Synthesis and Characterization of Some Mixed-Ligand Complexes Containing Azo Dye and 1,10-phenanthroline with Co^{II}, Zn^{II}, Cd^{II} and Hg^{II} Ions

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Abstract

In this work, the ligand was obtained from the reaction of diazonium salt of naphthyl amine with 1-amino-2-naphtol-4-sulfonic acid. The bidentate ligand type (NO) donar atoms was reacted with 1,10-phenanthroline and matel salt in a 1:1:1 mole ratio to give the complexes, using NaOH as a base. Physical-chemical teqnichas were used to characteriz the prepared compounds FT-IR,U.V-Vis, fluorescence and ^{1}HNMR spectroscopy, atomic absorption , chloride content along with conductivity and melting point measurements .Finally, thermal analysis was used to confirm the presence of coordination H_2O molecule in the complexes structure. According to memtioned characterization methods, the general formula proposed for Co^{II} Zn^{II}, Cd^{II} and Hg^{II} complexes is $[M(L)(phen)(H_2O)Cl]$.

Keywords: Azo-dye , Ligand , Complex , 1,10-Phenanthroline

Introduction

Azo compounds are a very important class of chemical compounds that received attention in scientific research. They are highly colored and have been used as dyes and pigments for a long time [1,2]. Furthermore, they have been studied widely because of their excellent thermal and optical properties in applications such as optical recording medium [3-6], link-jet printing[7,8] and oil- solution light fast dyes [9]. 1,10- phenanthroline has a rigid framework and possesses a super, ability to chelate many metale ions via two nitrogen donors, which shows potential for technological applications , due to their high charge transfer mobility , bright light-emission and good electro- and photo -active properties [10-15]. Azo metal chelates have also attracted increasing attention due to their interesting electronic and geometrical features in connection with their application for molecular memory storage , nonlinear optical elements and printing systems [4],[6]and[16].The presence of aromatic and /or heteroaromatic groups in the structure of nitrogen donors gives these ligands additional properties, for instance, poly nitrogen donors containing aromatic and/or heteroaromatic groups conjugate the ligational ability with the photophysical properties typical of these groups and accordingly, they have been widely used as chemosensors for metal ions in solution, since their coordination may affect the properties of the photosensitive group giving rise to an optical response [17-19]. Recently Man Singh and Sushma Anant prepared a new azo dye, N,Ndimethylazoleucine, composed of leucine ($NH_2CH\text{-}(CH_2CH(C_2H_6))COOH$) as basic moiety and 1,2-dimethyl aniline ($C_6H_5N(CH_3)_2$) and its Pr(III) complex with 1,10 phenanthroline (heterocyclic compound) adduct to assess the possibilities of Pr(III) complexes formation and spectral changes that occur due to complexation [20].In this paper, Preparation of new complexes of mixed ligand containing(2-(2-(naphthalen-5-yl)diazenyl)-4-amino-3-hydroxynaphthalene-1-sulfonic acid) and 1,10-phenanthroline were reported, the fluorescence properties of the prepared compound were studied.

Experimental

An electro thermal apparatus stuart melting point was used to measure the melting points. Infrared spectra were performed using FI-IR testscan shimadzu (FT-IR)- 8300 series spectrophotometer in the range (4000– 400 cm^{-1}); Spectra were recorded as potassium bromide discs at College of Education for pure science Ibn- Al- Haitham/ Baghdad University. The electronic spectra of the compounds were obtained using (U.V–Vis) spectrophotometer UV-1800, in the range (1100– 200 nm) using quartz cell of (1.0) cm length with concentration (10^{-3}) mole L^{-1} of samples in DMSO at 25°C and Fluorescense spectrophotometer type agilent, the measurement conducted was in central labrotary of College of Education for pure science Ibn- Al- Haitham Bagdad University .Atomic absorption and chloride content were determined by potentiometric titration method on (686-titro proceccor-665 dosinat metrome Swiss) in Ibn Sina Company Ministary of Industary Baghdad Iraq . Electrical conductivity measurements of the complexes were recorded at (25°C) for (10^{-3}–10^{-5}) M solutions of the samples in DMSO using Eutech 150 conductivity meter. 1HNMR, Spectra for the ligand and CdII complex were recorded in DMSO-d6 using Brucker, model: ultra sheild 400 MHz, origin: Switzerland and are reported in ppm (s), at Kashan University Iran, TG and DSC, thermograph were obtained using apparatus type STA PT-1000 LINSEIS as the temperature range of 30-600C0, the measurement obtained in central laboratory, College of Education for pure science Ibn- Al- Haitham Bagdad university.

Synthesis of the ligand (HL)

This compound was prepared as described in the literature [21,22]. A mixture of 1-naphthyl amine (1g, 6.98 mmol) in water (10 mL) and concentrated hydrochloric acid (2.62 mL, 30.00 mmol) was stirred until a clear solution was obtained.The mixture was cooled to 0– 5°C and a solution of sodium nitrite (0.76g, 11.00 mmol) in water (5 mL) was then added dropwise, maintaining the temperature below 5°C. The resulting mixture was stirred for an additional 1h in an ice bath and then a little urea was added and was buffered (pH 6–7) with solid sodium acetate (solution 1). 1-Amino-2-naphtol-4-sulfonic acid (1.67g, 6.98 mmol) was dissolved in 10 mL aqueous NaOH (10 mmol) solution, cooled to 0 - 5°C in an ice bath (solution 2).The solution was then gradually added to the cold solution 1, and the resulting mixture was continually stirred at 0- 5°C for 2 hrs. The resulting crude precipitate was filtered by acidification and washed several times with cold water.Yield 72%,m.p(290°C dec.).

Synthesis of the complex [Co(L)(phen) (H_2O)Cl]

To a solution of (0.5 g,1.27 mmol) of the ligand dissolved in 15 mL of methanol,it was added (0.051 g, 1.27 mmol) of sodium hydroxide dissolved in 10 mL methanol and (0.25 gm, 1.27 mmol) of 1,10-phenanthroline in 10 mLwith stirring and heating, of cobalt (II) chloride (0.30 g, 1.26 mmol) hexahydrate dissolved in 10 mL methanol was added to the a bove solution .The resulting mixture was refluxed for 2 hrs., the product was filtered off, washed with absolute methanol and recrystallized from methanol.Yield 77%,m.p(over 320 °C dec).

Synthesis of the complexes [Zn(L)(phen)(H_2O)Cl], [Cd(L)(phen) (H_2O) Cl] and [Hg(L) (phen) (H_2O) Cl]

A similar produce described to that mentioned in preparation of CoII complex was used to prepare the complexes of [HL] with [ZnII ,CdII and HgII],ions . Table No.(1) shows some physical properties of the prepared [HL], and its complexes and their reactants quantities.

Results and Discussion

Mixed potentially bidentate ligand type NO donor atoms and 1,10-phenanthroline with metal salt were used in a 1:1:1 molar ratio to synthesiz complexes using methanol as a solvent as shown in Schem No.(2). The ligand contains labile proton [HL] and by removing this proton an ionic(-1) bidentate system is formed. The ligand [HL] was synthesized by the reaction of diazonium salt with 1-amino-2-naphtol-4-sulfonic acid in a 1:1 molar ratio using methanol as a solvent as shown in Schem No.(1).

I.R. spectrum for ligand [HL], Figure No.(1), displayed a band at (3234.62) cm^{-1} may be attributed to the interference of the v(N.H)asym and v(N.H) sym bands of (NH_2) group [23] .The band at (3095.75)cm^{-1} may be referred to aromatic (C-H) [24]. A broad band at (2912.51)cm^{-1} may refer to the phenolic OH group [25-27]. This band was ascribed of intramolecular hydrogen bonding (N…H-O) and (NH…O-H) groups [28] .The spectrum showed a weak band at (1656.85)cm^{-1} referred to the bending of (N-H) group. The medium bands at (1598.99-1527.62)cm^{-1} assigned to (C=C) stretching of aromatic rings [24] . While band at (1467.83)cm^{-1} can be attributed to (N=N) azo group [29] . The spectrum showed band at (1352.10)cm^{-1} attributed to phenolic (C-O) stretching [23] . Finally the spectrum showed two bands at (1166.93 and 1043.49)cm^{-1}can be attributed to the v(SO3H)asym ,v(SO_3H)sym stretching respectively [30] . The spectrum of the 1,10-phenanthroline. Figure No.(2) shows the band at (3363.86)cm^{-1} attributed to OH group of the adsorbed water molecules ($C_{12}H_8N_2.H_2O$) and bands at (3062.96)cm^{-1} , (1645.28)cm^{-1} and (1585.49-1525.70)cm^{-1} may be assigned to v(C-H)aro. , v(C=N)phen. and v(C=C)aro groups respectively , while the bands at (1344.38-1217.08)cm^{-1} may be assigned to the v(C-N) stretching, the assignment of characteristic bands are summarized in table No.(2). The I.R. spectra for complexes of CoII, ZnII, CdII and HgII are shown in figures No.(3),(4),(5) and (6) respectively . The bands arising due to vibrational v(C=N)phen. mode at (1645.28)cm-1 frequency in the free 1,10-phenanthroline were observed to be shifted to lower frequencies , and appears at (1620.21)cm^{-1},(1624.06)cm-1, (1618)cm^{-1} and (1614.42)cm-1 for complexes of CoII, ZnII, CdII and HgII respectively .The movement of v(N=N) stretch of the complexes to relatively lower frequencies ,at (1425.04), (1423.47), (1421) and (1450.47)cm^{-1} for complexes CoII, ZnII, CdII and HgII respectively , compared to that of free dyes or ligand indicates coordination via the N=N group [31-33]. On the other hand the band at (1352.10)cm-1 referred to v(C-O) of free ligand , shifted to lower frequency and appeared at (1280.37), (1296.16), (1230) and (1257.59)cm^{-1} for complexes of CoII, ZnII, CdII and HgII respectively.The see result indicaties the involvement of nitrogen of pyridime ring oxygen and nitrogen the azo ligand in the complex formation[34-36]. The spectra of complexes displayed a band at (3215.34),(3307.92) and (3273.20)cm^{-1} for complexes of Co, Zn, Cd and Hg respectively can be attributed to v(NH2) group[24]. While the band at (3431)cm-1in Cd complex spectrum may assign to interference of v(NH2) stretch with the OH group of the coordinated water molecule [23].The spectrum of Co complex displayed a band at (3379.29) cm^{-1} may indicates OH group of the coordinated water molecules[23].Finally the spectra showed new bands at [(653.87 , 657.73 , 675 and 665.44)] ; [(601.79 ,634.58 , 621 and 663.15)] and [(551.64 , 611.43 , 523 and 561.29)] cm^{-1} can be refer to v(M-N)azo. , v(M-N)phen. and v(M-O) for complexes Co, Zn, Cd and Hg respectively. The new bands also supported the coordination of the ligand to the metal center through azo group nitrogen , phen. group nitrogens and phenolic oxygen atoms. These results are supported by several reports [37,38]. The characteristic bands are summarized in table No.(2) .

The U.V-vis spectrum for [HL], Figure No.(7) , shows peak at (253nm) (39525 cm-1) (εmax= 3843molar^{-1} cm^{-1}) assigned to π–π* transition of the aromatic ring [23]. Peak at (333nm) (30030 cm-1) (εmax= 2750molar^{-1} cm^{-1}) assigned n–π* transition. Finally peak at(437nm)(22883 cm-1)(εmax= 1063molar-1cm^{-1}in ligand spectrum may be attributed to the azo group (N=N) [27]. U.V-Vis spectrum for 1,10-phenanthroline , Figure No.(8) , shows peak of shortest wave length presenting at (257nm) (38910 cm^{-1}) (εmax= 1352molar^{-1} cm^{-1}) may be assigned to over lape of n–π* and π–π* transition ,[39-41] .The absorption data of the ligand and 1,10-phenanthroline are given in table No.(3) Figures No.(9),(10), (11) and (12) show the (U.V-Vis) spectra of the complexes Co, Zn, Cd and Hg respectively. Table No.(3) summarized the absorption peaks of the complexes, in each case the spectrum showed intense peaks in the (U.V) region at (260nm) (38461 cm^{-1}) (εmax= 3933molar^{-1} cm^{-1}); (298nm) (33557 cm^{-1}) (εmax= 3887molar^{-1} cm^{-1}); (244nm) (40983 cm^{-1}) (εmax= 1756molar^{-1} cm^{-1}) and (228nm) (43859 cm^{-1}) (εmax= 735molar^{-1} cm^{-1}) for complexes of Co, Zn, Cd and Hg respectively , which can be assigned to π–π* transition [23]. The peaks at (260nm) (38461 cm^{-1}) (εmax= 3933molar^{-1} cm^{-1}); (300nm) (33557 cm^{-1}) (εmax= 3887molar^{-1} cm^{-1}); (293nm) (34129 cm^{-1}) (εmax= 3952molar^{-1} cm^{-1}) and (302nm) (43859 cm^{-1}) (εmax= 735molar^{-1} cm^{-1}) for complexes of Co, Zn, Cd and Hg respectively , assigned the n–π* transition .The bands at (339nm) (29498 cm^{-1}) (εmax= 4000molar^{-1} cm^{-1}); (345nm)(28985cm^{-1}) (εmax= 3730molar^{-1} cm^{-1}) ; (310nm) (32258 cm^{-1}) (εmax= 1833molar^{-1} cm^{-1}) and (425nm) (23529 cm^{-1}) (εmax= 755molar^{-1} cm^{-1}) for complexes of Co, Zn, Cd and Hg respectively , may assigned to M→L charge transfer transition [23].The N=N band of the free ligand at (437nm) (22883 cm^{-1}) (εmax= 1063molar^{-1} cm^{-1}) shifted to lower wave lengthes in the complexes and appeared at (403nm) (24813 cm^{-1}) (εmax= 3032molar^{-1} cm^{-1}); (421nm) (23752 cm^{-1}) (εmax= 2037molar-1 cm^{-1}); (411nm) (24330 cm^{-1}) (εmax= 951molar^{-1} cm^{-1}) and (441nm) (22675 cm^{-1}) (εmax= 755molar^{-1} cm^{-1}) for complexes of Co, Zn, Cd and Hg respectively as a consequence of coordination when binding with a metal , confirming that the azo nitrogen was coordinat to the metal atom. The peak in the visible region can be associated with d-d transition . The Co^{II} complex shows a band at (620nm) (16129 cm^{-1}) (εmax= 1788molar^{-1} cm^{-1}) assignable to $^4T_{1}g_{(F)}\rightarrow{}^4A_{2}g_{(F)}$ suggesting distorted octahedral geometry around Co^{II} ion . Finally Zn^{II}, Hg^{II} and Cd^{II} complexes with an electronic configuration of d^{10} did not show any (d-d) transitions. Instead the absorption bands in the spectra were due to charge transfer transitions which suffered from blue shift with hyper chromic effect [42].

The ^{1}HNMR spectra of DMSO-d^6 solution of ligand and it Cd^{II} complex showed well resolved signals Figure(13) and Figure(14), table(4) .The spectrum of the free ligand showed multiplet chemical shift at the range (δ =7.3-7.8 ppm) assigned to aromatic protons. The characteristic signals at(δ = 8.8 ppm) and (δ =11.1 ppm) were assigned to NH_2 and OH

phenolic groups respectively , because of intramolecular hydrogen bonding with the nitrogen atom on the phenolic ring [23].
The ^{1}HNMR spectrum of Cd^{II} complex showed the aromatic protons at chemical shifts at the range(δ = 7.2-8.2 ppm), signal observed at (δ =8.8 ppm) assigned to NH_2 group and (δ =9.2 ppm) assigned to NH proton of azo group, this information supports the fact that there is keto hydrazone tautomer for the synthesized ligand [43]. The chemical shifts at(δ = 11.1 ppm) which can be attributed to the proton of the phenolic group in the ligand spectrum , disappeared in the complex spectrum due to the deprotonation process.

Fluorescence Emission Spectra

The emission spectra of the ligand and 1,10-phenonthroline are shown in Figure (15) and Figure(16), respectively complexes spectra are shown in Figures (17-20).The fluorescence emission spectra of ligand display maximum emission wavelengths ($\lambda_{em,max}$) at 455 nm with excitation wavelengths at 437 nm , while 1,10-phenonthroline shows maximum emission wavelengths ($\lambda_{em,max}$) at 348 nm with excitation wavelengths at 257 nm the complexes (Co^{II}, Zn^{II}, Cd^{II} and Hg^{II}) exhibit maximum fluorescence emission wavelengths ($\lambda_{em,max}$) at 340,330,340 and 324 nm with excitation wavelengths at 339,300,310 and 302 nm respectively . The Co^{II} and Cd^{II} show very good fluorescence emission behavior upon excitation of the intraligand charge transfer band [44]. The shifts in complexes data compared with ligand indicate that the ligand successfully chelated to metal ions. Fluorescence spectral data are shown in table (5).
Molar conductivity measurement for the complexes in (DMSO) are summarized in table (6).

Thermal Analysis Studies

The TGA thermal analysis curves for $[Co(L)(phen)(H_2O)Cl]$and $[Cd(L))(phen)(H_2O)Cl]$ complexes is shown in figures (21) and (22),and data are listed in table(7). The Co^{II} complex decomposes in three steps. The first stage is the loss ($H_2O + N_2 + NH_3$) with mass losses of 9.25897% (calc. = 8.918766978%) within a temperature range of 30-248.93^{0}C, the decomposition of the complex in the 30-248.93^{0}C range is indicated by endothermic processes at 84.5 ^{0}C the second step involves the loss of the organic fraction , ($C_{10}H_8$+NaCl) within the temperature range of 248.93-446.80 ^{0}C, the decomposition of the complex in the 248.93-446.80^{0}C range is indicated by exothermic processes at 311.6^{0}C, with mass losses of 26.2889% (calc. =26.40114529%). The final weight of the compound observed at 57.1701% (calc. =64.68001614%) within a temperature range of 446.80-572.34^{0}C. The difference in the calculations in observed of the residue weight may be related to the sublimation upon thermal decomposition .The Cd^{II} complex decomposes in two steps. Stage one is the loss (1,10-phenanthroline+$C_{10}H_8+H_2O+N_2+NH_3$+Cl+SO_3Na) within the temperature range of 30-402.12^{0}C, the decomposition of the complex in the 30-402.12^{0}C range is indicated by endothermic and exothermic respectively processes at 130.8C^0 and 363.2^{0}C , within mass losses of 68.4125%(calc. =69.44892265%). The final stage is the loss (MO) within the temperature range of 402.12-593.61^{0}C within mass losses of 16.0405% (calc. =16.8993%), the difference in the calculations in observed of the residue weight may be related to the sublimation upon thermal decomposition[44].

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Table (1): Some physical properties of [HL], complexes and their reactant quantity

Empirical formula	M.wt	Wt of metal salt (g)	Wt of product (g)	Yield %	Color	Dec.C^0	Found,(Calc)% M	
							M	Cl
[HL]	393.42	-	-	72	Dark red	290	-	-
[Co(L)(phen) (H$_2$O)Cl]	706.37	0.302	0.7	77	Dark prown	>320	(8.34) 8.98	(5.01) 5.10
[Zn(L)(phen) (H$_2$O)Cl]	712.83	0.104	0.5	68	Light green	>320	(9.17) 9.22	(4.97) 4.98
[Cd(L)(phen)(H$_2$O)Cl]	759.85	0.056	0.8	69	Light orange	>320	(14.79) 14.85	(4.66) 4.75
[Hg(L)(phen)(H$_2$O)Cl]	848.03	0.140	0.4	61	Light prown	>320	(23.65) 23.71	(4.18) 4.25

Dec.= Decomposition

Table (2):FT-IR spectra data (wave number v^{-1})cm^{-1} of the ligand and its metal complexes

Compound	V(NH$_2$)	V(C-H)$_{aro.}$	V(OH) broad	V(C=C)$_{aro.}$	V(N=N) V(C=N)$_{phen.}$	V(C-O)	V(M-N)$_{azo.}$ V(M-N)$_{pheen.}$	V(M-O)	Additional peaks
HL	3234.62	3095.75	2912.51	1598.99-1527.62	1467.83	1352.10	- -	-	V(N-H)bending 1656.85 V(C-N)1282.66-1215.15 V(SO$_3$H)$_{asy}$ 1166.93 V(SO$_3$H)$_{sym}$ 1043.49
1,10-phenanthroline	-	3062.96	3363.86	1585.94-1552.70	- 1645.28	-	- -	-	V(C-N) 1344.38-1217.08
[Co(L)(phen)(H$_2$O)Cl]	3215.34	3061.03	3379.29	1581.63-1517.98	1425.04 1620.21	1280.73	653.87 601.79	551.64	V(C-N) 1317.38-1367.53
[Zn(L)(phen)(H$_2$O)Cl]	3307.92	3016.67	-	1564.27-1516.05	1423.47 1624.06	1296.83	657.73 634.58	611.43	V(C-N) 1388.75-1352.10
[Cd(L)(phen)(H$_2$O)Cl]	3431	3053	3431	1581-1520	1421 1618	1230	675 621	523	V(C-N) 1350-1387
[Hg(L)(phen)(H$_2$O)Cl]	3273.20	3134.33	-	1541.12-1510.26	1450.47 1614.42	1257.59	665.44 663.51	561.29	V(C-N) 1396.46-1369.46

Table (3): Electronic spectral data of ligand and metal complexes

Compound	Wave number		ε_{max} molar^{-1} cm^{-1}	Assignment	Suggested structure
	Nm	Cm^{-1}			
HL	253	39525	3843	$\pi-\pi^*$	Octahedral
	333	30030	2750	$n-\pi^*$	
	437	22883	1063	N=N	
1,10-phenanthroline	257	38910	1352	$\pi-\pi^*$, $n-\pi^*$	
[Co(L)(phen)(H_2O)Cl]	260	38461	3933	$\pi-\pi^*$	
	278	35971	3866	$n-\pi^*$	
	339	29498	4000	C.T	
	403	24813	3032	N=N	
	620	16129	1788	$^4T_1g_{(F)}\rightarrow{}^4A_2g_{(F)}$	
[Zn(L)(phen)(H_2O)Cl]	298	33557	3887	$\pi-\pi^*$	Octahedral
	300	33333	3966	$n-\pi^*$	
	345	28985	3730	C.T	
	421	23752	2037	N=N	
[Cd(L)(phen)(H_2O)Cl]	244	40983	1756	$\pi-\pi^*$	
	293	34129	3952	$n-\pi^*$	
	310	32258	1833	C.T	
	411	24330	951	N=N	
[Hg(L)(phen)(H_2O)Cl]	228	43859	735	$\pi-\pi^*$	Octahedral
	302	33112	3605	$n-\pi^*$	
	425	23529	755	C.T	
	441	22675	755	N=N	

Table (4): ^{1}HNMR data for ligand and [Cd(L)(phen)(H_2O)Cl] in DMSO-d^6 and chemical shift in ppm(δ)

Compound	Funct. Group	δ(ppm)
HL	C-C aromatic	7.3-7.8
	NH_2	8.8
	O-H phenolic	11.1
[Cd(L)(phen)(H_2O)Cl]	C-C aromatic	7.2-8.2
	NH_2	8.8
	O-H phenolic	9.2

Table (5): Fluorescence spectra for ligand and complexes

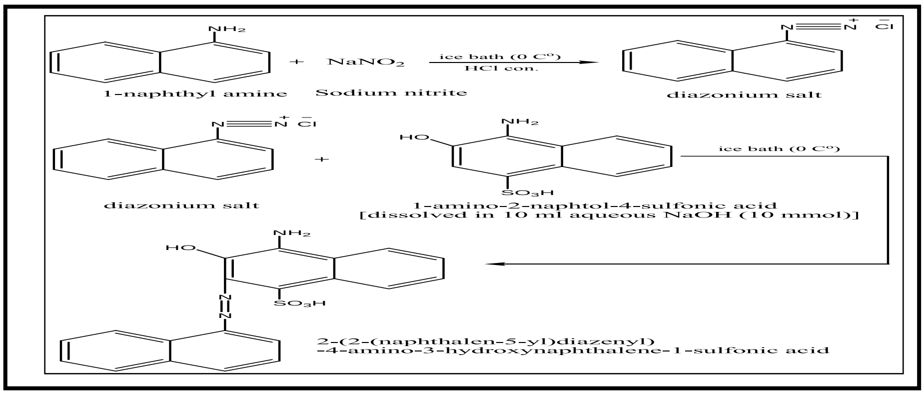
Compound	λ_{max} (em)	λ_{max} (ex)
HL	455	437
1,10-phenanthroline	348	257
[Co(L)(phen)(H_2O)Cl]	340	339
[Zn(L)(phen)(H_2O)Cl]	330	300
[[Cd(L)(phen)(H_2O)Cl]	340	310
[Hg(L)(phen)(H_2O)Cl]	324	302

Table (6): The molarconductivity of the complexes

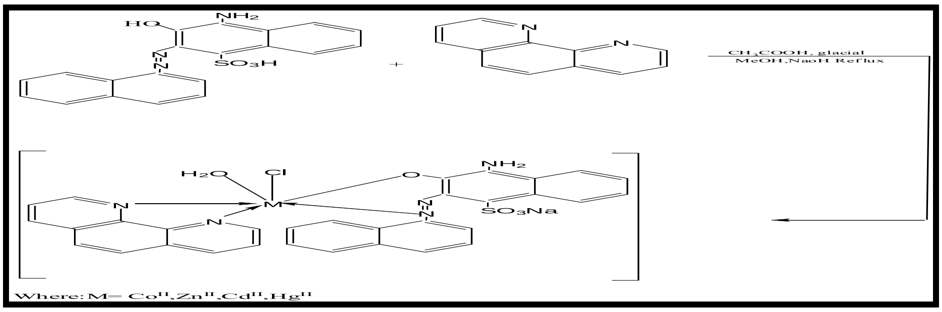
Compound	Λm S.cm^2 molar^{-1}
[Co(L)(phen)(H_2O)Cl]	15.2
[Zn(L)(phen)(H_2O)Cl]	10.32
[Cd(L)(phen)(H_2O)Cl]	4.14
[Hg(L)(phen)(H_2O)Cl]	14.57

Table (7): Thermoanalytical results (TG and DSC) of [Co(L)(phen)(H_2O)Cl] and [Cd(L)(phen)(H_2O)Cl]

Compounds	Mass loss temp./ºC	Mass loss Theoretically	Mass loss Practically	DSC
[Co(L)(phen)(H_2O)Cl]	30-248.93	8.918766978	9.25897	84.5(endo)
	248.93-446.80	26.40114529	26.2889	311.6
	446.80-572.34	64.68001614	57.1701	-
[Cd(L)(phen)(H_2O)Cl]	30-402.12	69.44892265	68.4125	130.8(endo) and 363.2 (exo)
	402.12-593.61	168993	16.0405	-



Scheme (1): Synthesis of the ligand (HL)



Scheme (2): Synthesis of the complexe

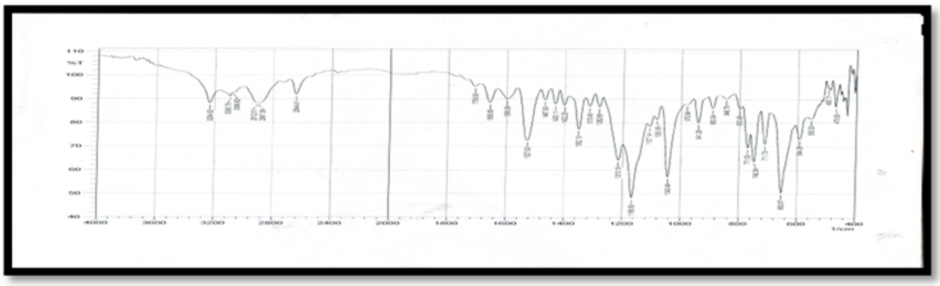


Fig (1): IR spectrum of the ligand(HL)

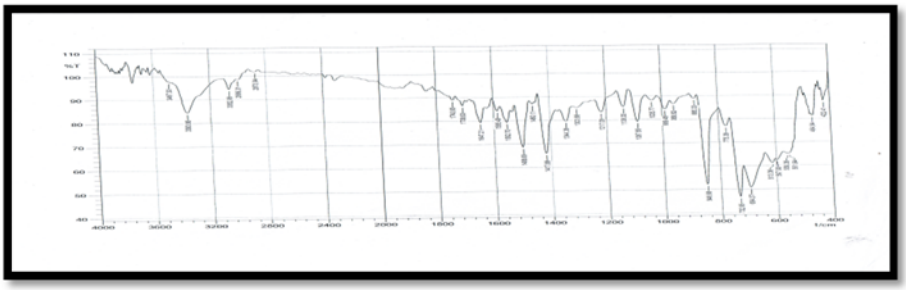


Fig (2): IR spectrum of the 1,10-phenanthrolin

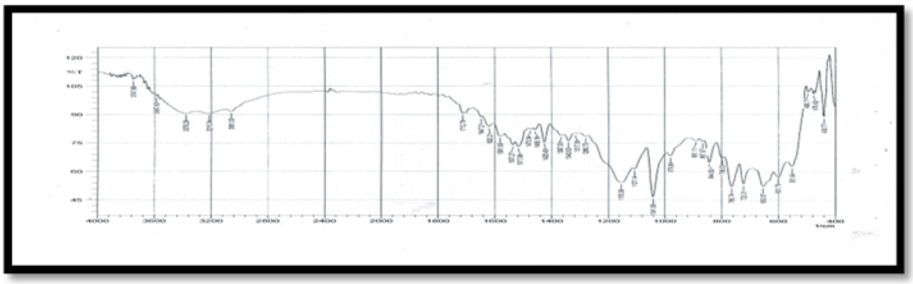


Fig (3):IR spectrum of the [Co(L)(phen)(H_2O)Cl]

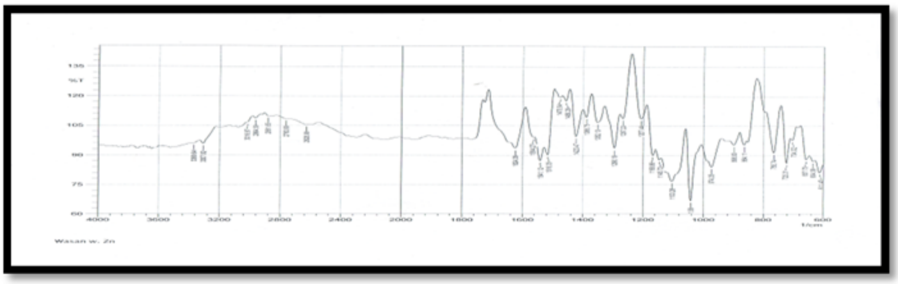


Fig (4): IR spectrum of the[Zn(L)(phen)(H_2O)Cl]

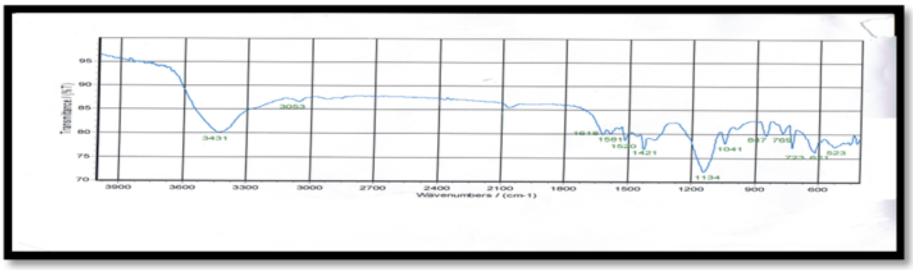


Fig (5): IR spectrum of the[Cd(L)(phen)(H_2O)Cl]

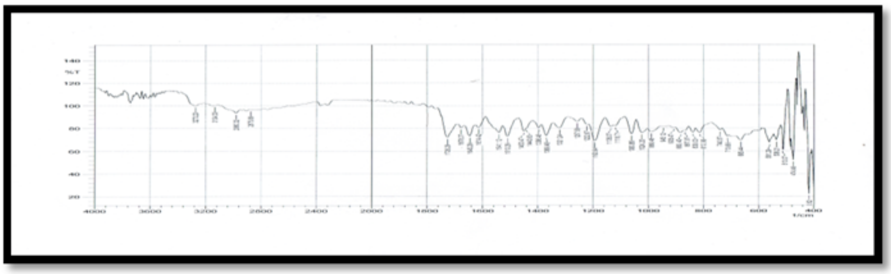


Fig (6): IR spectrum of the[Hg(L)(phen)(H_2O)Cl]

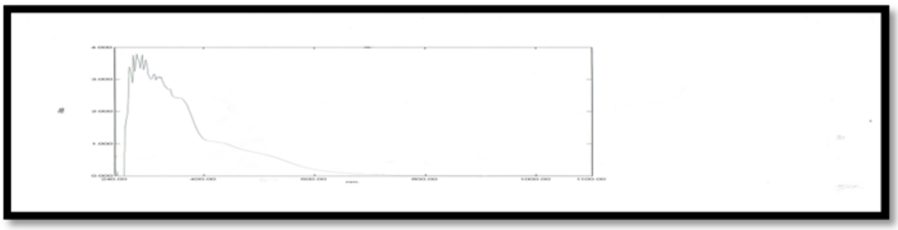


Fig (7): UV spectrum of the ligand

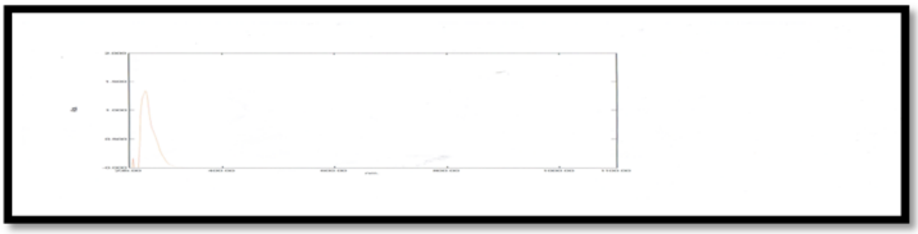


Fig (8): UV spectrum of the 1,10-phenanthrolin

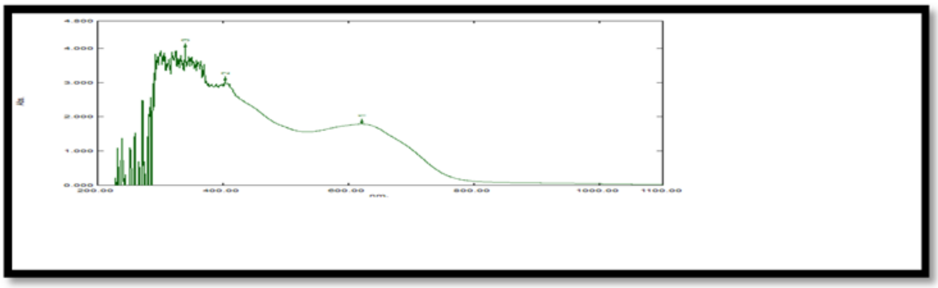


Fig (9): UV spectrum of the complex [Co(L)(phen)(H_2O)Cl]

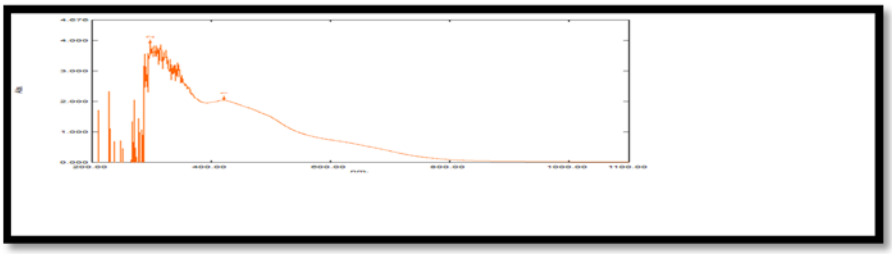


Fig (10):UV spectrum of the complex [Zn(L)(phen)(H_2O)Cl]

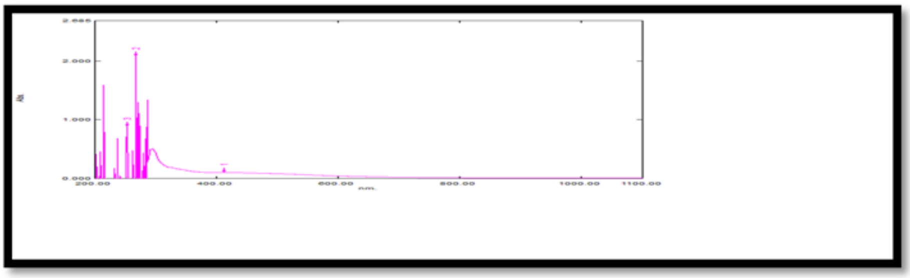


Fig (11): UV spectrum of the complex [Cd(L)(phen)(H_2O)Cl]

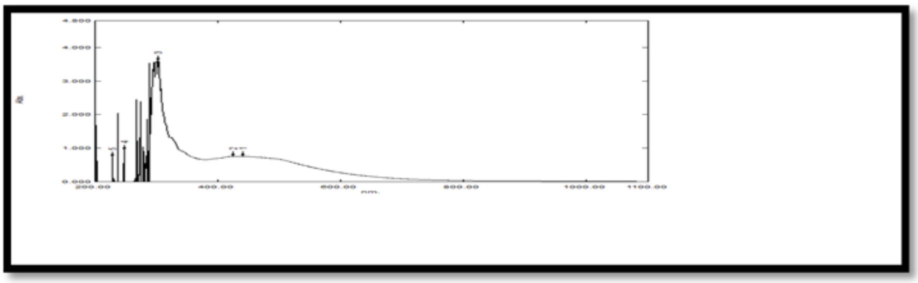


Fig (12): UV spectrum of the complex [Hg(L)(phen)(H_2O)Cl]

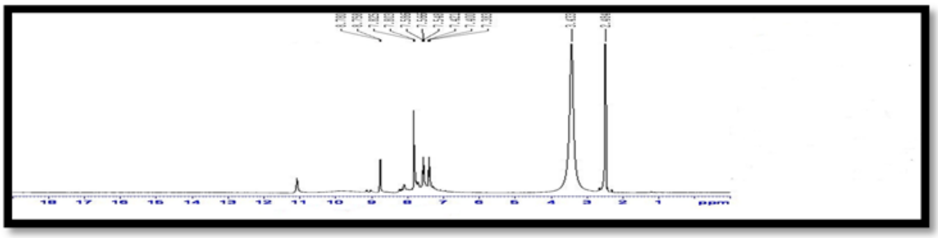


Fig (13):^{1}HNMR spectra for the ligand

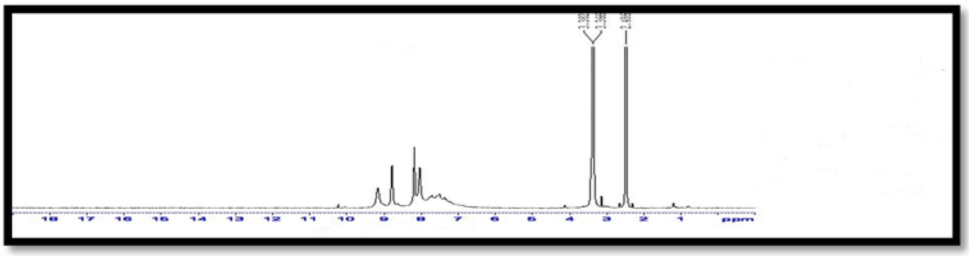


Fig (14):^{1}HNMR spectra for the complex [Cd(L)(phen)(H_2O)Cl]

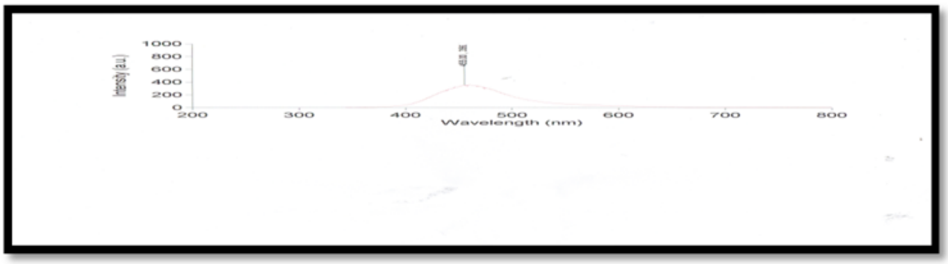


Fig (15):Emission spectra of the ligand

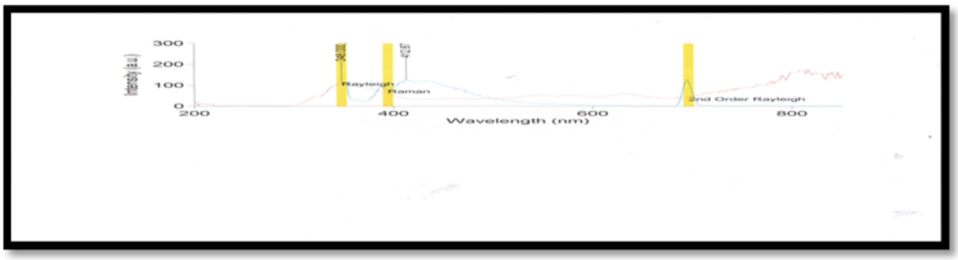


Fig (16):Emission spectra of the 1,10-phenanthroline

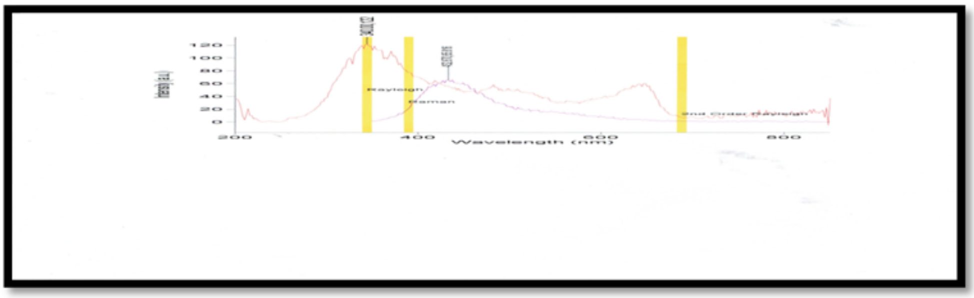


Fig (17):Emission spectra of the [Co(L)(phen)(H_2O)Cl]

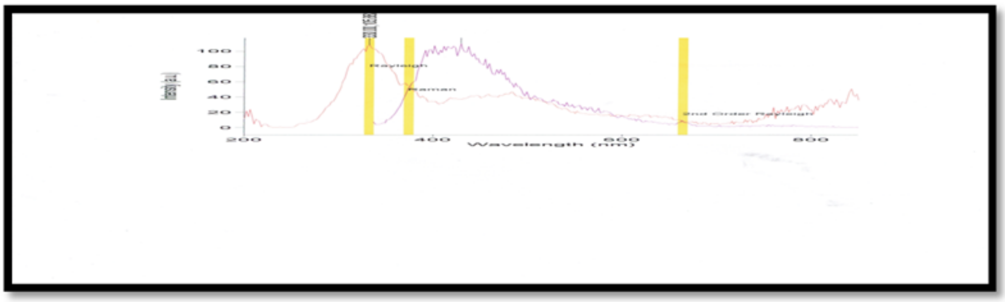


Fig (18): Emission spectra of the [Zn (L)(phen)(H_2O)Cl]

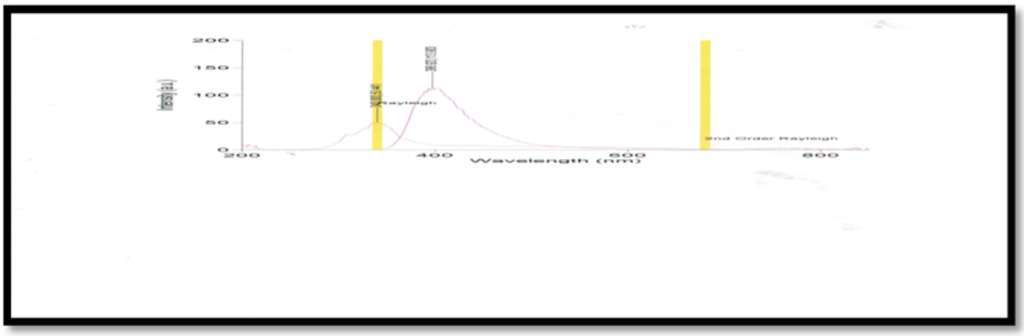


Fig (19): Emission spectra of the [Cd(L)(phen)(H_2O)Cl]

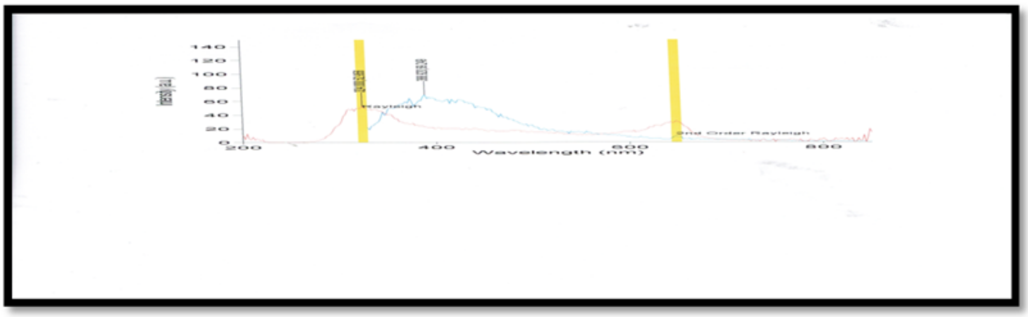


Fig (20): Emission spectra of the [Hg(L)(phen)(H_2O)Cl]

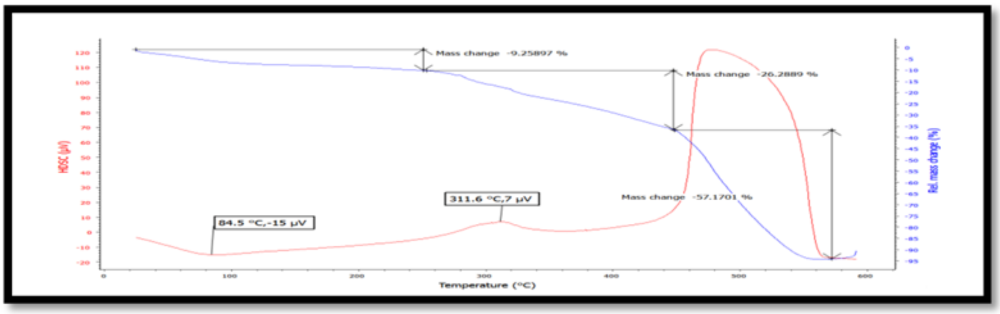


Fig (21): Thermograph of [Co(L)(phen)(H_2O)Cl]

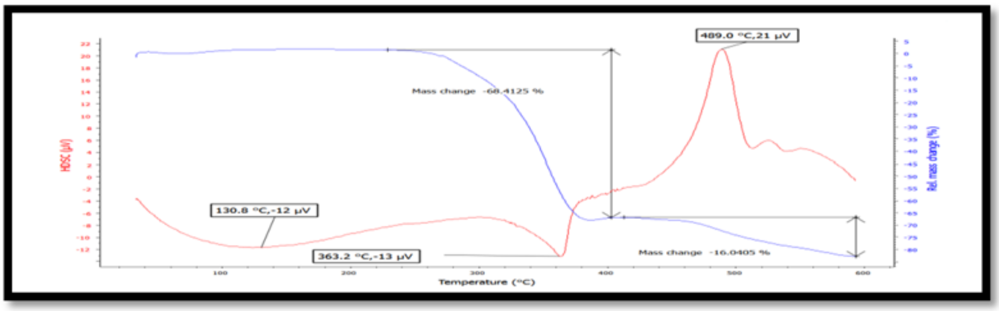


Fig (22): Thermograph of [Cd(L)(phen)(H_2O)Cl]

تحضير وتشخيص معقدات حاوية على ليكندات مختلطة من صبغة الازو و1,10-فينانثرولين مع ايونات الكوبلت، الكادميوم، الخارصين، الزئبق الثنائية التكافؤ

حسن احمد حسن
وسن محمد علوان
رياض محمود احمد
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قسم الكيمياء /كلية التربية للعلوم الصرفة (ابن الهيثم) /جامعة بغداد

استلم البحث في:10/حزيران/2015، قبل البحث في:19/تشرين الاول/2015

الخلاصة

تضمن العمل تحضيرمعقدات حاوية على ليكاندات مختلطة من صبغة الازو المحضرة من تفاعل ا-امينو-2-نفثول-4-سلفونيك اسيد مع ملح الدايزونيوم1-نفثايل امين كليكاند اولي ثنائي السن و 10,1-فينانثرولين كليكاند ثانوي مع املاح الفلزات وبنسبة (1:1:1) للحصول على المعقدات بنسبة 1:1:1 وباستعمال هايدروكسيد الصوديوم كوسط قاعدي. واستعملت التقنيات الكيميائية – الفيزيائية في تشخيص المركبات المحضرة بوساطة اطياف الاشعة تحت الحمراء وفوق البنفسجية-المرئية والفلورة وطيف الرنين النووي المغناطيسي والامتصاص الذري ومحتوى الكلور وقياسات التوصيلية الكهربائية مع درجات الانصهار.كما استعملت تقنية التحليل الحراري في اثبات وجود جزيئات الماء المتناسقة في تركيب المعقدات وبناءً على ما سبق اقترحت الصيغة الاتية لمعقدات الكوبلت الثنائي والكادميوم الثنائي والخارصين الثنائي والزئبق الثنائي.[M(L)(phen)(H_2O)Cl]

الكلمات المفتاحية: صبغة – الازو ، ليكاند ، معقد ، 1 ، 10- فينانثرولين