Synthesis and Characterization of a Quadridentate Ligand Type N₄ and Their Complexes with Some Metal Ions

تحضير وتشخيص الليكاند رباعي السن نوع N₄ ومعقداته مع بعض الايونات الفلزية

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Abstract

A quadridentate ligand type N₄ has been prepared by reaction of one equivalent of di-ethyl phthalate with two equivalent ethylene diamine under N_2 atmosphere , which gives one equivalent of [*N, N`-Bis*-(2-amino- ethyl) Phthalamide] (I), then reaction of the condensation of product (I) with [2,5- hexane- dione] under N_2 atmosphere gave quadridentate ligand [H₂L] type N₄,*(10,13-Dimethyl-6,7,8,11,12,15,16,17-octahydro-6,9,14 ,17-tetraaza-benzocyclohexadecene-5,18-dione*) . Complexes of the ligand [H₂L] with Cr^{+3} , Co^{+2} , Ni^{+2}, Pd^{+2}، Cu^{+2}, Zn^{+2}, Cd^{+2} and Hg^{+2} ions in general formula $[MLX_n]^m$ were prepared Where M=Cr^{+3}, X =Cl, n=2, m=+1, and M= Co^{+2} , Ni^{+2}, Pd^{+2}, , Zn^{+2}, Cd^{+2} and Hg^{+2} ions, X=0, n=0, m=+2, and M= Cu^{+2} , X=Cl , n=2, m=0. The Ligand and it's complexes have been characterized by microanalyses of elements, CHN along with conductivity measurement and magnetic moment, spectroscopic methods, (UV-Vis) IR and flame atomic absorption spectroscopy . The results showed that the suggested and proposed molecular structure for Cr^{+3} and Cu^{+2} complexes was octahedral, and for pd^{+2} and Ni^{+2} complexes was square planner, while for Co^{+2}, Zn^{+2} ,Cd^{+2} and Hg^{+2} complexes are tetrahedral.

الخلاصة

تم تحضير ليكاند رباعي السن نوع N₄ من تفاعل ثنائي أثيل الفثالات مع أثلين ثنائي الأمين وتحت جو خامل من غاز النتروجين , وأعطى ناتج التفاعل المركب (I): Phthalamide (2-amino- ethyl)-*Bis`-N,N* , ثم تفاعل المركب الناتج (I) مع هكسان- دايون وتحت جو خامل من غاز النتروجين أيضا ليعطي الليكاند [H₂L] رباعي السن .-*10,13 Dimethyl-6,7,8,11,12,15,16,17-octahydro-6,9,14 ,17-tetraaza-benzocyclohexadecene-5,18-dione*)

تم تحضير سلسلة من معقدات الليكاند [H₂L] مع الايونات الفلزية : الكروم III , الكوبالتII , النيكل II , النحاس II , البلاديوم II , الخارصين II , الكادميوم II والزئبق II ذوات الصيغة العامة m $[M L X_n]$ حيث تمثل : Cr=M+3 , X=Cl , n=2 , m=-1 وتمثل M = Cd+2, Zn+2 pd+2 , Hg+2 , Ni+2, Co+2 , X=0 , و X=0 , n= 0, m=0 و Cu+2 = M , X=Cl , n=2 , .m=-2.

استخدمت طرائق التحليل الكمي الدقيق للعناصر CHN , التوصيلية المولارية , قياس محتوى الكلور والحساسية المغناطيسية فضلا عن الطرائق الطيفية : امتصاص الأشعة فوق البنفسجية – المرئية UV-Vis والأشعة تحت الحمراء IR والامتصاص الذري أللهبي AAS لتحليل وتشخيص الليكاند وجميع معقداته.

أظهرت النتائج أن الشكل المقترح لمعقدات الكروم III و النحاس II هو ثماني السطوح, وان الشكل المقترح لمعقدات البلاديوم II و النيكل II هو المربع المستوي , بينما يكون شكل معقدات ايونات الكوبالتII الخارصين II , الكادميوم II و الزئبق II هو رباعي السطوح .

Introduction

Transition metals was play a central role in the construction of molecular materials, Which display magnetic properties and find applications in materials and supramolecular chemistry and biochemistry [1,2]. Metal Complexes of Cr^{+3}, Co^{+2} and Cu^{+2} with O, N and S donor macrocyclic ligands can increase the activity of insulin by binding to a small chromium binding to protein[3]. The well- known macrocyclic like crown ether phthalocynin and porphyrine form a series of coordination compounds with well defined structures. It plays an importants role in the numerous biological processes that involve electron transfer reactions or the activation of some anti-tumor substances[4]. Porphyrin compounds and phthalosyanin are used to extracted Co^{+2} , Mn^{+2}, Pd^{+2}, Co^{+2} , Pb^{+2}, Zn^{+2} , and Cu^{+2} metal ions at law concentration [5] .

This work was aimed to synthesis and characterization a new quadridentate ligand type N_4 and it 's complexes with Cr^{+3}, Co^{+2} Cu^{+2}, Ni^{+2}, Pd^{+2}, Zn^{+2}, Cd^{+2} and Hg^{+2} metal ions .

Experimental

1- Apparatus

A Shimadzu-160 double beam UV- spectrophotometer was used for the analysis and measuring of the prepared compounds in the region 200-1100 nm using 1cm quartz cells for 10^{-3} M solution in DMF at 25° C. A Shimadzu 8300 FT-IR spectrophotometer in the range 400-4000 cm^{-1} was used and recorded IR spectra as KBr disc. Heraeus (Vario EL) elemental microanalysis (CHN) analyzer was used. A 686-Swiss potentiometer was used as potentiometric titration method for the determination of chloride contents in complexes.

A PW-9526 digital conductivity meter was used for conduction measurements of the complexes solution $(10^{-3}M)$ in DMF.

2- Materials:

The reagents used in this, work were commercial products (Fluka, BDH and Duheng) .All glassware was cleaned with detergent and water, rinsed with distilled water, dried and rinsed with ethanol several time before using.

3- Preparation of Samples :

A- Precursor *N, N`-Bis*-(2-amino- ethyl) phthalamide:

(10g, 4.5 mmole) of Di-ethyl phthalate, was added drop wise with stirring to (5.4g, 9.0 mmole) of 1.2-ethylene diamine, a viscous mixture was obtained. Un-reacted starting material and produced methanol were removed by distillation under vacuum (reduced pressure). A white solid was collected, washed with 5ml diethyl ether, and dried. Table(1) shows Physical properties of the prepared compounds.

B- Synthesis of the ligand [H₂L]:

A solution of *N, N`-Bis*-(2-amino- ethyl) Phthalamide (2.66g, 10 mmole) in ethanol (15 mL) was added slowly to 2, 5-hexane- dione (1.14 gm, 10 mmole) dissolved in ethanol (15 mL). The mixture was reflux for two hours under inert nitrogen blanket, and then cooled to room temperature. (Table -1)

C- Synthesis of the metal complexes (C₁-C₈) :

A methanolic solution (0.1g, 0.3 mmole) of the ligand [H₂L] in 15 ml was added to (0.07g, 0.3 mmole) of the metal chloride [$CrCl_3$.$6H_2O$, $NiCl_2.6H_2O$, $CuCl_2$. $2H_2O$, $ZnCl_2$, $CdCl_2$ and $HgCl_2$]

dissolved in 15ml methanol. The mixture was refluxed for 2 hrs and left at room temperature for cooled. A colored crude of the (C_1 –C_7) complexes was formed, which was filtered, washed with ether several times, and dried. A similar method was carried out for preparation of Pd^{+2} complex by dissolving (1.21gm, 0. 08mmole) $PdCl_2$ in 2 mole of 2% HCl and (0.5g, 0.08mmole) NaCl to formed $PdCl_4^{-2}$, which consecutively reacted with 0.33mole of H_2L in 15ml methanol and refluxed for 2hrs . Table (1) summarized the quantities compounds; colored of complexes, weight of metal chloride some physical properties of these prepared complexes. Scheme (3-4).

D- Stoichiometric Ion Complex Formation:

The [H_2L] ligand complexes with Cr^{+3} , Co^{+2} ,Ni^{+2}, Pd^{+2}, Cu^{+2}, Zn^{+2}, Cd^{+2} and Hg^{+2} metal ions was studied in solution using methanol as solvent, in order to determination M:L ratio in the prepared complexes following continuous variation (Job's) method[6] . A series of solutions were prepared having varied concentration, of [H_2L] ligand and metal chloride at total constant concentration 10^{-3} M. The M: L ratio was found from the plot of absorbance at λ_{max} of the formation complexes versus the mole fraction.

Results and Discussion:

A- Characterization of N, N`-Bis-(2-amino- ethyl) Phthalamide (I):

The precursor N, N`-Bis-(2-amino- ethyl) Phthalamide (Scheme-1) was characterized by elemental analysis CHN, Table-2, and IR spectra (Table-3). The IR spectrum of starting material, 1,2-ethylenediamine , showed a bands at 3500- 3400cm⁻¹ attributed to asymmetric - stretching υ(N–H), and symmetric-stretching υ(N-H) , and a band at 1565 cm⁻¹ which referred to bending (N-H) [7], while the IR spectrum of phthalate di-ester showed a bands at 1726-1172 cm⁻¹ attributed to esterric stretching carbonyl group υ(C=O) and stretching υ(OCH₃) group respectively [8]. The IR spectrum of precursor showed the two amide groups are non–equivalent, this could be attributed to either the formation intra- or inter-hydrogen bonding in the ligand. While the band at 1670 cm⁻¹ attributed to υ (C=O), also the band at 3396-3269 cm-¹ attributed to NH_2 groups. The bands at 1552, 1496, 1161 cm⁻¹ can be referred to bending δ (N-H), υ (C=C), stretching υ(C-N) respectively [9].

B- Characterization of Ligand [H_2L]: *10,13-Dimethyl-6,7,8,11,12,15,16,17-octahydro-6,9,14 ,17-tetraaza-benzocyclohexadecene-5,18-dione*

The condensation reaction of *N,N`-Bis*-(2-amino- ethyl) Phthalamide with 2,5-hexane- dione, resulted in the preparation of the ligand [H_2L] according to the general method shown in Scheme-2. The ligand was characterized by elemental analysis, (Table-2), IR spectra (Table-3), UV-Vis spectra (Table-4). IR spectrum of the ligand, Fig.(4), displays a band at 1724, 1641 cm⁻¹ due to υ (C=O) and v(C=N) stretching of the imine groups [10,11]. These two groups are in different environment due to intra-molecular hydrogen bonding while the band at 3326 cm⁻¹ attributed to stretching (O-H). The appearance of the imine band indicated the formation of [H_2L]. Finally the spectrum displayed band at 1270cm⁻¹ which may be related to stretching phenolic (C-O) frequency.

The IR spectrum showed the two amide groups are non-equivalent, they could be assigned to either the formation intra or inter hydrogen bonding neither the ligands nor the appearance of the ligand in two different forms. The medium bands observed at 904 and 792 cm⁻¹ were assigned to the macrocyclic frame work [11],on the other hand, the bands at 1710 cm⁻¹ in 2,5- hexane-dione spectrum assigned to stretching v(C=O) was disappeared in the ligand spectrum [12, 13] . According to above of IR spectra result the following ligand that tetredintate di-base quadridentate.

UV-Vis spectrum of the ligand [H_2L], Fig. (7) Exhibits two absorption peaks at 304 nm and 373 nm, assigned ($\pi \rightarrow \pi^*$) and (n $\rightarrow \pi^*$) electronic transitions respectively [5] .

C- Characterization of complexes

All complexes were prepared by a similar method except (Pd^{+2}), shown in (Scheme-3, 4). The complexes are stable in solution and in solid state.

The molar conductance of the complex (C_1) in DMF solvent is 67 ohm^{-1} cm^2.$mole^{-1}$, which indicates 1:1 ratio, while indicates 2:1 ratio (C_5) for Cu^{+2} . The molar conductance of the complexes Co^{+2} , Ni^{+2} , Pd^{+2} , Zn^{+2}, Cd^{+2} and Hg^{+2} metal ions complexes [14] in DMF solvent at 28.9 ohm^{-1} cm^2.$mole^{-1}$ indicates non electrolytic behavior.

The chloride contents for all complexes showed that only the Cr^{+3} and Cu^{+2} complex contain chloride, which mean and indication the coordination between Cr^{+3} , Cu^{+2} and chloride.
The analytical and physical data Table -2, and spectral data Tables 3, 4 and 5 are compatible with the suggested structure. (Scheme-3.)

D- Stoichiometric Relationship:

Applying molar ratio method, the reaction stoichiometry of complexes between [H_2L] ligand with metal ions was found to be 1:1. The formation of the complexes was shown in the reaction scheme given below, (scheme- 3, 4).

IR- spectra

The IR spectra of all complexes, presented in (table-3) showed bands at range 1636-1602cm^{-1} due to v(C=N) which were shifted to lower frequency when it comparison with the band that of free ligand [H_2L], also two bands of v(C=O) which appeared at range 1688-1641cm^{-1} and 867-814cm^{-1} in spectra of all complexes were shifted to lower frequencies. The shift in v (C=N) and v(C=O) can be related to the delocalization of the metal electronic density in to the ligand (π-system) (HOMO → LUMO) and these shifting indicated the coordination between the ligand [H_2L] and metal ions was happened *via* N atoms [15]. On the other hand, the appearances of a very broad asymmetrical band at range 3443-3327 cm^{-1}, and bending of OH in the range 1630-1640 cm^{-1} assigned to binding of water molecule *via* hydrogen which were attributed to an asymmetric v(OH) and (HOH) bending vibrational modes respectively of lattice water[15,16]. The spectrum shows a new band at 1650cm^{-1} due to the stretching v(C=N) of the imine group, which is in an agreement with the results reported. The appearance band (Fig. 3–5) indicated the preparation of the ligand.

On the other hand the band at 1270 cm^{-1} due to v(C-O) in the free ligand was shifted to lower frequency and appeared at range 1260 cm^{-1} and 1255 cm^{-1} only in IR spectra of Ni^{+2} and Cu^{+2} complexes respectively, these shifting was confirmed by formation of new bands at 462 cm^{-1} and 450 cm^{-1} in IR spectra of Ni^{+2} and Cu^{+2} complexes. The IR spectra showed new bands at range 561-402 cm^{-1} attributed to (M-N) which supports the participation of N-atoms of C=N and amido groups to linkage for metal and ligand[17] [H_2L].

UV-Vis spectra:

The electronic spectral data of all complexes are summarized in (table -4). The UV-Vis spectra of complexes displayed absorption peak at 279-344 nm assigned to ligand field and C.T. The electronic spectra of all complexes exhibited a new absorption peak at range 359- 377 nm which are attributed to charge transfer M→L. The UV-Vis spectrum of (C_1),(Fig. 9) showed absorption peaks at 285, 308nm which attributed to the ligand field , the peaks at 399 and 490 nm attributed to (d-d) electronic transition of type (^4A_2g $^{(F)}$ → ^4T_2g $^{(P)}$)and (^4A_2g $^{(F)}$ → ^4T_1g $^{(F)}$) this results was a good evidence for octahedral geometry [18]. The new absorption peak at 670 nm in electronic spectrum of [C_5] complex (Fig-11) was assigned to (d–d)electronic transition type (^2E_2g → ^2T_2g) , in fact this result is a good agreement with the previous work of cu^{+2} complexes of distorted octahedral geometry because of Jahn – Teller effect [19].
The UV-Vis spectrum of [C_3] complex show a new absorption peak at 390 nm due to (d - d) electronic transition type (^1A_1g → ^1A_2g) ,(^1A_1g → ^1B_1g) suggesting square planner structure [20,

[21].The [C$_4$] electronic spectrum appeared a new peak at 520 nm assign to(d - d) electronic transition type ($^1A_2g \rightarrow {}^1B_1g$) which is a good evidence for square planner geometry for Pd $^{+2}$ complex [22]. The UV-Vis spectra of [C$_2$] appeared a new peak at 650 nm assign to(d-d) electronic transition type ($^4A_2 \rightarrow {}^4T_1{}^{(p)}$) this results was a good evidence for tetrahedral geometry [23,24] . The UV-Vis spectra of [C$_6$, C$_7$ and C$_8$] complexes shows no absorption peak at range 279-1000 nm , which indicated that no (d-d) electronic transition occurred (d^{10} - system) in visible region , then this is a good prove result for Zn^{+2}, Cd^{+2} and Hg $^{+2}$ tetrahedral complexes [25,26] .
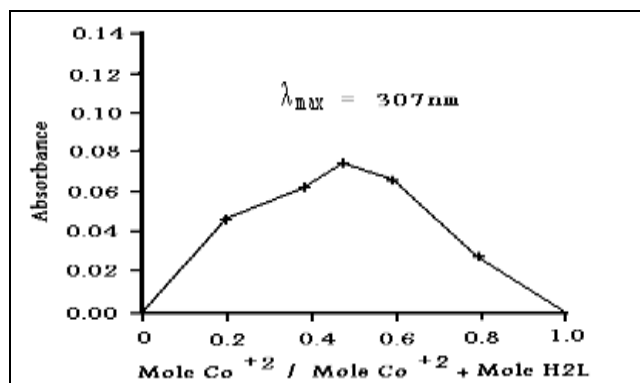
Fig.(1): continuous variation (Job's)method for [H$_2$L] ligand complexes

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Table-1: Physical properties of the prepared compounds and weight of metal chloride salt

Product compounds		color	m.p. °C	Metal chloride	Wt .of metal chloride salt (g)	Wt. of product (g)	Yield%
precursor	[I]	white	68	-	-	3.1	57
[H₂L]	[II]	brown	-	-	-	1.9	56
[Et₃NH] [Cr(L)Cl₂]	C₁	green	300	CrCl₃.6H₂O	0.081	0.07	47
[Co(L)]H₂O	C₂	brown	285	CoCl₂6H₂O	0.072	0,068	53
[Ni(L)] H₂O	C₃	brown	290	NiCl₂6H₂O	0.08	0.07	53
[Pd(L)] H₂O	C₄	brown	292-293	Na₂PdCl₄	0.088	0.09	60
[Et₂NH]₂ [Cu(L)Cl₂]	C₅	brown	290	CuCl₂ .2H₂O	0.039	0.08	57
[Zn(L)] H₂O	C₆	white	300 dec	ZnCl₂	0.040	0.06	42
[Cd (L)] H₂O	C₇	white	317-319 dec	CdCl₂ .H₂O	0.07	0. 08	53
[Hg (L)] H₂O	C₈	Gray	300 dec.	Hg Cl₂	0.08	0.10	55

dec. = decompose

Table -2: Elemental analysis data of [H₂L] and its metal complexes

Compounds	M. wt	% C		% H		% N		% Cl		Metal	
		Cal.	Found	Cal.	Found	Cal.	Found	Cal.	Found	Cal.	Found
C₁₂H₁₈N₄O₂	250	57.58	55.02	7.25	6.06	22.38	22.95				
C₁₈H₂₄N₄O₂	328	65.83	62.49	7.37	6.52	17.06	17.17				
C₁	551	52.27	50.80	6.95	3.80	12.70	12.95	12.86	11.00	9.43	09.40
C₂	403	53.60	51.60	6.00	4.25	13.89	15.91			14.61	11.52
C₃	403	53.63	52.62	6.25	5.09	14.56	14.99			14.56	11.55
C₄	450	47.95	46.11	5.37	4.70	12.43	12.01			23.61	21.11
C₅	665	54.16	50.10	8.18	7.40	15.31	12.50	12.63	10.20	9.55	8.10
C₆	409	52.76	53.00	5.90	4.99	13.67	13.48			15.96	14.71
C₇	456	47.33	45.52	5.30	3.70	12.26	12.10			24.61	22.95
C₈	545	39.67	36.11	4.44	3.31	10.59	11.21			36.81	33.98

Table-3: Infrared spectral data (wave number)cm^{-1} of the ligand [H$_3$L] and its complexes

Compound	υ(N–H),asy υ((N-H)sy	υ(C=O)	υ(C=N)	δ(N-H)	υ(O-H)	υ(C-N)	υ(C=C)	υ(M–N)	Additional bands
Diethylene diamine	(3500-3400)			1565 s		1045		-	2920 aliph υ(C–H)
Phthalate di -ester		1726					1465		2993 υ(C–H)
[I]	3396 3269	1708 s 1670		1552		1161	1440		
[II] Ligand	3217 s	1724 s	1641	1541		1170	1406		υ (C-H)3101 aliph
C$_1$		1701	1639	1535	3403	1172	1444	561	υ (C-H)3101
C$_2$	3331	1690s	1637	1546	3443	1234	1444	488	3090m υ(C–H) arom. 2978vw υ(C–H) aliph
C$_3$	3260 3315	1697 m	1602 w	1591 w	3327	1161	1430	414w	3034m υ(C–H) arom. 2970vw υ(C–H) aliph
C$_4$	3332	1678s	1612 m	1535 m	3335	1147	1452	532	3066m υ(C–H) arom. 2954vw υ(C–H) aliph
C$_5$	3338	1674 m	1636 m 1617 m	1544s	3378	1147	1463	441	3010m υ(C–H) arom. 2940vw υ(C–H) aliph
C$_6$	3280	1690s	1620 m	1540s	3377	1149	1456	480	3001m υ(C–H) arom. 2900vw υ(C–H) aliph
C$_7$	3490	1710	1602	1560	3360	1160	1440	480	3090 arom. 2940 aliph.
C$_8$	3220	1692	1643	1559	3360	1156	1438	430	3070 arom. 2990 aliph.

Table -4 : Electronic spectral data of [H_2L] and its metal complexes

Compound	λ nm	υ cm^{-1}	ε_{max}molar^{-1} cm^{-1}	Assignment	Suggested Structure
[II]	304	32894	2500	$\pi \rightarrow \pi^*$	
	373	26809	2000	$n \rightarrow \pi^*$	
C$_1$	285	35087	1100	Ligand field	Octahedral
	308	32467	550	$^4A2g^{(F)} \rightarrow {}^4T2g^{(P)}$	
	420	23809	400	$^4A2g \rightarrow {}^4T1g^{(F)}$	
	700	14285	110	$^4A2g \rightarrow {}^4T2g^{(F)}$	
C$_2$	305	32186	1450	Ligand field	Tetrahedral
	370	27027	900	Charge transfer	
	650	153846	20	$^4A_2^{(F)} \rightarrow {}^4T_1^{(P)}$	
C$_3$	290	34482	700	Ligand field	Square planner
	390	28450	100	($^1A_1g \rightarrow {}^1B_1g$)	
C$_4$	303	33003	2300	Ligand field	Square planner
	370	27027	100	Charge transfer	
	520	19230	50	$^2B_2g \rightarrow {}^2B_1g$	
C$_5$	295	33898	2502	Ligand field	distorted Octahedral
	387	25839	1650	Charge transfer	
	670	14925	100	$^2B_2g \rightarrow {}^2B_1g$	
C$_6$	344	29069	1175	Ligand field	Tetrahedral
	359	27855	849	Charge transfer	
C$_7$	279	35842	1591	Ligand field	Tetrahedral
	378	26455	500	Charge transfer	
C$_8$	301	33222	1331	Ligand field	Tetrahedral
	377	26525	668	Charge transfer	

Table-5: Magnetic moment analysis data of the metal ion complexes

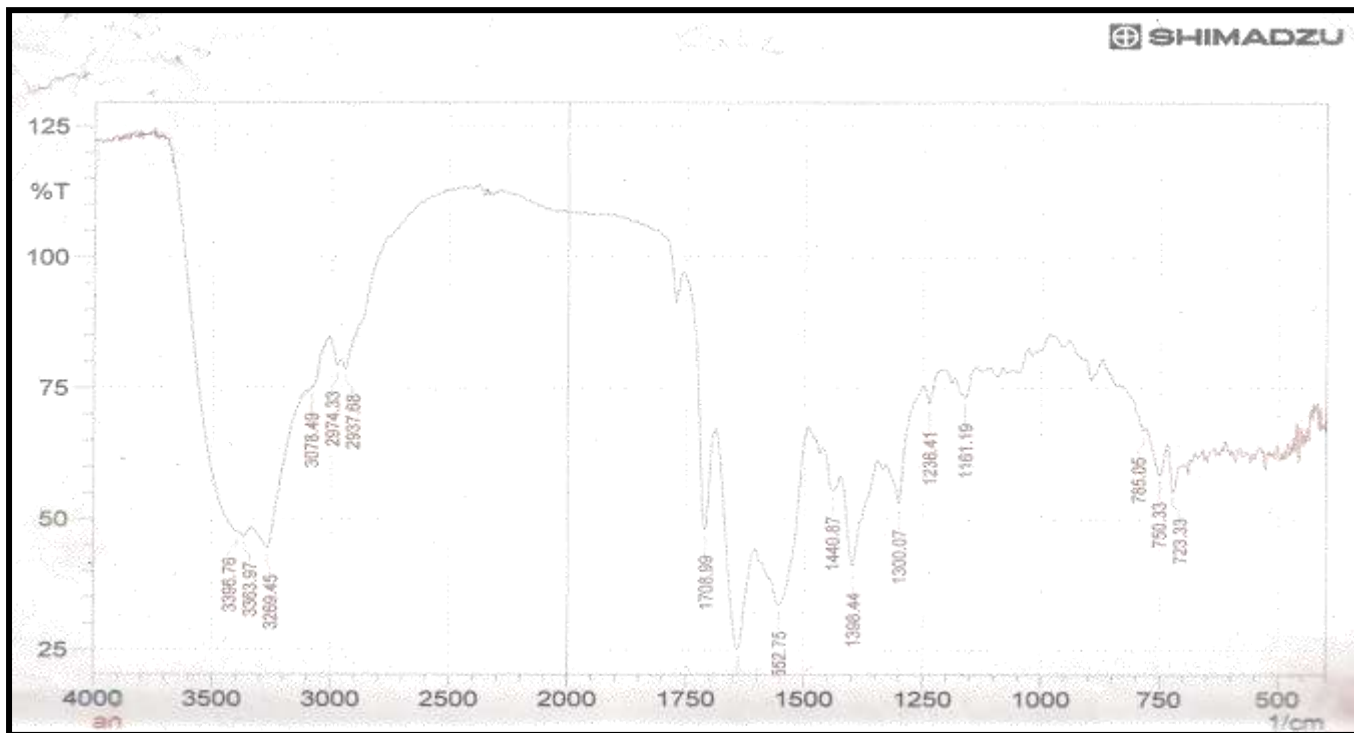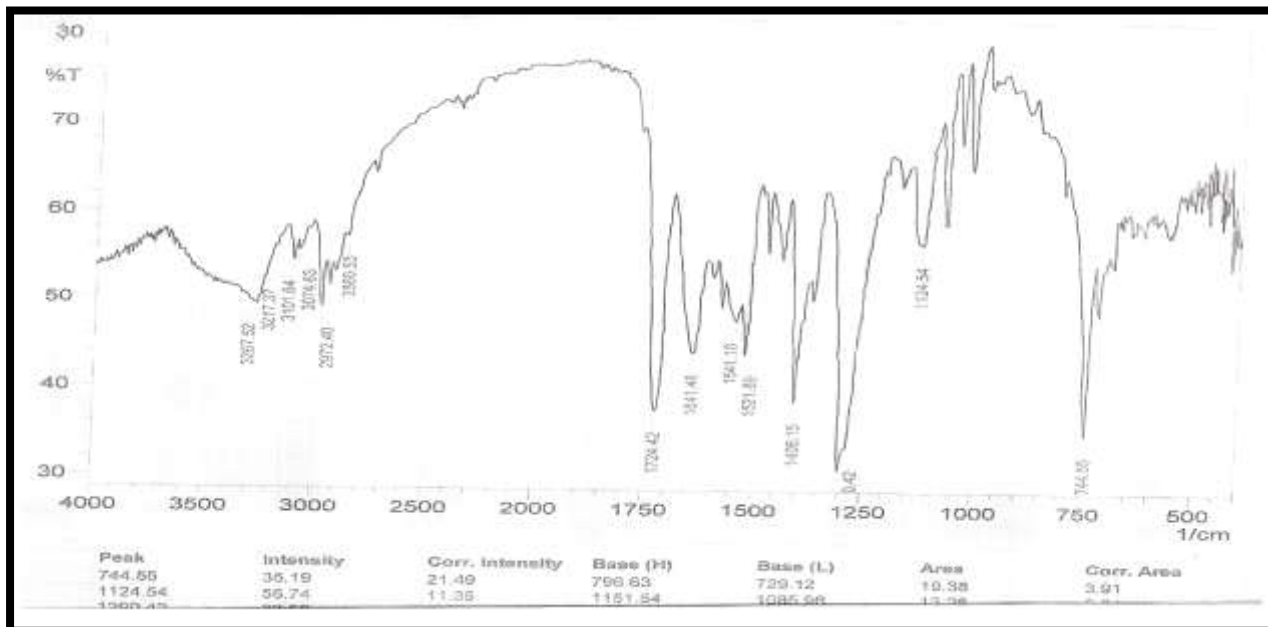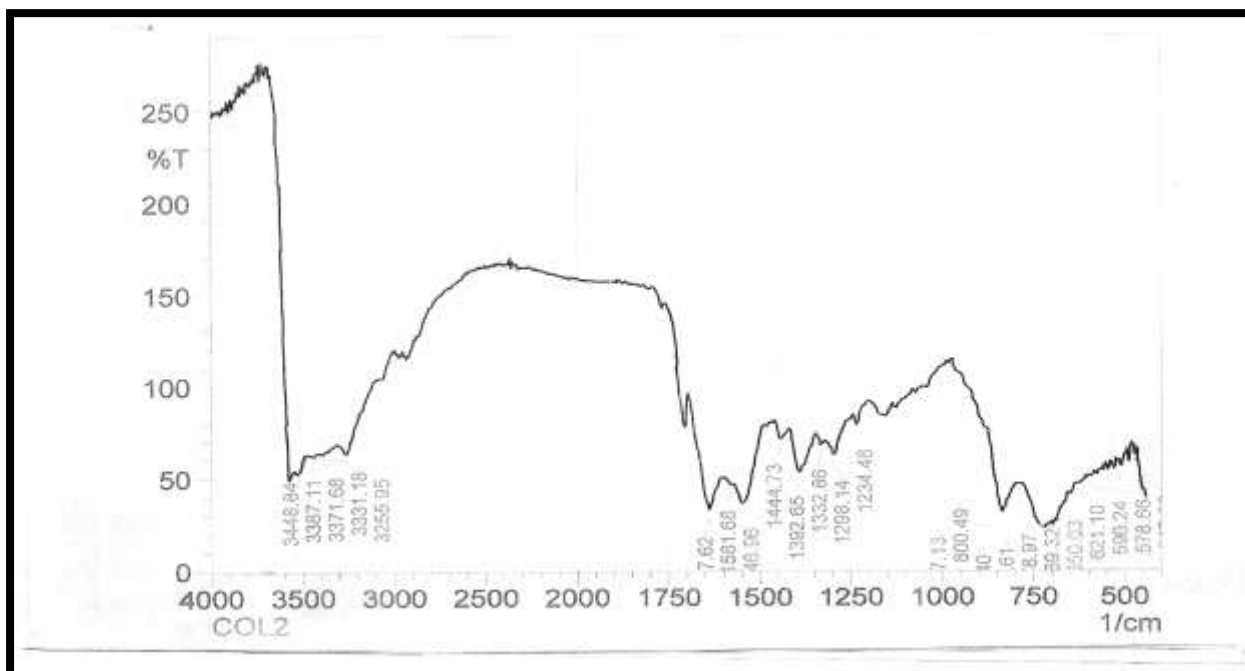| Complex	μ_{eff} B.N experiment	μ_{eff} B.N calculation	Suggested Structure
C$_1$	3.70	3,88	Octahedral
C$_2$	3.23	3.88	Tetrahedral
C$_3$	0.0	0.0	Square Planner
C$_4$	0.0	0.0	Square Planner
C$_5$	1.85	1.73	Distorted Octahedral
C$_6$	0.0	0.0	Tetrahedral
C$_7$	0.0	0.0	Tetrahedral
C$_8$	0.0	0.0	Tetrahedral

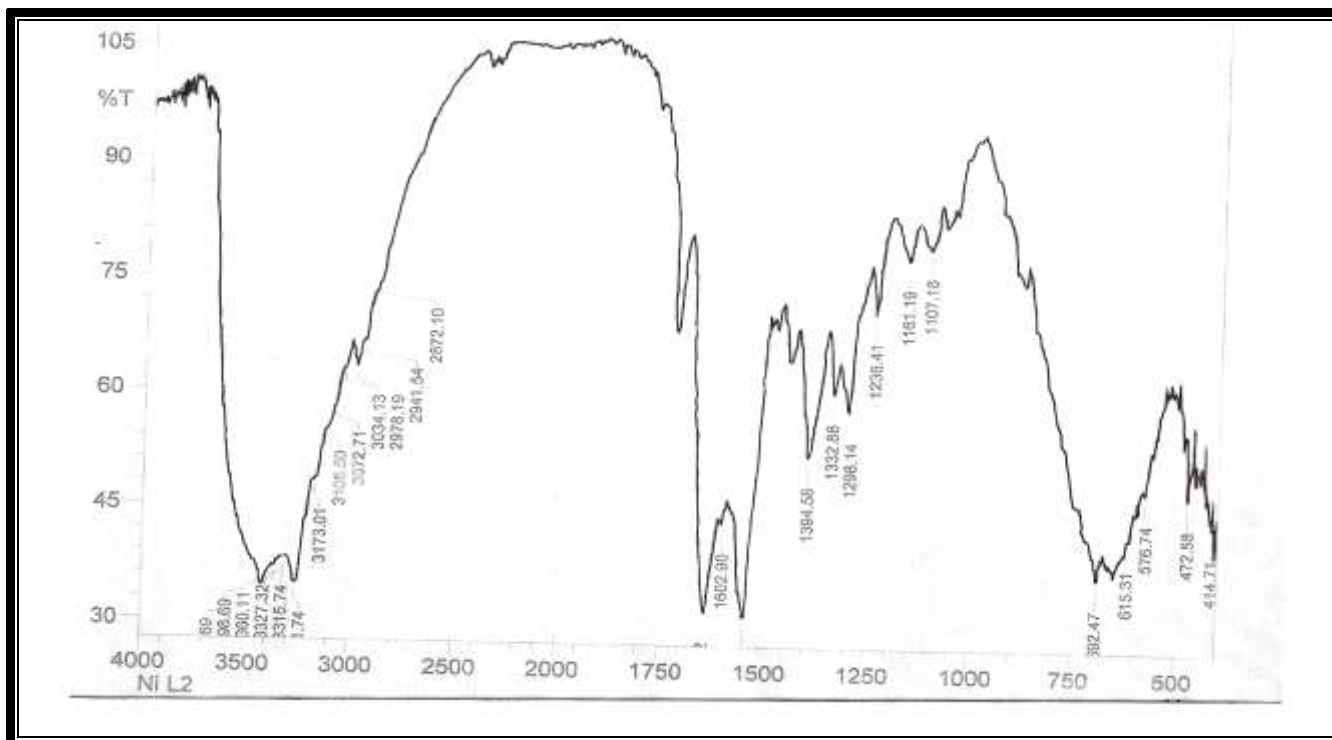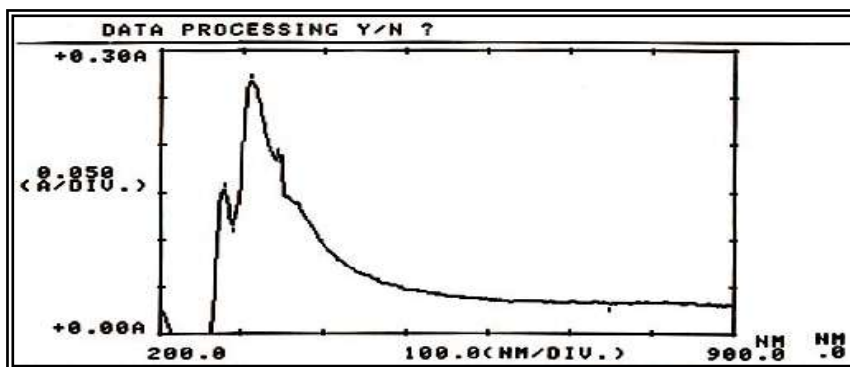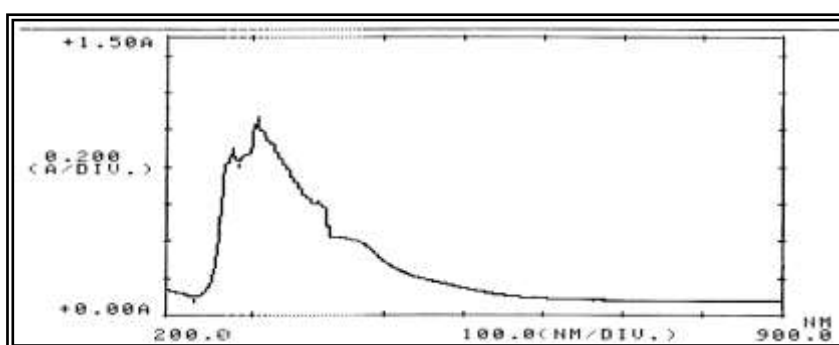Fig-2:- IR spectrum of *N, N`-Bis*-(2-amino- ethyl) Phthalamide (I)

Fig-3:- IR spectrum of the ligand [H_2L]

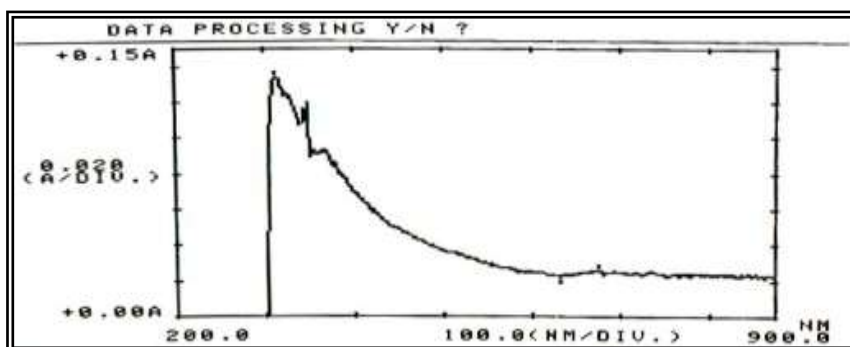Fig-4:- IR spectrum of the **C₂ complex**
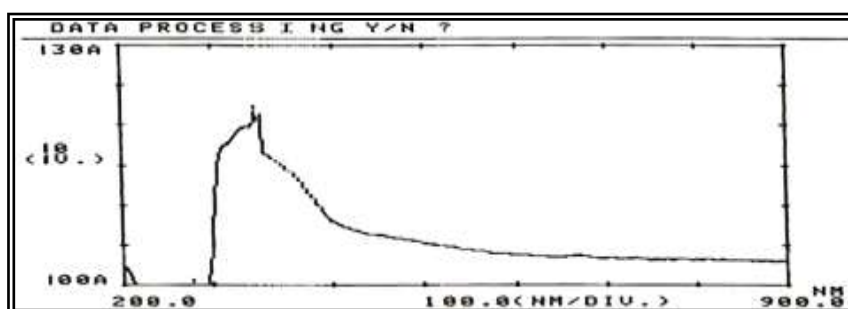

Fig-5:- IR spectrum of the **C₃ complex**

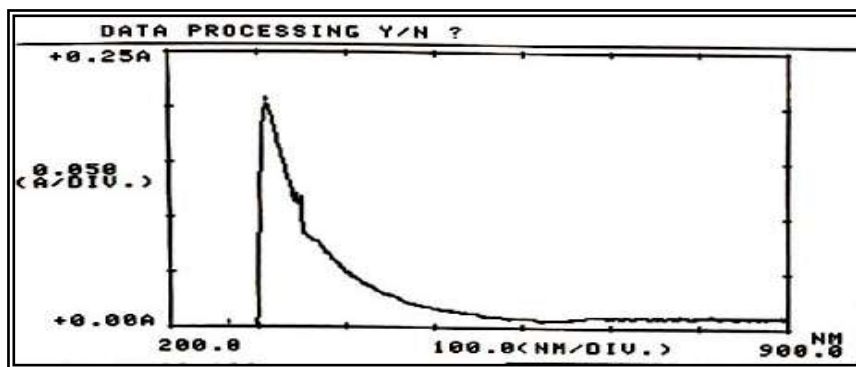**Fig.–6: Electronic spectrum of H$_2$L ligand**
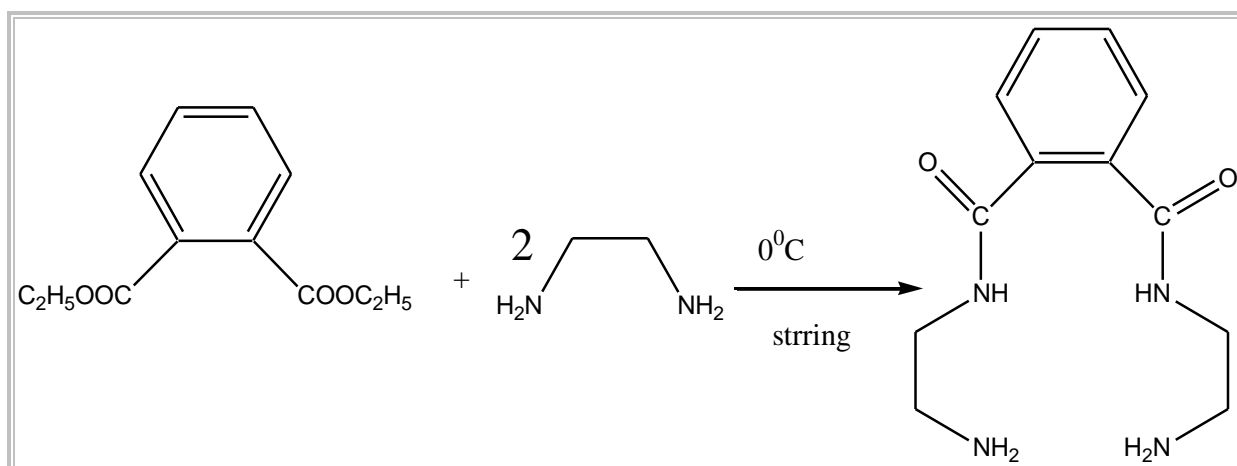
Fig. 7: Electronic spectrum of C$_1$ complex
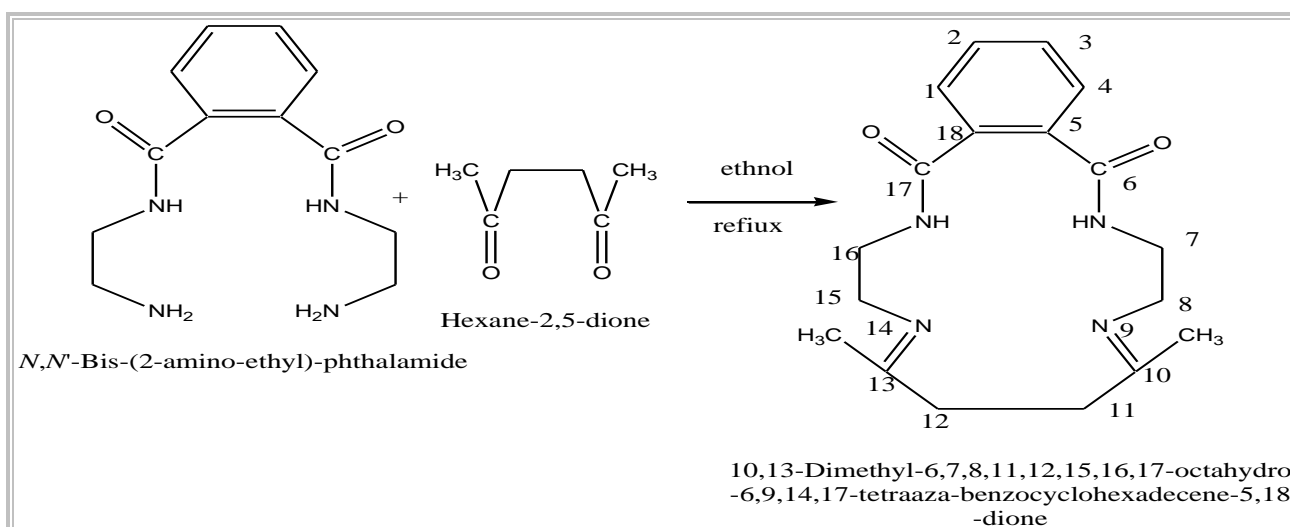
Fig. -8:- Electronic spectrum of C$_2$ complex

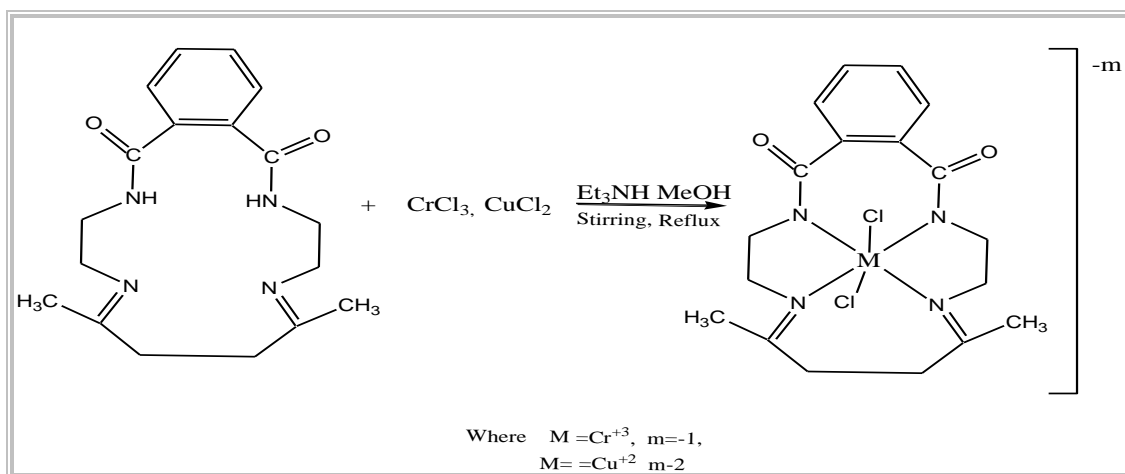**Fig.-9:- Electronic spectrum of distorted tetrahedral C$_5$ complex**

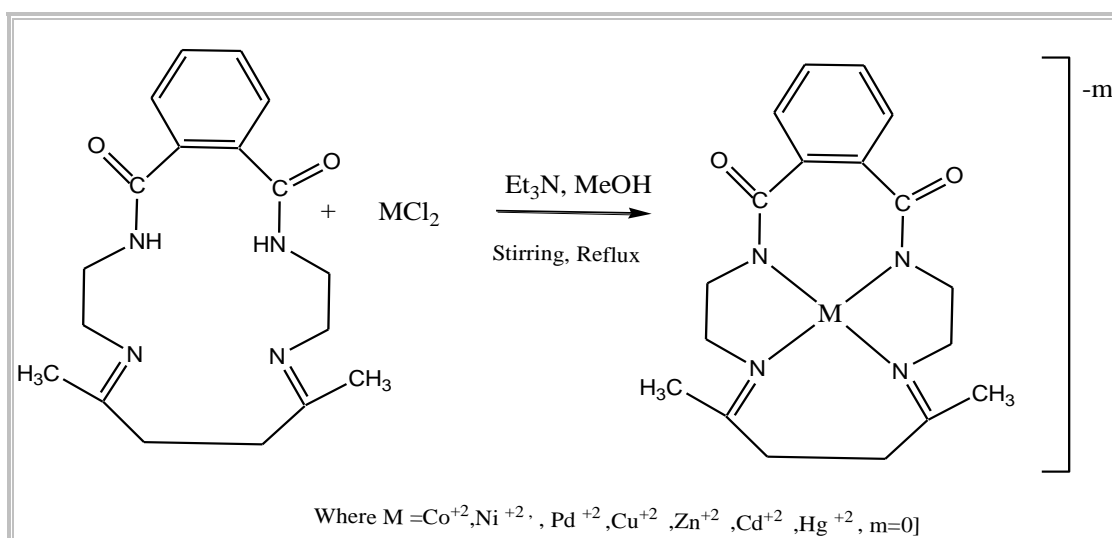**Fig. -10:- Electronic spectrum of C₈ complex**

Scheme -1: Preparation route of precursor (I).

10,13-Dimethyl-6,7,8,11,12,15,16,17-octahydro
-6,9,14,17-tetraaza-benzocyclohexadecene-5,18
-dione

Scheme (2): Preparation of ligand [H₂L]

Scheme (3): Preparation of [C₁.C₅] complexes

Scheme (3): Preparation of [C₂, C3, C4, C6, C₇, C8] complexes