.7	5.02 (4.89)	Octahedral
<i>C</i> ₅	$[CoL_1L_2Cl(H_2O)]Cl.0.$ $5H_2O$	940 633 442	10638 15797 23696	$4Tlg \rightarrow 4T2g$ $4Tlg \rightarrow 4A2g$ $4Tlg \rightarrow 4Tlg(P)$	27.2	3.96 (3.87)	Octahedral

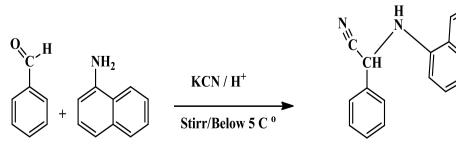
Comp.	Formula	Groups	Chemical Shifts δ(ppm)
L_1	$C_{18}H_{14}N_2$	(-NH)	4.35 5.94
		$(-CH-C\equiv N)$ $(Ar-H)$	6.71-7.98
L_2	$C_{12}H_8N_2H_2O$	(Ar-H)	7.58–8.83
<i>C</i> ₁	[VOL ₁ L ₂]SO ₄ .0.5H ₂ O	(-NH) (-CH-C≡N) (Ar-H)	4.43 5.81 6.73-7.87
<i>C</i> ₂	$[CrL_1L_2Cl_2)]Cl.H_2O$	$(-NH)$ $(-CH-C\equiv N)$ $(Ar-H)$	4.53 5.67 6.91-8.45
СЗ	$[MnL_1L_2Cl(H_2O)]Cl$	$(-NH)$ $(-CH-C\equiv N)$ $(Ar-H)$	4.13 5.92 6.23-8.33
C_4	$[FeL_1L_2Cl(H_2O)]Cl.2H_2O$	$(-NH)$ $(-CH-C\equiv N)$ $(Ar-H)$	4.58 5.83 6.40-7.99
C_5	$[CoL_1L_2Cl(H_2O)]Cl.0.5H_2O$	$(-NH)$ $(-CH-C\equiv N)$ $(Ar-H)$	4.65 5.91 6.74-8.38

 $Table \ 4 \\ H-NMR of \ the \ ligands \ (L_1 \ and \ L_2) \ and \ some \ of \ their \ metal \ ion \ complexes$

 Table 5

 Inhibition zones measured in (mm) of DMSO, Tetracycline and metal ion complexes

Compound	Inhibition-zone (mm) Escherichia coli	Inhibition-zone (mm) Pseudomonas aeruginosa	Inhibition- zone (mm) Staphylococcus aureus	Inhibition-zone (mm) Bacillus subtilis
DMSO				
Tetracycline			15.4	
C_1		27.8		
<i>C</i> ₂			15.5	
C_3	22	20	16	18
C_4		13		14
<i>C</i> ₅	28	24	21	22





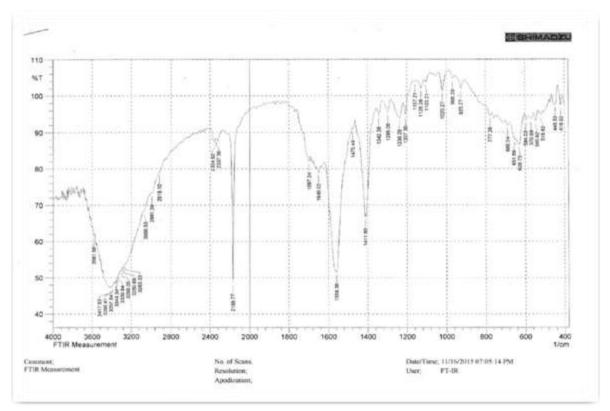


Figure 1: FT-IR spectrum of 2-(naphthalen-l-ylamino)-2- phenylacetonitrile [L1]

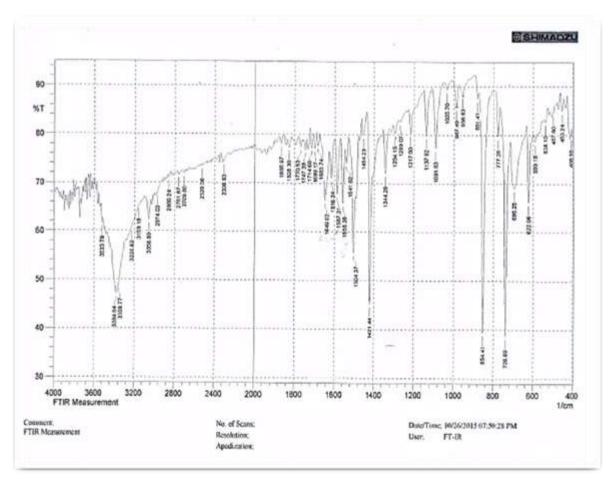


Figure 2: FT-IR spectrum of 1,10-iphenanthroline [L₂]

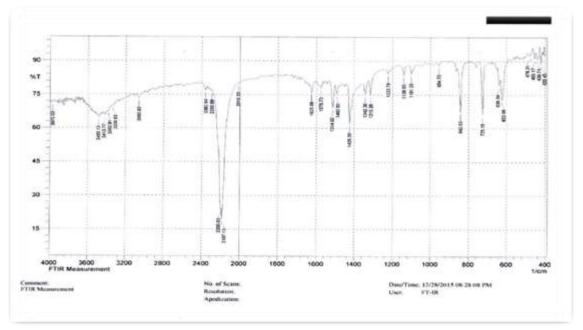


Figure 3: FT-IR spectrum of [FeL₁L₂Cl (H₂O)] Cl.2H₂O

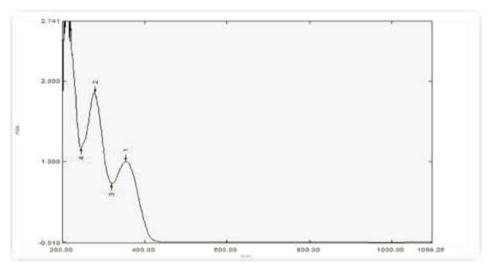


Figure 4: UV-VIS spectrum of 2-(naphthalen-l-ylamino)-2- phenylacetonitrile [L1]

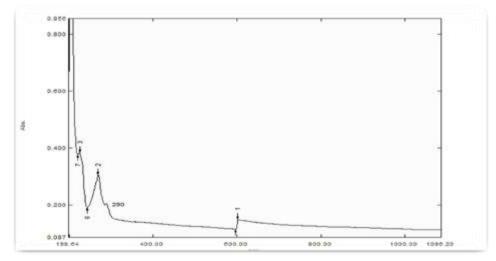


Figure 5: UV-VIS spectrum of [Fe L₁L₂Cl(H₂O)]Cl.2H₂O[C₄]

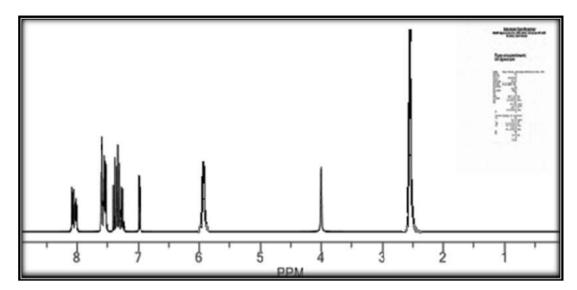


Figure 6: ¹H -NMR spectrum of 2-(naphthalen-l-ylamino)-2- phenylacetonitrile [L₁]

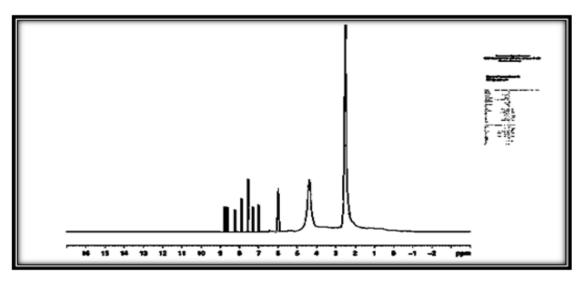


Figure 7: ¹H -NMR spectrum of [VOL₁L₂]SO₄.0.5H₂O

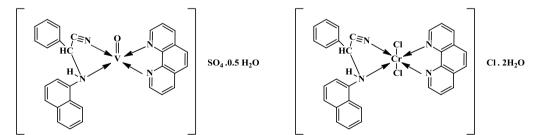
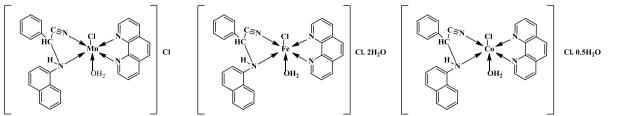


Figure 8: Probable structures of C₁ [VOL₁L₂]SO₄.0.5H₂O and C₂ [CrL₁L₂Cl₂)]Cl.H₂O



 $\label{eq:Figure 9: Probable structure of C_3 [Mn \ L_1 \ L_2 \ Cl(H_2 \ O)] Cl, C4 [Fe \ L_1 \ L_2 \ Cl(H_2 \ O)] Cl. 2H_2 O \ and \ C_5 [Co \ L_1 \ L_2 \ Cl(H_2 \ O)] Cl. 4H_2 O \ O) \ Cl. 2H_2 O \ and \ C_5 [Co \ L_1 \ L_2 \ Cl(H_2 \ O)] Cl. 4H_2 O \ O) \ Cl. 2H_2 O \ and \ C_5 \ Co \ L_1 \ L_2 \ Cl(H_2 \ O)] \ Cl. 2H_2 O \ and \ C_5 \ Co \ L_1 \ L_2 \ Cl(H_2 \ O)] \ Cl. 2H_2 O \ and \ C_5 \ Co \ L_1 \ L_2 \ Cl(H_2 \ O)] \ Cl. 2H_2 O \ Cl$

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