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Analytical and spectroscopic study of copper compounds with diamine donor new ligands

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Abstract--Naphthylenediamine dihydrochloride and 4-nitroaniline were utilized to make N-(1-Naphthylethylenediamine dihydrochloride) naphthalen-1-amine, an azo ligand, in this case. It was possible to synthesize four new Cu (II) complexes by using four different compounds, including the primary ligand azo, as well as the secondarily used sodium-pyriphosphate-oxalic- and malonic-acid-based secondary ligands. Spectroscopy (UV-Vis, FTIR, mass, ¹H-NMR spectra; atomic absorption; elemental analysis); magnetics; and molar conductivity were all used to thoroughly evaluate the produced substances. They found that all of the complexes were non-electrolytic and that all of the bivalent ligands were in coordination with copper (II) using infrared spectrum and molar conductivity data. Tetrahedral geometry of the complexes was discovered through the use of magnetic susceptibility measurements, mass spectrometric data, elemental analysis, H-NMR data, and ultraviolet visible spectra.

Keywords---Schiff base complex, copper complexes, Azo ligand dye.

1. Introduction

Since the discovery of the compound (CoCl₃.6NH₃) in 1789, researchers have been interested in coordination chemistry. Perhaps in the second half of the twentieth century, due to its rapid development, it gained a wide area in chemistry to prepare complex coordination compounds in a practical manner, and to know the structures of these complexes, these complexes have played an increasingly important and growing role in various fields, including medicine, industry and agriculture[1-2] . In the majority of complexes, the basic structure is an ion or molecule surrounded by another ion or molecule. It is possible to link to them via

covalent-coordination connections or coordination bonds known as ligands (Organic reagents). The total of the charges of the central metal atom and the ligands determines whether the complexes are positive, negative, or neutral. There are two types of ligands: the Lewis base, which is the donor atom or donor of the electron pair, and the metal ion, which is the acceptor or gainer of the electron pair because it contains empty orbitals that may receive the double electrons. If it just has one donor atom, it is known as a (Monodentate ligand), but if it has more than one, it is known as a (Polydentate ligand), Compounds containing Polydentate that are more likely to be bound to the metal than other polydentate are known as (Chelating complexes). While the ligands that can bind with more than one metal atom at the same time are called (Bridge ligands) and the stability of the complexes depends on the chelating ring formed as a result of coordination, where the pentagonal and hexagonal rings are the most stable [3-4]. Since the ligand has an effective color-carrying group (Chromophoric group), complexes inorganic have been widely utilized in analytical research because of their brilliant hues, which usually show in the visible range of the spectrum. A group (chromic Auxo) (OH, -Br, -Cl, -SH, -NH₂) can also be included in the ligand. Ligands and their complexes become more soluble in solvents as a result of its use. If you're looking to measure modest amounts of ion concentrations, these complexes and ligands are ideal. [5-6].

2. Experimental

2.1 Materials

The Chemicals are pure and used as delivered; CuCl₂.2H₂O (B.D.H), oxalic acid, malonic acid and tetra sodium pyrophosphate (Fluka), DMSO and ethanol (B.D.H).

2.2 Instruments

Melting points are determined using Stuart SMP20 melting points system. Infrared spectra were carried out using a Tetscan Shimadzu FT.IR—8300s. The electronic spectra have been recorded in DMSO using a double-beam Shimadzu UV-Visible Spectrophotometer. Proton nuclear magnetic spectrum ¹H-NMR Bruker, Ultrasheid (400.81)MHz. Electrical conductivity measurements were recorded at 25 °C using a Digital conductivity meter. Elemental analysis of elements was performed via Euro vector. EA 3000, single V.3.Osingle. Magnetic susceptibility measurements were performed using a MEGNETICSUSCEPTIBILITY BALANCE- faraday scientific. Mass spectra were recorded with Mass spectra GC-Shimadzu Qp-2010 Plus. atomic absorption Shimadzu Atomic Absorption / Flame Emission Spectrophotometer(AA680).

2.3 Methodology

2.3.1 Azo ligand synthesis (C₁₈H₁₇N₅O₂)

***N*-(2-(2-Amino-5-nitrophenyl)diazenyl)ethyl)naphthalen-1-amine**

Thereafter, ethanol (10 ml) and hydrochloric acid (2ml) were used to dissolve *N*-(1-Naphthyl) ethylenediamine dihydrochloride (0.13g), and the solution was clear. An aqueous sodium-nitrite solution (one gram in 10 milliliters of water) was added drop by drop and used to keep the temperature below 5 degrees Celsius. The

mixture was agitated for 30 minutes after the acidity was adjusted to a range of (5-6) using an ice bath and a potassium dihydrogenphosphate buffer. 0.069g of 4-nitro aniline was dissolved in 10 ml of ethanol and cooled to between (0-5) °C in an ice bath for the preparation. Melting point of ligand (137-139) degrees Celsius, it yielded an 83 percent reddish brown strong precipitate after drying and filtering. M.wt:(335g.mol⁻¹). C; 64.48: H;5.07: N;20.9: O;9.55 were found. It reads: cal: C;63,88 :H;4.86 : N;22.61: O;9.19. 3524cm⁻¹: 3427cm⁻¹: 3307 cm⁻¹: 1654 cm⁻¹: 1469 cm⁻¹: 1412 cm⁻¹: 3070 cm⁻¹ and 2924 cm⁻¹ are the IR(KBr) values. At 289 and 401 nanometers, UV-Vis (DMSO).

2.4 copper Synthesis (II) complexes

2.4.1 Synthesis of [Cu(C₁₈H₁₇N₅O₂)Cl₂] complex

0.085g of salt (CuCl₂.2H₂O) put in 10ml of distilled water, 20ml of buffer solution, were added to the solution of 0.17gm of ligand (C₁₈H₁₇N₅O₂) gradually with stirring, and the PH was set to (2-3) and the mixture was heated to a temperature of (60-70°C) for 8 hours with stirring before cooling to (25°C). After being filtered, the sediment was washed in a 1:1 mixture of water and ethanol, then dried in diethyl ether. Weight in g/mol: 469.55 g/mol . m.p.: 100-103 °C; yield percentage: 55%; As a result of our research, we have discovered that C;45.45: H;4.01: N;16.61: O;5.69 : Cl;14.14: M;12.91, were all found to be true and cal: C;45.96 : H;3.62 : N; 14.89 : O; 6.81 : Cl; 15.11 : M;13.52 . There are IR(KBr) wavelengths of 3399 cm⁻¹: 3384 cm⁻¹: 1653 cm⁻¹: 3299 cm⁻¹: 1454cm⁻¹: 1394cm⁻¹: 3070cm⁻¹: 2937cm⁻¹, as well as 505 cm⁻¹. A molar conductance of 4.5 S.cm².mol⁻¹ was measured in DMSO at 301 nm: 386 nm: 616 nm: and 687 nm UV-Vis.

2.4.2 Synthesis of [Cu(C₁₈H₁₇N₅O₂)(Ox)]complex

Solution (0.085 g) of (CuCl₂.2H₂O) salt put in 10ml of water and 20ml buffer solution, was mixed and added gradually with stirring to the primary ligand solution drop by drop then Oxalic acid (0.0639gm) was added to the mixture at (60-70 °C) for 8 hours with constant stirring, then the solution was cooled to (25 °C) and the PH was set to (2-3). After being filtered, the sediment was washed in a 1:1 mixture of water and ethanol, then dried in diethyl ether. 486.55 g.mol⁻¹ is the molecular weight for this compound. m.p.: 110-114 °C. 83 percent yield. Cal: C; 49.28: H;3.49: N; 14.37: O; 19.71: M;13.05 and found: C; 50.05: H;4.09 : N; 16.06 : O;18.81: M;14.14. There are 3472, 3429, 1674, 3307, 1454, 1359,3072, 2929, 1516, 1215, 446,and 503cm⁻¹ . In UV-Vis (DMSO), the wavelengths are 307 nm, 362 nm, and 702 nm. DMSO has a molar conductance of 2.5 S.cm².mol⁻¹

2.4.3 Synthesis of [Cu(C₁₈H₁₇N₅O₂)(Ma)]complex

Solution (0.085 g) of (CuCl₂.2H₂O) salt put in 10ml of water and 20ml buffer solution, was mixed and added gradually with stirring to the primary ligand solution drop by drop then Malonic acid (0.0528gm) was added to the mixture at (60-70 °C) for 8 hours with constant stirring, then the solution was cooled to (25 °C) and the PH was set to (2-3). After being filtered, the sediment was washed in a 1:1 mixture of water and ethanol, then dried in diethyl ether. Weight in g/mol: 500.55. m.p.: 119-122 °C. 73 percent yield. As a result of our research, we have

discovered that: C; 51.15: H;4.09 : N; 14.95 : O;20.21 : M;11.01, and Cal: C;50.40 : H;3.80 : N;14.00 : O;19.20 : M;12.70. IR (KBr): 3421: 3372 : 1653 : 3300 : 1456 : 1393 : 3120 : 2964 :1516 : 1215 : 412 , and 509 cm^{-1} . There are four wavelengths of UV-Vis (DMSO): 304, 342 and 392, and 640 nm. Conductance: 5.9 $\text{S.cm}^2.\text{mol}^{-1}$, DMSO-induced

2.4.4 Synthesis of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Pyph})]\text{complex}$

Solution (0.085 g) of $(\text{CuCl}_2.2\text{H}_2\text{O})$ salt put in 10ml of water and 20ml buffer solution, was mixed and added gradually with stirring to the primary ligand solution drop by drop then Pyph (0.223gm) was added to the mixture at (60-70 $^\circ\text{C}$) for 8 hours with constant stirring, then the solution was cooled to (25 $^\circ\text{C}$) and the PH was set to (2-3). After being filtered, the sediment was washed in a 1:1 mixture of water and ethanol, then dried in diethyl ether. M.wt:618.55 g.mol^{-1} . m.p.: 125-129 $^\circ\text{C}$. 88 percent yield. It was discovered that the following values were obtained using the following formula: C; 35.61: H; 3.88: N;14.95: O; 22.02: M; 11.87. Calc: C; 34.89: H; 2.75: N; 11.31: O; 23.26 : M; 10.27. IR(KBr): 3472 : 3421 : 1651 : 3307 : 1456 : 1398 : 3072 : 2927 : 916: 1207: 412 , and 567 cm^{-1} . To wavelengths of UV-Vis (DMSO): 293 nm, 309 nm, 488 nm, and 789 nm. 3.0 $\text{S.cm}^2.\text{mol}^{-1}$ is the molar conductance in DMSO.

3. Results and Discussion

The complexes are partially soluble in ethanol, acetone and diethyl ether and completely soluble in DMSO. The molar conductance of the complexes was measured in DMSO were found between 2.5 - 5.9 $\text{S.cm}^2.\text{mol}^{-1}$ indicating their non-electrolytic behavior. The structures of the copper complexes were elucidated via elemental analyses, magnetic, spectroscopic data as the following:

3.1 UV-Vis spectral

Table 1 summarizes Cu(II) spectral data of the complexes (**Figures 1 - 5**). The free ligand spectrum showed a strong absorption band at 289 nm (34602 cm^{-1} , $15100 \text{ L.mol}^{-1}.\text{cm}^{-1}$) which reflected $\pi \rightarrow \pi^*$ transition and a peak at 401 nm (24937 cm^{-1} , $9960 \text{ L.mol}^{-1}.\text{cm}^{-1}$) attributed to $n \rightarrow \pi^*$ transition (charge transfer within the azo group). In addition to the above two bands of the free ligands the spectra of complexes showed additional peaks characteristic to $d-d$ transition in a tetrahedral geometry [7]. The bands at 616 nm (16233 cm^{-1} ; $1630 \text{ L.mol}^{-1}.\text{cm}^{-1}$) and 687 nm (14556 cm^{-1} ; $2190 \text{ L.mol}^{-1}.\text{cm}^{-1}$) refer to ${}^2\text{B}_1 \rightarrow {}^2\text{E}$ and ${}^2\text{B}_1 \rightarrow {}^2\text{B}_2$ transition for the complexes (**1**) respectively , 702 nm (14184 cm^{-1} ; $2060 \text{ L.mol}^{-1}.\text{cm}^{-1}$), 640 nm (15625 cm^{-1} ; $5860 \text{ L.mol}^{-1}.\text{cm}^{-1}$) and 789 nm (12674 cm^{-1} ; $2090 \text{ L.mol}^{-1}.\text{cm}^{-1}$) refer to ${}^2\text{T}_2 \rightarrow {}^2\text{E}$ transition for the complexes (**2**), (**3**) and (**4**); respectively [8].

3.2 IR spectral data

Table 2 summarizes the Four complexes' FTIR spectral data. the IR spectra of the four complexes (**Figures 6-10**) were compared with the free ligand to configure the groups that may be involved in coordination. The amine group's bending frequency (δ - NH_2) is represented by the band at 1654 cm^{-1} in the spectrum of the free

ligand [9]. The stretching frequencies of the amine group ($\nu_{\text{asym}}-\text{NH}_2$), ($\nu_{\text{sym}}-\text{NH}_2$) and ($\nu_{\text{asym}}-\text{NH}$) are represented by the bands at 3524 cm^{-1} , 3427 cm^{-1} , and 3307 cm^{-1} , respectively. The bands at 1469 cm^{-1} and 1412 cm^{-1} stretching vibration, designated for the stretching frequency of the azo group ($\nu_{\text{N}=\text{N}}$), were discovered modified to $(1454; 1394)$, $(1454; 1359)$, $(1456; 1393)$, and $(1456; 1398)\text{ cm}^{-1}$ in complexes (1), (2), (3), and (4), respectively. The liberation of hydrogen bonding with the NH_2 may compensate for the anticipated shift in the frequency of the $\text{N}=\text{N}$ group upon coordination[10]. The presence of (PO_3) in complex spectra suggests that the $[\text{P}_2\text{O}_7]^{4-}$ ring adopts a bent conformation in conjunction with copper. Complex (4)'s IR spectrum revealed new bands at 1207 cm^{-1} and 916 cm^{-1} attributed to asymmetric and symmetric stretching vibrations of the terminal $\nu(\text{PO}_3)$ [11]. Cu(II) complex spectra revealed two novel bands assigned to (M-N) and (M-O), respectively, at 505 (1), 446 (2), 412 (3), and 412 (4) cm^{-1} and 503 (2), 509 (3), and 567 (4) cm^{-1} [12]. Measurements of the magnetic susceptibility measurements of the complexes confirmed the of the Paramagnetic nature and the existence of with divalent Copper (d^9) in a tetrahedral geometry.

3.3 Cu(II) complex mass spectra

The mass spectrum of the complex $[\text{Cu}(\text{azo})\text{Cl}_2]$ (**Figure 11**) (M. wt. = 469.55 g/mol) showed a parent peak at $m/z = 470$ (M^+). The mass spectrum of the complex $[\text{Cu}(\text{azo})(\text{Ox})]$ (**Figure 12**) (M. wt. = 486.55 g/mol) showed a parent peak at $m/z = 487$ (M^+). The mass spectrum of the complex $[\text{Cu}(\text{azo})(\text{Ma})]$ (**Figure 13**) (M. wt. = 500.55 g/mol) showed a parent peak at $m/z = 500$ (M^+). The mass spectrum of the complex $[\text{Cu}(\text{azo})(\text{pyph})]$ (**Figure 14**) (M. wt. = 618.55 g/mol) gave a parent peak at $m/z = 619$ (M^+). These values confirm the proposed formulae molecular weights.

Additionally, using analytical techniques such as Job's continuous and mole-ratio methods, the complexes' stoichiometries were verified as follows [13-14-15]:

3.4 Job's continuous and the mole-ratio methods

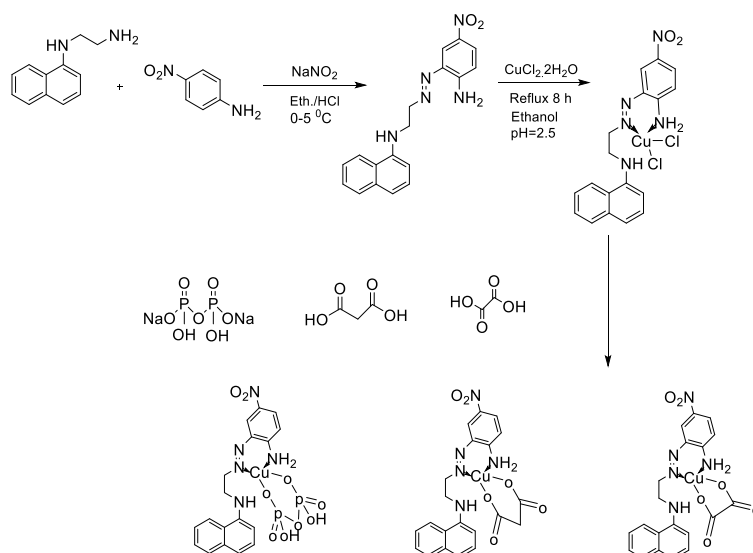
For the y- and x-axis plots, an acidic solution ($\text{pH} = 3$) was used to make a series of solutions, which were then combined with varying quantities of ligand to keep the final volume constant. **Figure 15** depicts the Job's approach curve for the Cu(II) ion solution with ligand at ($\text{max} = 687$). A 1.0 mole azo ligand and 1.0 mole copper (II) were found to be perfectly matched (II).

Mole-ratio method

A series of solutions with fixed copper (II) concentrations and variable volumes of the same concentration of the ligand solution were monitored at an optimal acidic function ($\text{pH} = 3$). Straight lines intersecting at the molar ratio of the reactants represent this ratio (ligand: metal). The color of the solution grows in intensity until it reaches the point at which the color stability begins to manifest, signifying the creation of the complex. It is possible to tell by looking at **Figure 16** what absorbance values correlate to what ratio of the copper-ion solution is present in the two intersecting regions. It was proven by the mole-ratio approach that 1:1 (M:L) complexes were formed. each azo ligand was linked to a 1.0 mole of copper (II).

Conclusions

A well-prepared and spectroscopically described azo dye ligand was used. On the basis of the bidentate azo ligand as a primary, four Cu (II) complexes were created. According to magnetic and UV-Vis measurements, the four complexes have tetrahedral geometries. The azo ligand was used to determine the Cu(II) ions spectroscopically.



Scheme 1: The synthesis of the azo ligand and the Copper complexes

Table (1): Results of UV-Vis measurements of complex solutions

No.	Compound	Wave length (nm)	Transition
1	Ligand N-(2-(2-Amino-5-nitrophenyl)diazenyl)ethyl)naphthalen-1-amine. [C ₁₈ H ₁₇ N ₅ O ₂]	28	$\pi \rightarrow \pi^*$
		9	$n \rightarrow \pi^*$, C
		40	T
		1	
		30	$\pi \rightarrow \pi^*$
2	[Cu(C ₁₈ H ₁₇ N ₅ O ₂)Cl ₂]	1	
		38	$n \rightarrow \pi^*$, C, T
		6	
		61	² B ₁ →
		6	² E
		68	² B ₁ →
		7	² B ₂
3	[Cu(C ₁₈ H ₁₇ N ₅ O ₂)(Ox)]	30	$\pi \rightarrow \pi^*$
		7	$n \rightarrow \pi^*$, C,
		36	T
		2	

		70	${}^2T_2 \rightarrow {}^2E$
		2	
		30	$\pi \rightarrow \pi^*$
		4	
4	$[Cu(C_{18}H_{17}N_5O_2)(Ma)]$	34	$n \rightarrow \pi^*$
		2	
		39	C.T
		2	
		64	${}^2T_2 \rightarrow {}^2E$
		0	
		29	$\pi \rightarrow \pi^*$
		3	
5	$[Cu(C_{18}H_{17}N_5O_2)(Pyph)]$	30	$n \rightarrow \pi^*$
		9	
		48	C.T
		8	
		78	${}^2T_2 \rightarrow {}^2E$
		9	

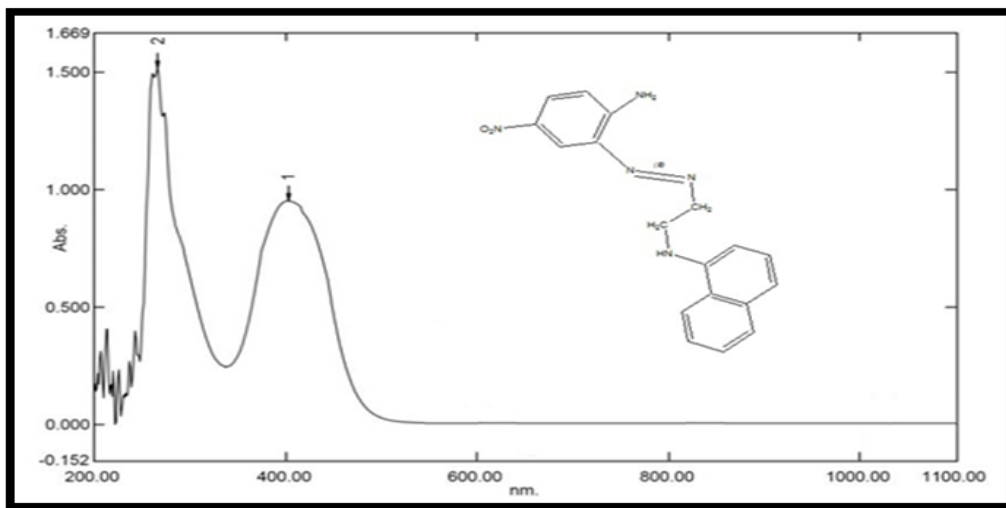


Figure 1: Primary ligand electronic spectrum ($C_{18}H_{17}N_5O_2$)

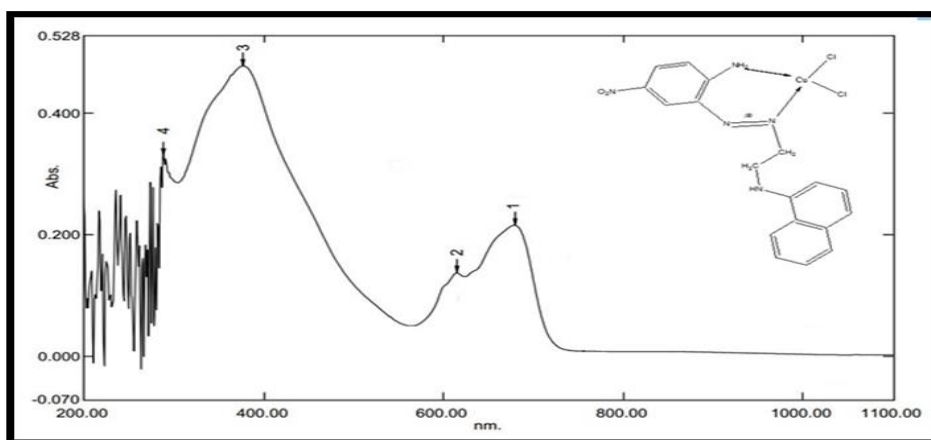


Figure 2: $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)\text{Cl}_2]$ electronic spectrum

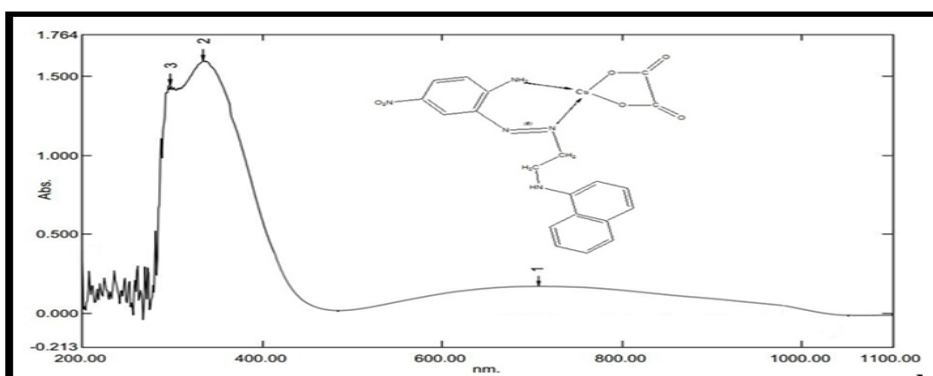


Figure (3) Electronic spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ox})]$

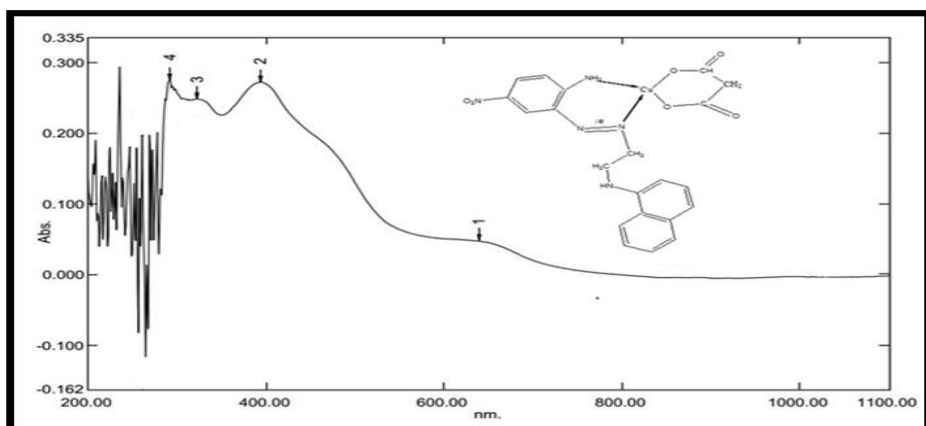


Figure (4) Electronic spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ma})]$

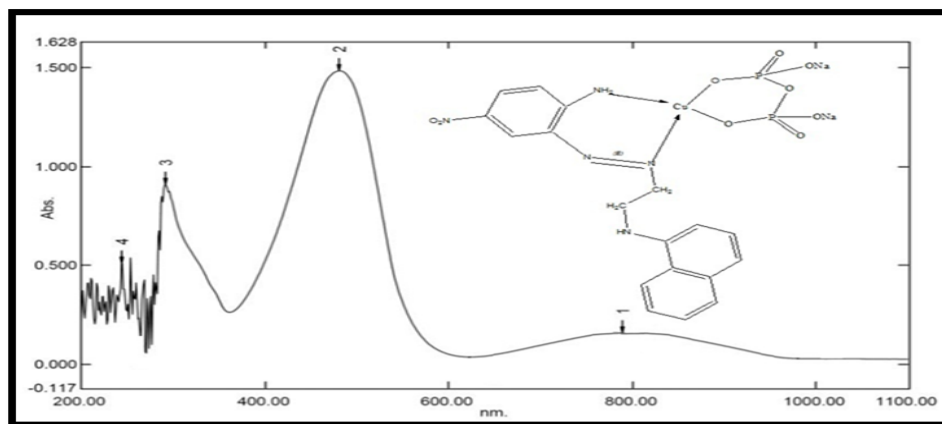
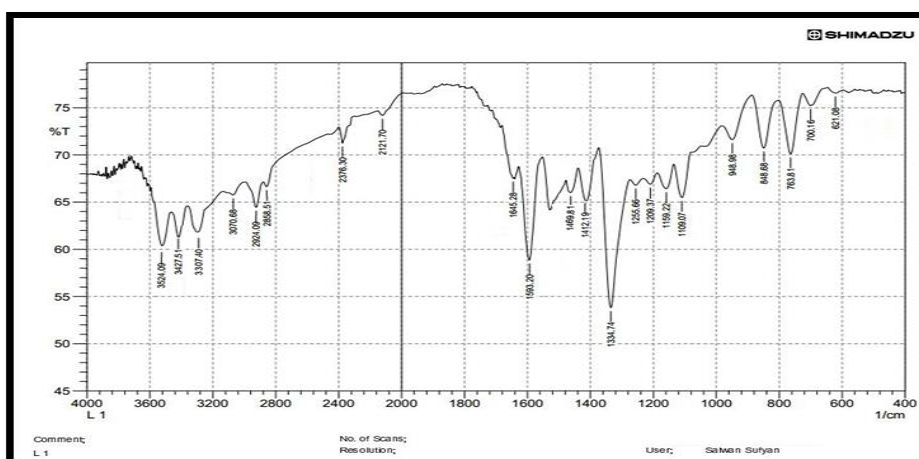
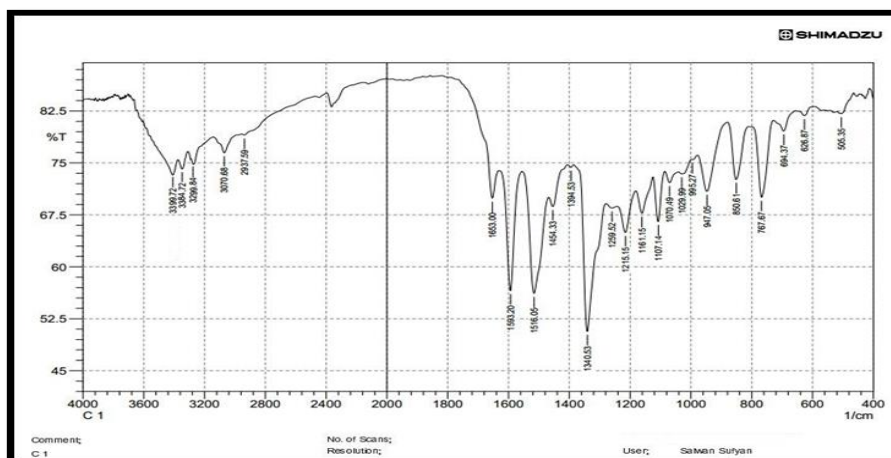
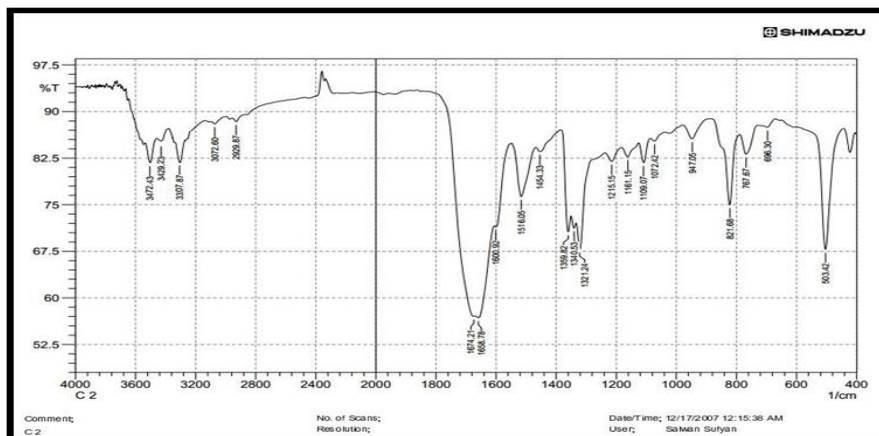
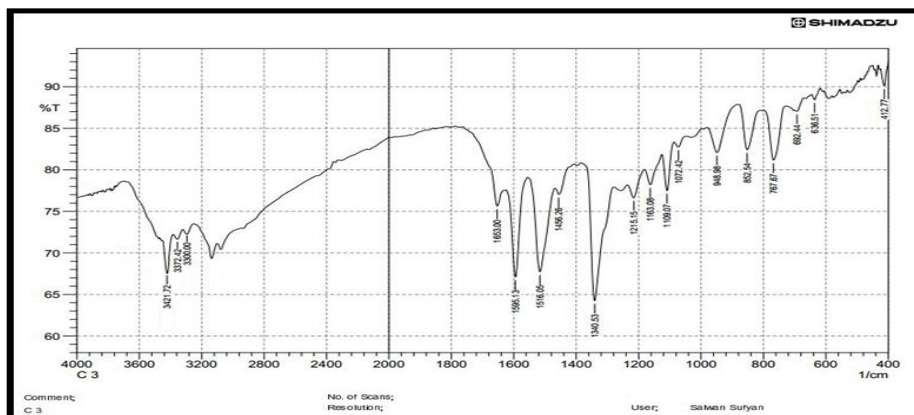
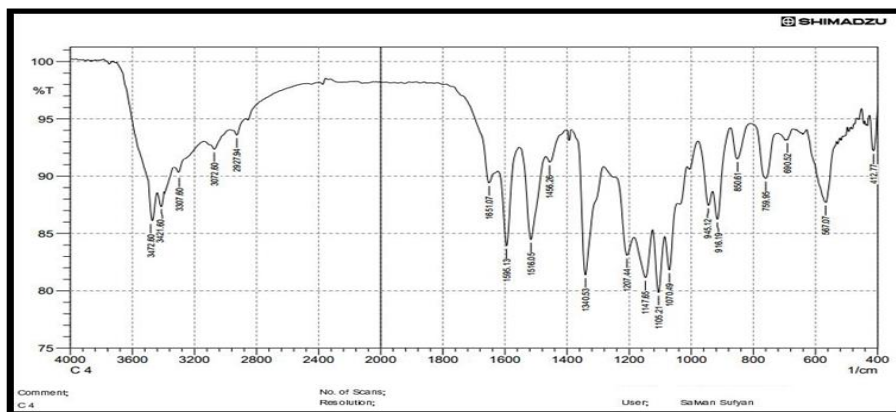
Figure (5) $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Pyph})]$ electronic spectrum

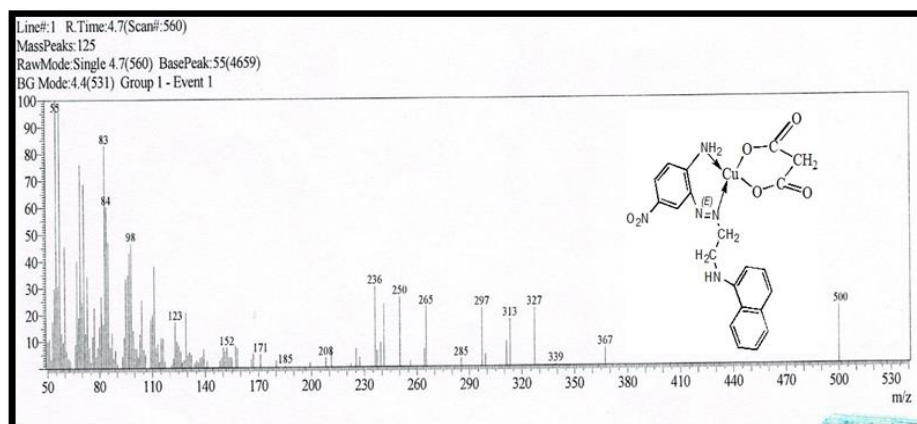
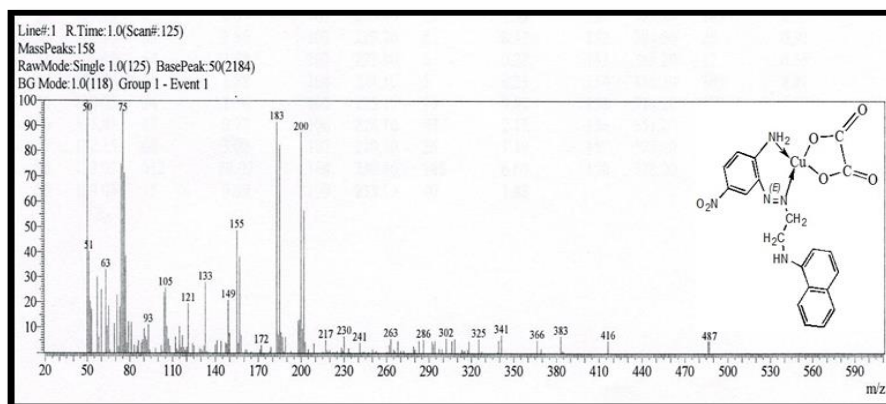
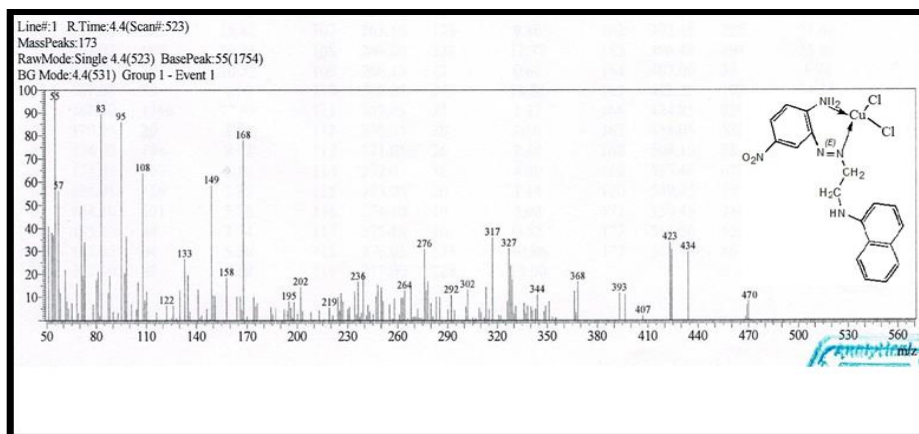
Table (2) FTIR Spectrum of the ligand and prepared complexes

Compound	N H	N H	N H ₂ δV	N H	N =	C O	CO O	P =	O- P-O	M -N	M -	CH Aro m	°C H Ali ph
Primary Ligand	3 5 2 4	3 4 2 7	16 54	33 07	1 4 6 9 1 4 1 2	- - - - -	- - - -	- - - -	--- --	-- --	- - - -	3 0 7 0	2 9 2 4
$[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)\text{Cl}_2]$	3 3 9 9	3 3 8 4	16 53	32 99	1 4 5 4 1 3 9 4	- - - - -	- - - -	- - - -	--- --	5 0 5	- - - -	3 0 7 0	2 9 3 7
$[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ox})]$	3 4 7 2	3 4 2 9	16 74	33 07	1 4 5 4 1 3 5 9	1 5 1 6	1 2 1 5	- - - -	--- --	4 4 6	5 0 3	3 0 7 2	2 9 2 9
$[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{M})]$	3	3	16	33	1	1	1	-	---	4	5	3	2

a)]	4	3	53	00	4	5	2	-	--	1	0	1	9
	2	7			5	1	1	-		2	9	2	6
	1	2			6	6	5	-				0	4
					1								
					3								
					9								
					3								
[Cu(C ₁₈ H ₁₇ N ₅ O ₂)(Py	3	3	16	33	1	-	-	9	12	4	5	3	2
ph)]	4	4	51	07	4	-	-	1	07	1	6	0	9
	7	2			5	-	-	6		2	7	7	2
	2	1			6	-	-				7	2	7
					1	-	-						
					3								
					9								
					8								

Figure (6) The Primary Ligand's FTIR-Spectrum (C₁₈H₁₇N₅O₂)Figure 7: The FTIR spectrum of [Cu(C₁₈H₁₇N₅O₂)Cl₂]

Figure 8: The FTIR spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ox})]$ Figure 9: The FTIR spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Ma})]$ Figure 10: The FTIR spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Pyph})]$



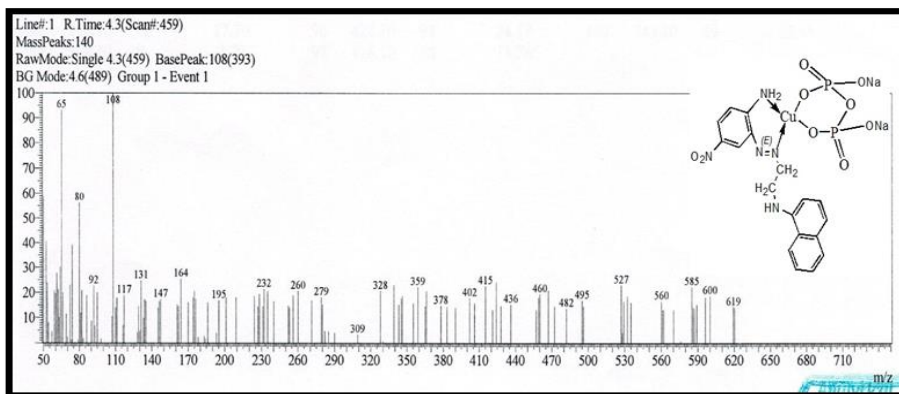
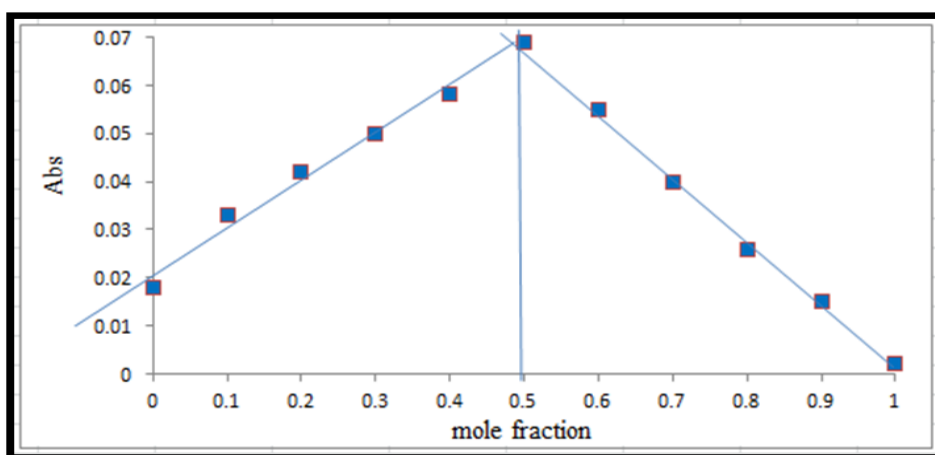
Figure (14) Mass spectrum of $[\text{Cu}(\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2)(\text{Pyph})]$ 

Figure (15) Curve of the Gob method to the Cu: L ratio

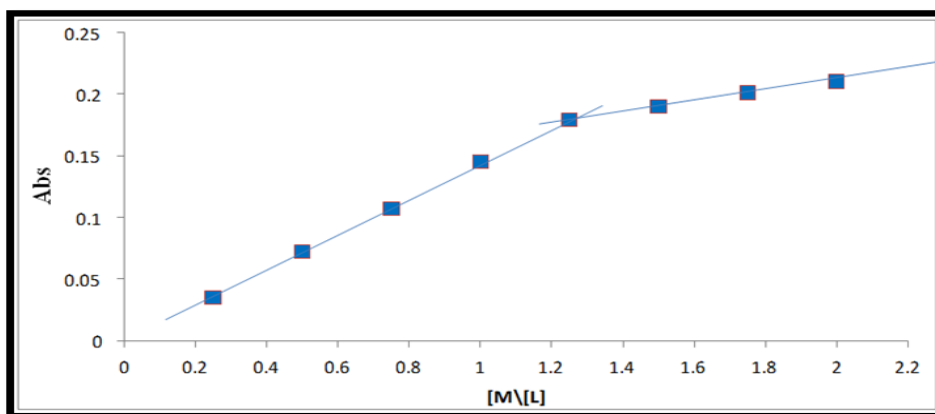


Figure (16) Curve mole ratio method for Cu:L

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