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SYNTHESIS AND CHARADERIZATION OF SOME NEW COMPLEXES OF MIXED LIGANDS DERIVED FROM CHALCONES AND DIAMINELIGANDS WITH MN (Ⅱ), FE (Ⅱ), CO (Ⅱ), NI(Ⅱ), CU(Ⅱ) AND ZN (Ⅱ) IONS

Mohammed A. Shihab* and Amaal Y. Assaf

College of Education for Pure Sciences / Department of Chemistry/ University of Mosul /Iraq.

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***Corresponding Author**
Mohammed A. Shihab
College of Education for Pure Sciences / Department of Chemistry/ University of Mosul /Iraq.

ABSTRACT

In this study chalcone was prepared by using 3-acetyl pyridine with aromatic aldehyde in basic solution (NaOH) acoording to Claissn-Schmidt condensation, metal complexes of M [Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II)] with mixed Ligands derived from chalcone (L) and diamine Ligand (A) such as ethylenediamine and 1,10-phenanthroline were prepared with ratio (2L: M : A) WhereL = 3-(2-hydroxy phenyl) -1- (pyridin-3-yl)prop-2-en-1-one). (HPPO). M= Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II). A = diamine Ligands: ethylenediamine (en), 1, 10- phenanthroline (phen). The prepared complexes characterized by varions spectroscopic techniq such as: (elemental analysis (C.H.N), molar conductance, magnetic properties as well as the specotral studies such as electronic spectra, IR, ^{1}HNMR Spectra). From the results obtained we can suggest octahedral geometry for all complexes, The chalcone Ligand acting as abidentate chlelating Ligand that coordinating from two oxygen atoms from hydroxyl group of aromatic aldehyde and neutral carbonyl group of chalcone. Two nitrogen atoms of diamine Ligand (en, phen) involved in the coordination with metal ion.

KEYWORDS: Chalcone, diamine Ligands, Chelate, Octahedral.

1- INTRODUCTION

Due to the biological and Industrial application of chalcones The chemistry of Chalcones has generated great scientfic Interest through the world(Thakare&Mandlik,2017) The fact that

they can be effectively utilized as antibacterial (Nowakowska *et al*., 2008), antifungal, antivical, antiparasitic, anticancer, antileishmanal and antitubercular agents have been subject of intensive.

Pharmaceutical Field (Bonakdar *et al*., 2017) chalcones have a strong ability to form metal complexes that play a promineut role in modern coordination chemistry (Balaji, 2019).

The name ((Chalcones)) was given by Kostaneck; and Tambor (Momison & Boyd, 2004).

In chalcones two aromatic rings are liked by an aliphatic three carbon chain (Ali& Khlafulla, 2016). Chalcone bears avery good synthon so that variety of novel hetero cycles with good profile can be designed. (Seema & Prafullakumar, 2015) The gereneral formula of Chalcone in fig (1)

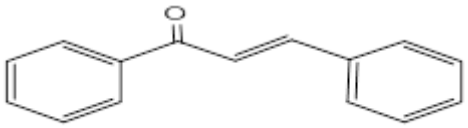


fig (1): (*E*)-chalcone

In present communication we report the reaction of 3-acetyl pyridine with aromatic aldehyde in the presence of basic medium to from chalcones. These chalcones were characterized and their complexing behavior with Mn(Ⅱ) ,Fe(Ⅱ),Co(Ⅱ),Ni(Ⅱ),Cu(Ⅱ) and Zn(Ⅱ) ions and diamine ligands has been studied(1,10- phenanthroline, ethylenediamine).

2- EXPERIMENTAL

All material and solvent required for synthesis of ligand and complexes were analytical grade.

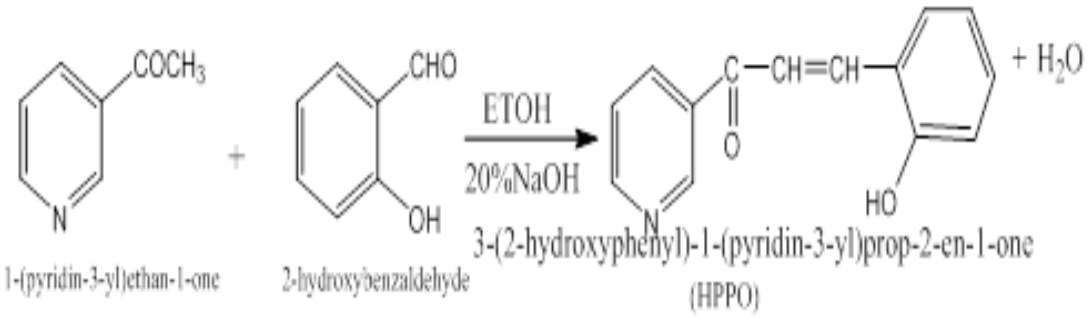
2.1 MATERIAL AND METHODS

Melting points of ligand and complexes were obtained with an electrothermal melting apparatus. Conductivity was measured (10^{-3}M) in DMSO with Conductivity Meter Model (Eutech pc700), magnetic moment were obtained by Gouy method at room temperature, Electronic spectra were run on a Shimadzo UV-Visible spectra photometer using 1cm cell (200-900nm).Infrared spectra on KBr pellets were recorded on shimadzo in the range(400-4000cm^{-1}). Carbon, hydrogen, nitrogen analyses of Ligand, complexes were done using

Elementalvariol (Ⅲ) and the metal analysis were done on NOVAA350-Analytikjena,Atomic absorption spectrophotometal.

2-2-Preparation of 3-(2- hydroxy phenyl) -1- (pyridin-3-yl) prop-2-en-1-one). (HPPO) (L)

The Chalcone was prepared by the method of preparation in the basic medium by reacting 3-acetyl pyridine (1.2g, 0.01mol) and salicylaldehyde (1.2g,0.01mol) into ethanol solvent (20ml) with continuous stirring. To this reaction aqueous solution of 20% Na OH added to it ,During the time the color was changed and the yellow precipitate was formed .The solution was kept at (20-30ȍ) for four hours with stirring .by kepping the solution at cool over night neutralized by ice cold (10%HCl) solution, the soild separated was filtered and washed with deionized water several tines to remove the trace of acid and salt formed, then air drying the precipitant, recrystallized the product from ethanol.(scheme1). (Echeverria, 2009; Thakare& Mandlik, 2017)



Scheme 1: Preparation of HPPO.

The completion of the reaction (monitored by TLC).

2-3-Synthesis of metal complexes (2: 1: 1) (L: M: Phen), (L: M: en)

The Complexes were prepared by adding (0.45g, 0.002mol) of L (HPPO) in (10ml) of hot ethanol to a solution (0.238g, 0.001mol) of $CoCl_2.6H_2O$ (5ml) ethanol. The mixture was stirred for two hours, during the time the color was changed and the precipitate was formed.

To this mixture (0.198g, 0.001mol) of 1, 10-phenanthroline in (5ml) ethanol was added with stirring for two hours. The resulting metal complex was fillered off, washed with cooled ethanol, ether, recrystallized from ethanol Thakare & Mandlik, 2017; Tamiru *et al.*, 2019). (Other Complexes were prepared by the same procedure a bove,using metal(Ⅱ) Chlorides of Mn^{2+}, Fe^{2+}, Co^{2+} ,Ni^{2+}, Cu^{2+}, Zn^{2+} with 1,10-phenanthroline (phen), ethylenediamine(en) Ligand. Table (1) shows the weight of metal ions and ligands.

Table 1: The weight of Metal ions and Ligands.

No.	Com. Ligand(chalcone)	Weight (2 : 1 : 1) (L : M : phen-en)
□	L	0.45gm
	Metal	
1	$MnCl_2.4H_2O$	0.198 gm
2	$FeCl_2.4H_2O$	0.199 gm
3	$CoCl_2.6H_2O$	0.238 gm
4	$NiCl_2.6H_2O$	0.238 gm
5	$CuCl_2.2H_2O$	0.171 gm
6	$ZnCl_2$	0.136 gm
	Ligand[diamine =(A)]	
1	□phen.H_2O	0.198 gm
2	en	0.060 gm

RESULTS AND DISCUSSION

The melting points of Complexes were determined and compared with the corresponding Ligand in order to ensure the formation of complexes .All complexes were in soluble in water but were soluble in DMF and DMSO.

Elemental analysis (C.H.N) and metal content values were in a good agree meat with proposed structure formula. (Table 2).

3.1 Molar conductivity measure ments

The molar conductance of the complexes were determined at 10^{-3}M dilution and at 25c° in DMF solvent (Table3). The values of conductivity with in range (11-18$cm^{-1}.ohm^{-1}.mol^{-1}$) suggested that non-electrolyte in nature (Numan` *et al*, 2015).

Table 2: Physical properties , clemental and Metal analysis of ligands and complexes.

						Elemental analysis			
Com.	Mol.Formula	Color	M.wt $gm.mol^{-1}$	Yield	M.P (C°)	C % Cal (Found)	H % Cal (Found)	N % Cal (Found)	M % Col (Found)
I	HPPO (L)	brown	225	44	180-182	74.66 (73.95)	4.888 (4.48)	6.222 (5.874)	----- ------
1	$[Mn(L)_2(phen)]$	green	682.94	56	186-188	70.28 (69.60)	4.099 (4.025	8.199 (8140	8.044 (7.085
2	$[Fe(L)_2(phen)]$	dark red	683.85	58	168-170	70.19 -----	4.094 -----	8.188 ----	8.166 -----
3	$[Co(L)_2(phen)]$	green	686.93	48	175-177	69.87 (69.10	4.076 (4.013)	8.152 (7.085)	8.578 (7.915
4	$[Ni(L)_2(phen)]$	dark g reen	686.69	58	300d	69.90 ----	4.077 -----	8.155 ----	8.581 ----
5	$[Cu(L)_2(phen)]$	Light green	691.55	59	171-173	69.40 (68.60)	4.048 (3.065)	8.097 (8.015)	9.189 (9.115)
6	$[Zn(L)_2(phen)]$	Light green	693.39	61	178-180	69.22 (68.60)	4.038 (3.057)	8.076 (7.090)	9.430 (9.350)
7	$[Mn(L)_2(en)]$	dark brown	562.94	41	153-155	63.94 -----	4.973 -----	9.947 -----	9.937 -----
8	$[Fe(L)_2(en)]$	grey	563.85	32	172-174	63.84 (62.00)	4.965 (4.872)	9.931 (9.853)	9.905 (9.818)
9	$[Co(L)_2(en)]$	dark brown	563.63	63	188-190	63.87 -----	4.967 -----	9.935 ----	10.45 -----
10	$[Ni(L)_2(en)]$	Light green	566.69	44	226-228	63.52 (62.63)	4.940 (4.857)	9.881 (9.793)	10.35 (9.047)
11	$[Cu(L)_2(en)]$	green Dark	571.55	40	172-174	62.98 (62.07)	4.898 (4.807)	9.797 (9.706)	11.11 (10.22)
12	$[Zn(L)_2(en)]$	Light green	573.39	71	160-162	62.78 ------	4.883 ------	9.766 ------	11.40 (10.66)

3.2 ^{1}HNMR Spectrum of HPPO (L)

The ^{1}HNMR spectrum of HPPO Ligand Fig (2) was measured using DMSO solvent with TMS as internal reference. The spectrum showed double band at δ(7.87ppm) and at δ(8.10ppm) refers to aliphatic (-CH=CH-) , multi bands at δ(6.90-9.27ppm) belonging to 4H of pyridine. (Balaji, 2019).

3.3 Magnetic Susceptibility and Electronic spectra measurements

The Ligand and all prepared complexes were showed bands between (32051-44247cm^{-1}) assigned to $\pi \rightarrow \pi^*$ and (27932-33557cm^{-1}) $n \rightarrow \pi^*$ respectively (Table3) Fig (3), Fig (4), Fig(5).

(Kannan *et al*., 2019). The magnetic moment of Mn(Ⅱ) complexes (1,7) were found. to be (5.22,5.91 B.M)(Table3) due to the high spin octahedral geometry(Nicholls,1984; Balaji,2019) While the d-d transition in electronic spectra do not show any band as expected, Fig(4).

The magnetic moment of iron (Ⅱ) complexes are (5.46, 5.61B. M) suggesting an octahedral geometry of these complexes.

The electronic spectra of the iron(Ⅱ) complexes (2,8) show bands at(13054,15673cm^{-1}) referring to ($^5T_2g \rightarrow {}^5Eg$) transition confirms the octahedral (Table 3). geometry around Fe(Ⅱ) ion . (Al-Mukhter& Mustfa, 1988; Barakat et al., 2015)

The magnetic moments of the Co (Ⅱ) complexes (3,9) are (4.22,5.1B.M) suggesting an octahedral geometry of these complexes. This geometry octahedral high spin was confirmed electronic spectra as show three bands at (11185, 11560cm^{-1}), (17123, 17605cm^{-1}), (20000, 20920cm^{-1}) due to.

$^4T_1g \rightarrow {}^4T_2g$ (F)υ_1 , ^4T_1g(F)$\rightarrow {}^4A_2g\ \upsilon_2$, ^4T_1g(p)$\rightarrow {}^4T_2g\ \upsilon_3$, (Table 3).

Transition respectively. (Sutton, 1968; Tseetharama rao *et al*., 1988)

The magnetic moment of Ni(Ⅱ) complexes(4,10) are (2.97-3.69B.M) indicating the octahedral geometry (Akila&Rajavel,2012; Habib,2017) (Table 3) the three peaks in visible region at (11111,11415cm^{-1}) , (16129,16722cm^{-1}) , (21739,22831) due to, $^3A_2g(F) \rightarrow {}^3T_2g(F)$ υ_1 , $^3A_2g(F) \rightarrow {}^3T_2g(F)\ \upsilon_2$and $^3A_2g(F) \rightarrow {}^3T_2g(p)\ \upsilon_3$

Transitions respectively are characteristic of the octahedral nickel ion (Akila & Rajavel, 2012; Seema, 2017).The magnetic moment of Cu(Ⅱ) complexes(5,11) are (1.92,2.48 B.M) in dicating an octahedral (Afkar,1994; Verma& Aravindhakshan, 2017) geometry of Cu (Ⅱ) was further confirmed by the bands appeared at (15772,16666cm^{-1}) (Table 3).

Referring to $^{2}Eg \rightarrow {}^{2}T_{2}g$ which is in a good agerrment with octahedral geometry (Table3) (Akila& Rajavel, 2012; Priya Verma *et al*., 2017)

The Zinc (Ⅱ) complexes (6,12) have only charge transfer transition from metal to ligand as expected. These complexes were diamagnetic and octahedral (Kannan *et al*., 2019; Rajeswari et al., 2021). As show in (Table3).

Table 3: Electronic spectra, Conductivity and Magnetic moments of Complexes.

		UV-Vis spectra(cm^{-1})		Conductivity $cm^{-1}.ohm^{-1}.mol^{-1}$ DMSO 10^{-3}	$\mathcal{M}$eff (B.M)	Sug.
No.	**Compounds**	**A.**	**Transition**			
I	L (HPPO)	27932 33112,44247	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$	-----	----	----
1	$[Mn(L)_2(phen)]$	27932 32258	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$	12	5.91	Oh
2	$[Fe(L)_2(phen)]$	32467 39062 15673	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $^{5}T_{2}g \rightarrow {}^{5}Eg$	15	5.61	Oh
3	$[Co(L)_2(phen)]$	32051 40983 11560 17123 20000	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $T_1g^{4} \rightarrow T_2g^{4}$ $T_1g^{4} \rightarrow A_2g^{4}$ $T_1g^{4} \rightarrow T_2g^{4}$	11	5.1	Oh
4	$[Ni(L)_2(phen)]$	32051,45045 11111 16129 22831	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $^{3}A_2g\ (F) \rightarrow {}^{3}T_2g\ (F)$ $^{3}A_2g\ (F) \rightarrow {}^{3}T_1g\ (F$ $^{3}A_2g\ (F) \rightarrow {}^{3}A_2g\ (F)$	17	3.69	Oh
5	$[Cu(L)_2(phen)]$	32258,39370 15772	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $^{2}Eg \rightarrow {}^{2}t_2g$	18	1.92	Oh
6	$[Zn(L)_2(phen)]$	32679 , 41987	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$	12	----	----
7	$[Mn(L)_2(en)]$	30487,44247	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$	13	5.22	Oh
8	$[Fe(L)_2(en)]$	32258,43103 13054	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $^{5}T_{2}g \rightarrow {}^{5}Eg$	17	5.46	Oh
9	$[Co(L)_2(en)]$	27932 11185 17605 20920	$n \rightarrow \pi^{*}$ $\pi \rightarrow \pi^{*}$ $T_1g^{4} \rightarrow T_2g^{4}$ $T_1g^{4} \rightarrow A_2g^{4}$ $T_1g^{4} \rightarrow T_2g^{4}$	12	4.22	Oh

10	$[Ni(L)_2(en)]$	42735 11415 16722 21739	$n\rightarrow\pi^*$ $\pi\rightarrow\pi^*$ $^3A_2g\ (F)\rightarrow{}^3T_2g$ (F) $^3A_2g\ (F)\rightarrow{}^3T_1g$ (F $^3A_2g\ (F)\rightarrow{}^3A_2g$ (F)	16	2.97	Oh
11	$[Cu(L)_2(en)]$	32051 16666	$n\rightarrow\pi^*$ $\pi\rightarrow\pi^*$ $^2Eg\rightarrow{}^2T_2g$	14	2.48	Oh
12	$[Zn(L)_2(en)]$	33557, 42016	$n\rightarrow\pi^*$ $\pi\rightarrow\pi^*$	11	----	----

3.4 Infrared spectra

The changes between the free ligands and theis complexes displayed by IR spectra to give an idea about the type of coordination and their structure. (Table 4), Fig (6), Fig (7). (Little *et al.*, 2016) in ligand (chalcone) (L) (HPPO) hydroxyl strefching band is appeared at (3452cm^{-1}) which is completely disappeared in metal complexes indicating deprotonation phenolic group in coordination with metal ion.

This has been supported by wpward shift in υ(C-O) (Phenolic) stretching frequency to the extent (4-10cm^{-1}) (Thakare&Mandlik,2017) Aband due to carbonyl group at (1681cm^{-1}) again shifted to lower frequency at extent (25-41 cm^{-1}) suggest coordination of carbonyl oxygen with metal ion . (Seema, 2017; Thakare& Mandlik, 2017).

At the same time new spectral band appeared in spectra of complexes in region (532-595 cm^{-1}) due to M-O stretching vibration (Seema, 2017). The phen shows a stroug absorption at (1557 cm^{-1}) which assigned to C=N the complexes of phen showed absorption at (1417-1458 cm^{-1}) (Pnagababu *et al.*, 2009)

And the presence of a new band between (420-480 cm^{-1}) which are assigned to the υ(M-N) in spectra of complexes in dications of (Hutchinson et al.,1970). coordination of nitrogen atom of en and phen with metal ion(Table4) .

Table 4: Infrared of selected bands for ligands and complexes cm^{-1} suggested compositions.

No.	Com.	C=O	C=C Alkyl	C=C Aro	C-O	C-H	C=N AIMP	C=N hen	O-H	M-N	M-O	M-OH_2	NH_2
I	(HPPOL)	1681	1625	1594	1342	3078	----	----	3452	----	----	----	----
1	$[Mn(L)_2(phen)]$	1645	1628	1595	1346	3047	---	1421	----	470	570	-----	----
2	$[Fe(L)_2(phen)]$	1640	1625	1596	1352	3051	---	1425	---	420	532	-----	----
3	$[Co(L)_2(phen)]$	1656	1623	1595	1350	3053	----	1417	---	459	543	-----	----
4	$[Ni(L)_2(phen)]$	1656	1623	1593	1350	3082	---	1458	-----	449	588	-----	----
5	$[Cu(L)_2(phen)]$	1656	1625	1595	1350	3051	----	1419	---	478	595	-----	----
6	$[Zn(L)_2(phen)]$	1652	1625	1596	1350	3055	---	1425	---	450	594	-----	----
7	$[Mn(L)_2(en)]$	1655	1623	1596	1350	3030	---	---	----	480	568	-----	----
8	$[Fe(L)_2(en)]$	1656	1625	1595	1350	3068	---	---	----	464	570	-----	----
9	$[Co(L)_2(en)]$	1656	1625	1595	1350	3068	---	----	----	464	570	-----	----
10	$[Ni(L)_2(en)]$	1656	1625	1593	1350	3062	----	----	-----	475	592	-----	----
11	$[Cu(L)_2(en)]$	1656	1625	1593	1350	3045	----	------	----	472	595	-----	-----
12	$[Zn(L)_2(en)]$	1656	1627	1598	1352	3053	----	----	----	472	582	----	-----

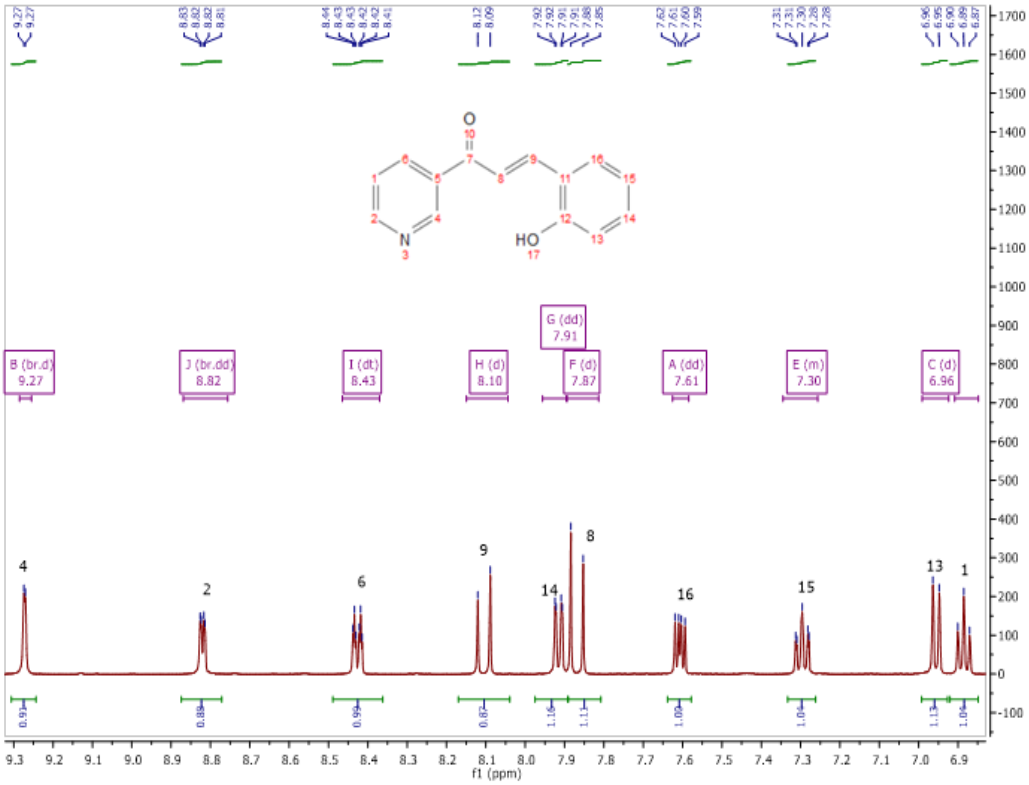


Fig. 2: The nuclear magnetic resonance spectrum of the proton L, (HPPO).

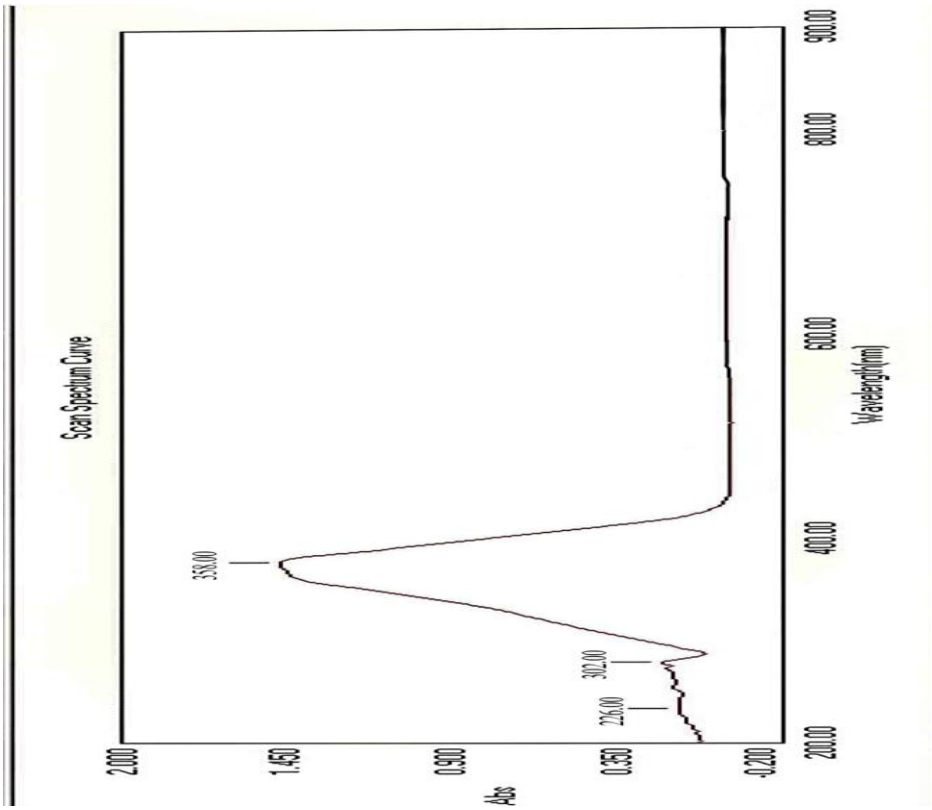


Fig. 3: Visible and Ultraviolet spectrum of the ligand L, (HPPO).

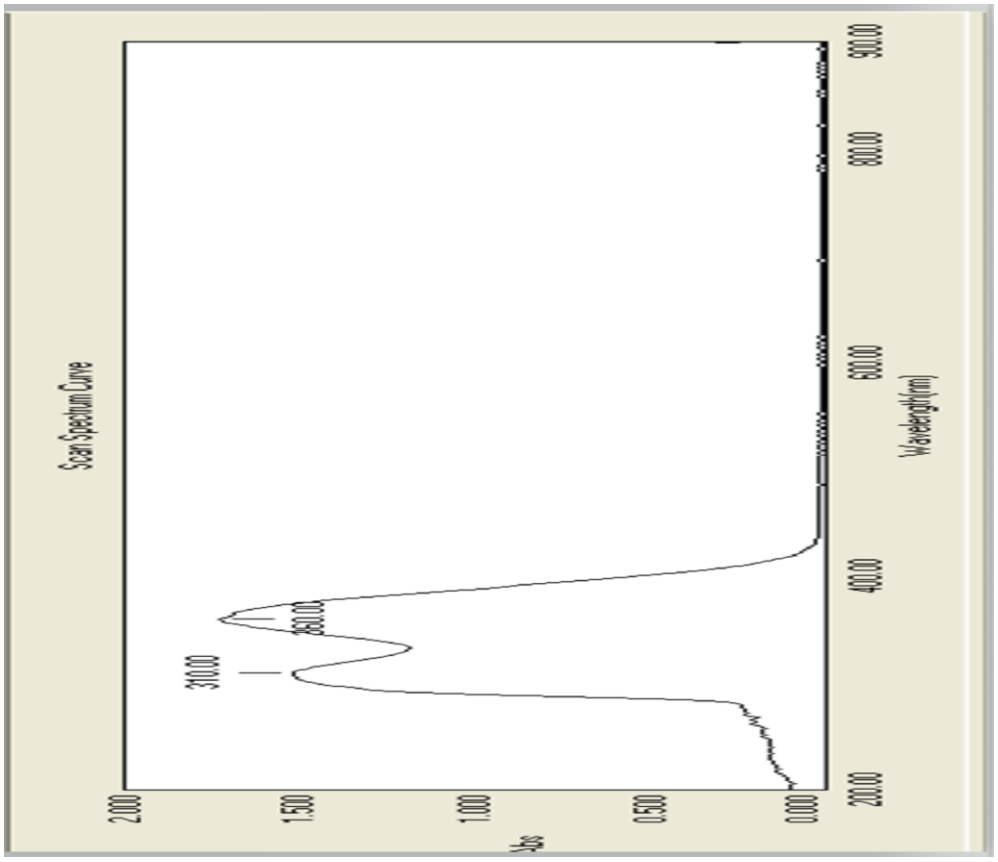


Fig. 4: The spectrum of visible and ultraviolet rays for complex [Mn(L)$_2$(phen)].

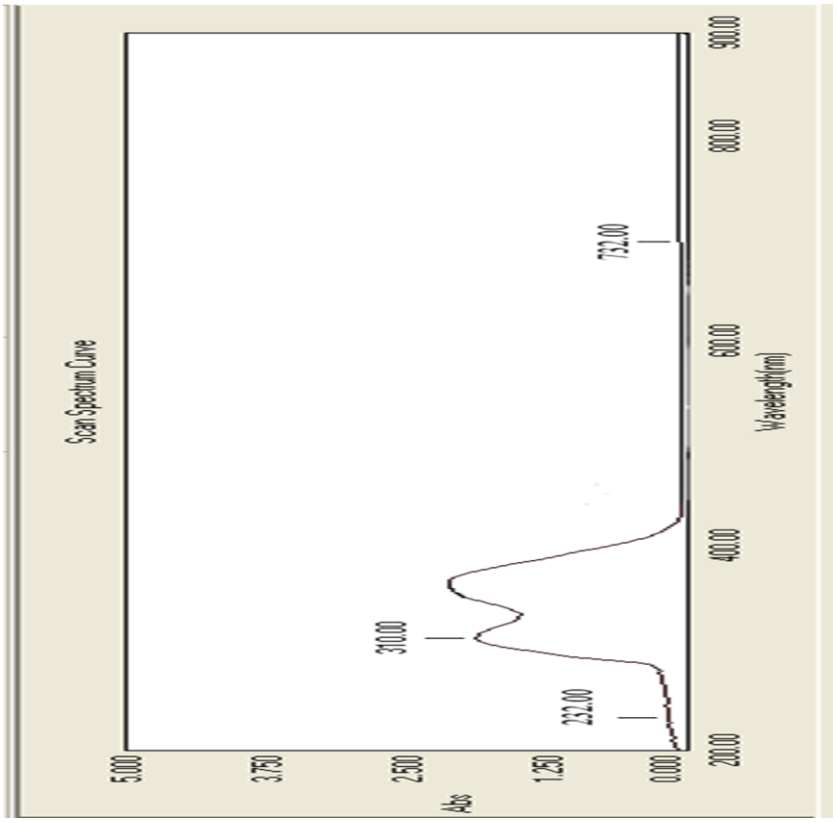


Fig. 5: The spectrum of Visible and Ultraviolet rays for complex $Fe(L)_2(en)$].

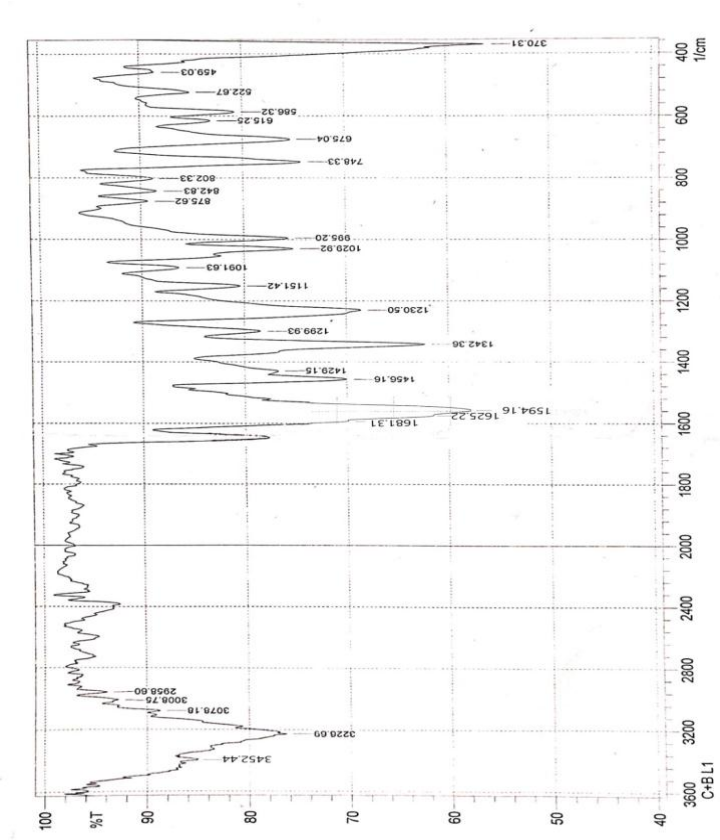


Fig. 6: Infrared spectrum of the Ligand L (HPPO).

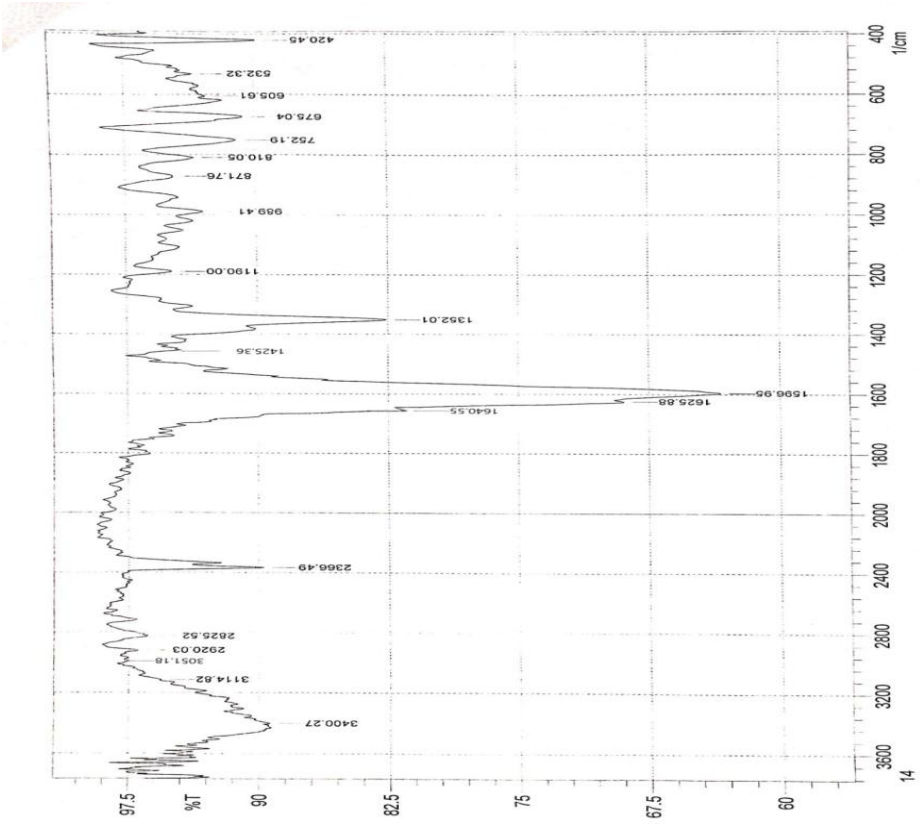


Fig. 7: Infrared spectrum of the complex [Fe (L) 2(phen)].

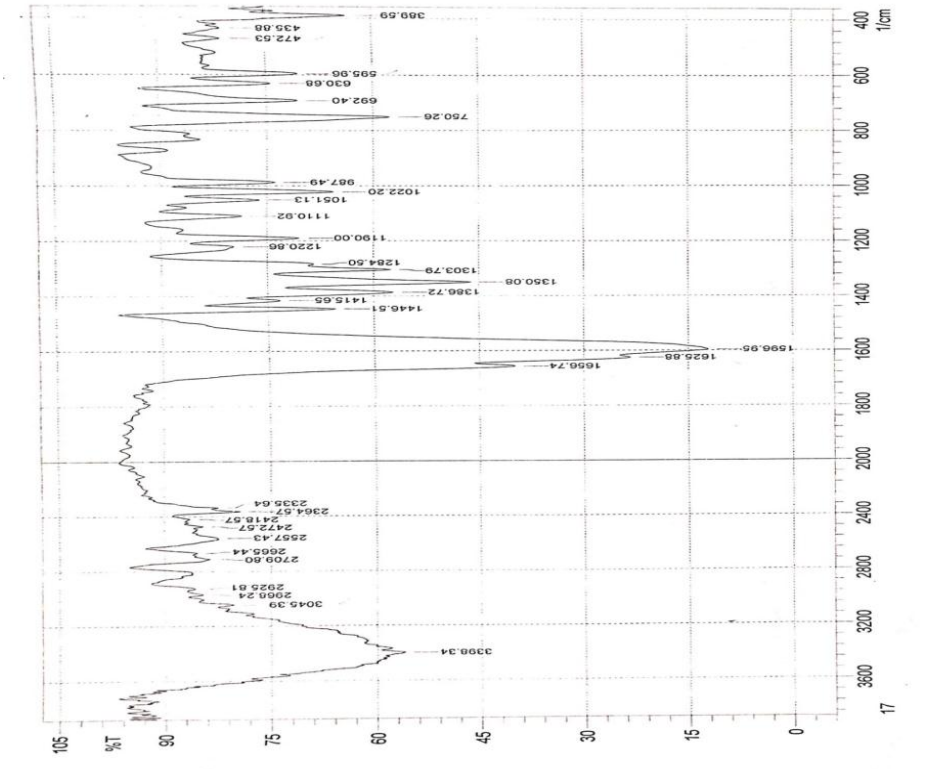


Fig. 8: Infrared spectrum of the complex [Cu $(L)_2$(en)].

Suggested compositions

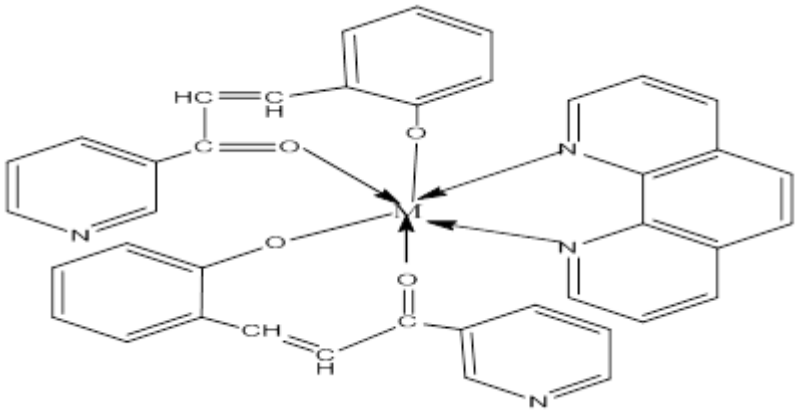


Fig. 9: Octahedral geometry for [M(HPPO)$_2$ phen].

L= HPPO

Phen=1, 10- phenanthroline

M= Mn(Ⅱ), Fe(Ⅱ), Co(Ⅱ), Ni(Ⅱ), Cu(Ⅱ) and Zn(Ⅱ).

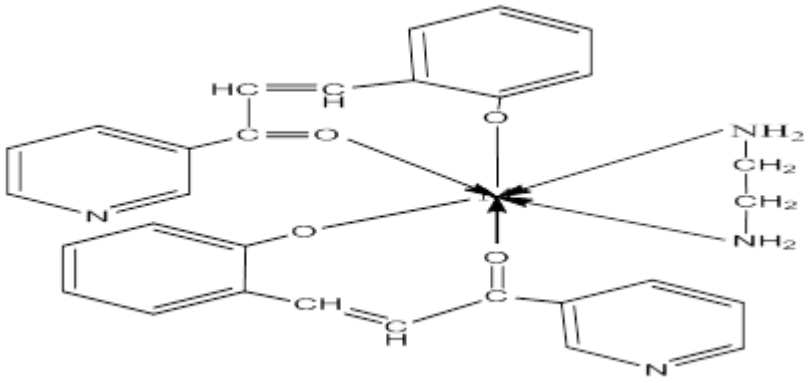


Fig. 10: Octahedral geometry for [M(HPPO)$_2$ en].

L= HPPO

en= ethylenediamine

M= Mn (Ⅱ), Fe(Ⅱ), Co(Ⅱ), Ni(Ⅱ), Cu(Ⅱ) and Zn(Ⅱ).

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