Spectroscopic and Conductometric Studies of Hexamethylenediamine Schiff Base Cu (II) Complexes

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NEW Schiff base, o-HOC$_6$H$_4$CH:N(CH$_2$)$_6$N:CHC$_6$H$_4$OH-o, and its copper complexes have been characterized by using elemental analysis, UV-VIS, IR, ^{1}H NMR, mass spectra, TGA, magnetic measurements and A.C electric conductivity. The formula of complexes were found (ML.nH$_2$O.X) for 1:1 M:L where X=Cl$^-$, Br$^-$, NO^{3-}, Ac$^-$, L = Ligand. The ligand and its complexes have been characterized by IR ,UV-VIS, ^{1}H NMR spectra and mass spectra which indicate that methine group and hydroxylic group in ortho position are involved in chelates besides to anions Cl$^-$, Br$^-$, NO$_3^-$ and Ac$^-$. Tetrahedral, octahedral and square planar geometries are proposed for the chelates based on their electronic spectra and magnetic moments. The investigated compounds behaved as semi-conducting materials.

The chemistry of Schiff base transition metal complexes attracted the focus of interest of several investigations in the field of bioinorganic and coordination chemistry[1]. The presence of lone pair on the nitrogen atom of imino group enables the coordination of numerous metal cations[2]. Transition metal complexes with oxygen and nitrogen donor Schiff bases are of particular interest because of their ability to have unusual configuration being structurally labile and their sensitivity to molecular environments[3]. Also, Schiff base can accommodate different anions of the same centeral metal involving various coordination modes thereby allowing successful synthesis of homo and hetro metallic complexes with varied stereochemistry. These features are employed for modeling active sites in biological system[4]. In the present work Schiff base was obtained from the condensation of hexamethylenediamine with salicylaldehyde. The structures of copper complexes with different anions have been confirmed by elemental analysis, FT-IR UV- VIS spectra, TGA, electric and magnetic measurements.

Experimental

Materials

Synthesis of N,N'hexamethylenebis (Salicylidenamine)-Schiff base (HBS)

The ligand was synthesized by dropwise addition of salicylaldehyde (5.47 ml, 44.86 mmole) in 100ml methanol to 1,6-hexanediamine (3gm, 25.81 mmole) in methanol as a solvent. The reaction mixture was heated under reflux for 2hr. The obtained yellow product was filtered off and washed with methanol then diethylether. The obtained product was recrystallized from methanol. The yield was (7gm, 83.73%), m.p 72ºC, Calcd for $C_{20}H_{24}N_2O_2$: C, 74.07; H, 7.41; N, 8.64, Found: C, 73.65; H, 7.77; N, 8.59 %. Scheme 1 indicate the preparation ligand (HBS).
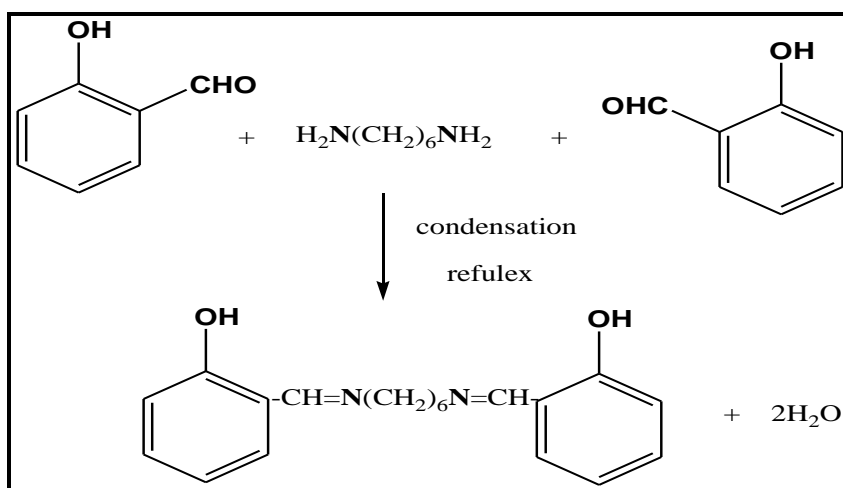
Scheme 1. Preparation of the Schiff base ligand (HBS).

Synthesis of schiff base metal complexes

Reaction of $CuCl_2.2H_2O$ with (HBS): A green solution $CuCl_2.2H_2O$ (0.341gm, 2 mmole) dissolved in (30 ml) methanol was added gradually and with constant stirring to a solution of ligand (HBS) (0.324 gm, 1mmole) in (30ml) methanol. The stoichiometry of metal ion to ligand was (2:1). The solution was refluxed for 2hr. A brown precipitate which obtained after cooling was filtered off and washed with small amount of methanol then diethylether. The yield was 0.2117gm (31.83 %), m.p >300 ºC.

Reaction $Cu(NO_3)_2.3H_2O$ with (HBS) : A blue solution $Cu(NO_3)_2.3H_2O$ (0.375 gm, 2mmole) in (30 ml) methanol was added gradually with constant stirring to a solution of ligand (HBS) (0.324 gm, 1mmole) in (30 ml) methanol. The solution was refluxed for 2 hr. A green precipitate was filtered off and washed with small amount of methanol then diethyl ether. The yield was 0.245gm (30.8 %), m.p >300 ºC.

Synthesis of L (HBS) Cu (CH₃COO)₂.H₂O complex

A blue green solution of $Cu(OAC)_2.H_2O$ (0.3993, 2 mmole) in (30ml) methanol was added gradually and with constant stirring to solution of ligand (HBS)(0.324, 1mmole) in (30ml) methanol. The solution was refluxed for 2 hr. A green precipitate was filtered off and washed with small amount of methanol then diethylether. The yield was (0.34g, 47%), m.p>300 ℃.

Synthesis of L (HBS) CuBr₂ complex

A green solution of $CuBr_2$ (0.4467 gm, 2 mmole) was added gradually with constant stirring in (30 ml) methanol to a solution of ligand L_1 (HBS) (0.324 gm, 1 mmole) in (30 ml) methanol. The solution was refluxed for 2hr. A green precipitate was filtered off and washed with small amount of methanol then diethylether. The yield was 0.452 gm (58.64 %), m.p >300 ℃

Techniques

The FTIR spectra (4000-400cm^{-1}) of different compounds were recorded as KBr discs using FTIR (Shimadzu) spectrophotometer model 4000. The FT-IR spectra (600-200 cm^{-1}) of various compounds were recorded as KBr discs using (Nexus 670) FT-IR spectrophotometer, Resolution 4 cm^{-1} polyethylene detector. Nicolet (U.S.A). Ultraviolet and visible spectra, were carried out on a Perkin Elmer Lambda 35 UV-Vis spectrophotometer in the range 190-500 nm.The solution spectra of ligands and complexes were carried out in 10^{-6} M of DMF. ^{1}H-NMR spectra were recorded using a Varian spectrometer, 200 MHz. TGA curves of different uncalcined compounds were measured at temperatures reaching to 850 ℃ at heating rate of 10^0C/ min. The data were obtained using a Shimadzu TGA-50H instrument. Mass spectra of the compounds were recorded on a Hewlett Packard mass spectrometer model MS 5988. Samples were introduced directly to the probe, fragmentations were carried out at 3000C and 70 eV. Molar conductivities were measured using WAP, GMP 500 conductivity meter. Magnetic susceptibilities of the complexes were measured by the Gouy method at room temperature using a magnetic susceptibility balance, Sherwood Scientific, Cambridge Science Park, Cambridge England. Effective magnetic moments were calculated from the expression $\mu_{eff} = 2.84(X_M)^{\frac{1}{2}}$ B.M., where X_M is molar susceptibility.

Results and Discussion

All the complexes provide satisfactory C, H, N and metal analyses and confirm the general formula $[Cu_2LX_2(H_2O)_2]$ and $[Cu_2L_2X_2(H_2O)]$ (where L = ligand, X = Cl$^-$, Br$^-$, NO$_3^-$ and Ac$^-$). The isolated solid complexes are stable in air. The product obtained was insoluble 1,2 dichloromethane, ethanol, methanol, chloroform but soluble in dimethylformamide (DMF) and dimethylsulfoxide (DMSO). The analytical and physical properties of the prepared complexes are in Table 1.

Molar conductance

The molar conductance (A_M) values of the complexes have been carried out using DMF as the solvent at concentration of 10^{-3} M are in the range of 3.91 –

14.7 ohm^{-1} cm^2 mol^{-1} for all complexes. The low values indicate that the nonelectrolytic nature of the complex may be formulated [Cu$_2$LX$_2$(H$_2$O)] and [Cu$_2$L$_2$X$_2$(H$_2$O)].

Magnetic susceptibility measurements

The complexes show at room temperature magnetic moments in the range 2 - 3.1 BM (Table 1) corresponding to one unpaired spin of Cu(II). But these values are noticeally higher than spin only value. The deviation from spin only value is due to mixing of angular momentum from exited state via spin orbit coupling [5].

TABLE 1. Elemental analyses and some physical properties of the ligand copper complexes.

Reactant	Mol. formula	Mol. wt	Colour	m.p°C.	Molar cond.	Analysis Found Calc				μeff (BN)
						C	H	N	Cu	
Ligand (1)	C$_{20}$H$_{24}$N$_2$O$_2$	324	yellow	70	3.91	73.65 74.07	7.77 7.41	8.59 8.64		
CuCl$_2$.H$_2$O+L$_1$	C$_{20}$H$_{26}$N$_2$O$_4$Cu$_2$Cl$_2$	556	brown	>300	14.7	44.61 43.16	4.69 4.32	5.35 5.04	22.73 22.82	2.61
Cu(NO$_3$)$_2$.4H$_2$O+L$_1$	C$_{40}$H$_{56}$O$_{20}$N$_8$Cu$_2$	1095	green	>300	6.87	43.09 43.83	5.57 5.11	9.78 10.22	10.97 11.59	2.53
Cu(OAc)$_2$.H$_2$O+L$_1$	C$_{44}$H$_{56}$N$_4$O$_{10}$Cu$_2$	927.3	Dark green	>300	4.86	57.12 56.95	5.94 6.04	6.70 6.04	13.80 13.70	2.73
CuBr$_2$+L$_1$	C$_{40}$H$_{54}$N$_4$O$_8$Br$_2$Cu$_2$	1004.8	Pale green	>300	7.02	46.87 47.77	5.38 5.37	5.55 5.57	13.13 12.63	2.9

IR spectrum of the ligand

The infrared spectrum of hexamethylenediamine therefore basically retained for the reaction product. Some spectral changes are expected to appear as being associated with the formation of new species of the expense of vanished amino group. Confirming this is the disappearance of the vibrational absorptions characteristic of the amino group at 3200 cm^{-1} and 3120 cm^{-1} (asymmetric and symmetric of NH$_2$ group, respectively). Consistent with the shift and change of intensity for methylene group at 2930 cm^{-1} and 2854 cm^{-1} asymmetric and symmetric of CH$_2$, 1460 cm^{-1} corresponding δ asymmetric CH$_2$ in plane bending, 1400 cm^{-1} δ symmetric CH$_2$ in plane bending and 881 cm^{-1}(γ CH$_2$ out of plane deformation). At the expense of vanished amino group species. New spectral absorption bands appeared (Fig.1) to be associated with the newly formed species consistent with this is the appearance of new broad absorption bands at 3445 cm^{-1} characteristic of ν_{OH} stretch and showed a new absorption band at 1631 cm^{-1} characteristic of $\nu_{C=N}$ stretch. New absorption band appeared at 1280 cm^{-1} can be assigned to the deformation consistent with this is the appearance of new absorption characteristic of ν_{C-O} which appeared at 1211 cm^{-1}. The most important bands in the IR spectrum of the complexes compared to its ligand are in Table 2 along with their tentative assignment. The positions of these bands provide significant indications regarding the bonding sites of the ligand molecule when complexed to Cu (II), hydroxyl group in ortho position

and imino group. The ν_{OH} band at 3445 cm^{-1} is absent from the IR spectra of the ligand which overlapped with H$_2$O coordinate but we could indicate the shift of ν_{C-O} band. But the ν_{OH} (H$_2$O coordination) at 3421 cm^{-1} is present, indicating that central metal coordinated by water molecule and OH in ortho position. This coordination is confirmed by the presence of two new bands in the range of ν_{M-O} and ν_{M-N}. Further in the spectrum of ligand the medium band observed at 1631 can be characterized to $\nu_{C=N}$ frequency. This vibrational absorption characteristic to C=N shift towards lower wave number by 20 – 35 cm^{-1} (Table 2) in the spectra of all metal complexes, suggesting the coordination of nitrogen lone pair of the azomethine group to central metal atom these complexes consistent with this the[6] metal – nitrogen bond is indicated by the absorptions in the region 458 – 522 cm^{-1}. Form the above interpretation of infrared spectra it is concluded that the Schiff base derived from hexanemethylenediamine is bonded to the metal ions as tetradentate ligand. The two bonding sites are the oxygen of hydroxyl groups of the benzene rings and nitrogen of azomethine which leading to form stable six membered chelating rings.

TABLE 2. IR spectra of the metal complexes.

Ligand/ Complex	νOH/ phenolic /H2O	νCHaro matic	νCHaliph (Sym) (Asym)	νC=N	ν C-O	νM-N	νM-O	νM-X	νAC
L$_1$(HBS)	3445w, br	3060 w	2931m 2854w	1631vs	1211m				
1-[Cu$_2$(L$_1$)(Cl)$_2$(H$_2$O)$_2$]	3421m, br	3066vw	2929m 2871 w	1629vs	1212m	462w	425w	330w	
2-[Cu$_2$(H$_2$L$_1$)$_2$ (NO$_3$)$_2$ (H$_2$O)$_2$]. 2NO$_3$.2H$_2$O	3424w,br	3056w,br	2924m 2852 w	1619vs	1201m	472w	420w		
3-[Cu$_2$(HL$_1$)$_2$(Ac)$_2$(H$_2$O)$_2$]	3446w,br	3021w,br	2918m 2849 w	1624vs	1196m	458w	420w		1536s 1347w
4-[Cu$_2$(HL$_1$)$_2$(Br)$_2$(H$_2$O)$_2$] 2H$_2$O	3416w,br	3018w,br	2920m 2852m	1623vs	1197m	522w	470w	320w	

IR spectra of anions

The Cu (II) complexes with different anions chloro, bromo, nitrato and acetato confirmed by infrared where the nitrato show these absorption bands at 1420, 1310 and 1028 cm^{-1} which are characteristic of nitrato group[7]. The positions of these bands indicate a monodentate coordination[8]. Also, chloro and bromo complexes show a spectral band for Cu-Cl and Cu-Br at 330 and 320 cm^{-1}. On the other band , acetate anion can, however coordinate in monodentate, bidentate or bridging bidentate manner [9]. The monodentate behavior of acetate group in the investigated complex is deduced from the frequency difference ($\Delta\nu$) between $\gamma_{as\ COO^-}$ and $\gamma_{s\ COO^-} \geq 185$ cm^{-1} (this range is reported for the acetate and amino acid, the monodentate behavior) between 1535 and 1347 cm^{-1}.
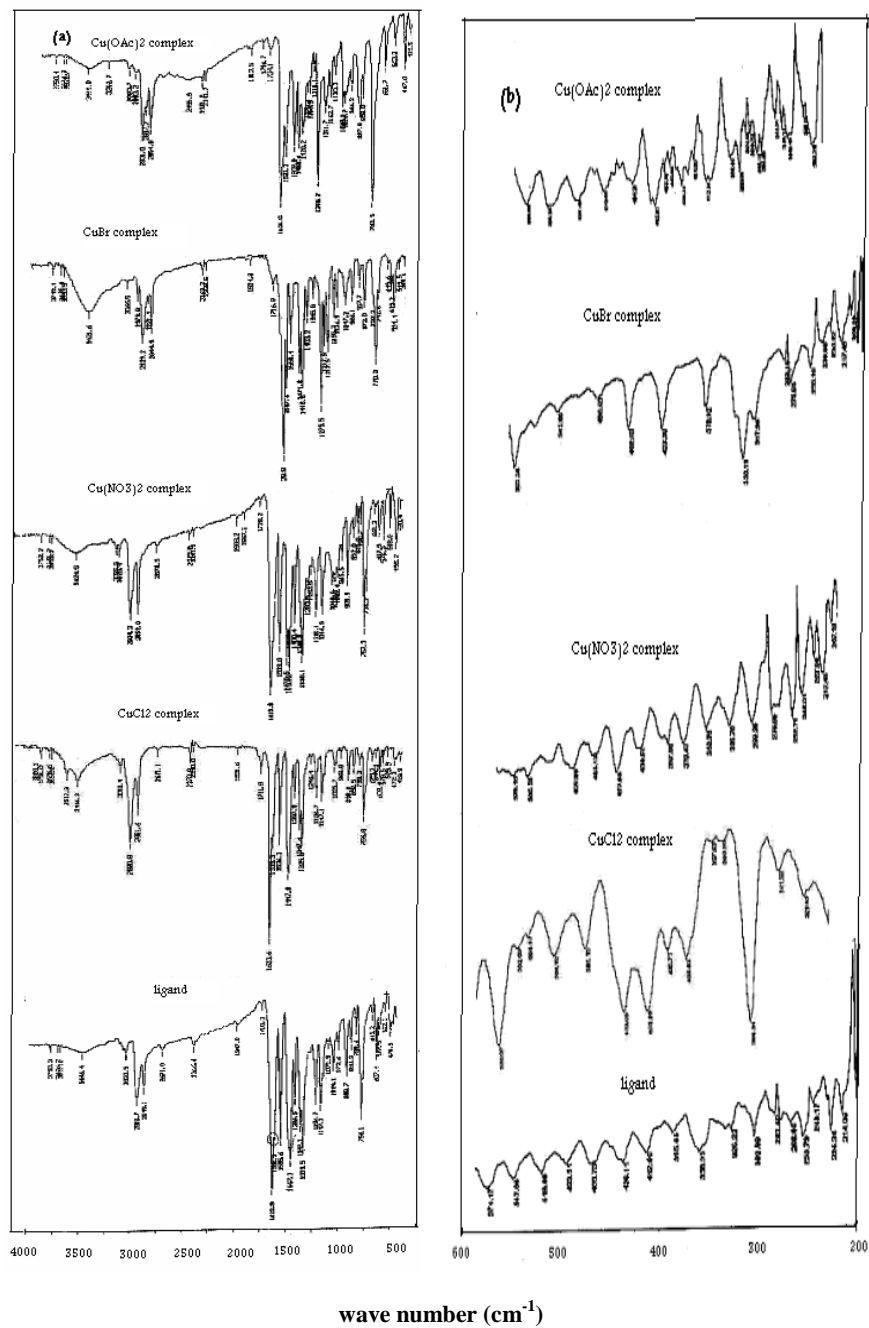
wave number (cm⁻¹)

Fig. 1 (a, b). Infrared spectra of ligand L (HBS), CuCl₂. 2H₂O, Cu(NO₃)₂. 4H₂O, CuBr and Cu(OAc)₂. 2H₂O Schiff base complexes.

Electronic spectra

The electronic spectra of the four copper complexes show bands in the range 622.73 – 672.94 nm which may be assigned to the $^2E_2g \rightarrow \ ^2T_2g$ transition in octahedral [10] geometry except Cu(II) complex (1) may be assigned to tetrahedral. The spectra of four complexes show band at 363 nm which is assigned to the (M→ L, CT transition). The electronic spectrum of brown Cu(II) complex (1) shows one band at 622.73 nm. Normally absorption band in the region 900 – 600 nm indicated the geometries octahedral, square planar or tetrahedral. Unfortunately, this is more difficult to distinguish that the type of geometry using the electronic spectrum, from the elemental analysis and other spectroscopic techniques. It is believed that the Cu(II) complex (1) has a coordinated number equal to four indicating the geometries is tetrahedral.

^1H-NMR spectra

The ^1H-NMR of the ligand shows four types of signals methylene protons, aromatic protons, azomethin and hydroxyl proton at (1.194 – 3.519 ppm), (6.6 – 7.4 ppm), (8.3–8.5 ppm) and (13.4–13.6 ppm), respectively. The signals of methylene are not influenced by chelation but the multiplet due to the aromatic protons   became broader and shift to lower field. The hydroxyl signal at (13.4 ppm) disappeared and azo methine signal is shifted, thus indicating that CH=N and OH group are involved in chelation. Figure 2 depicts ^1H-NMR of ligand.
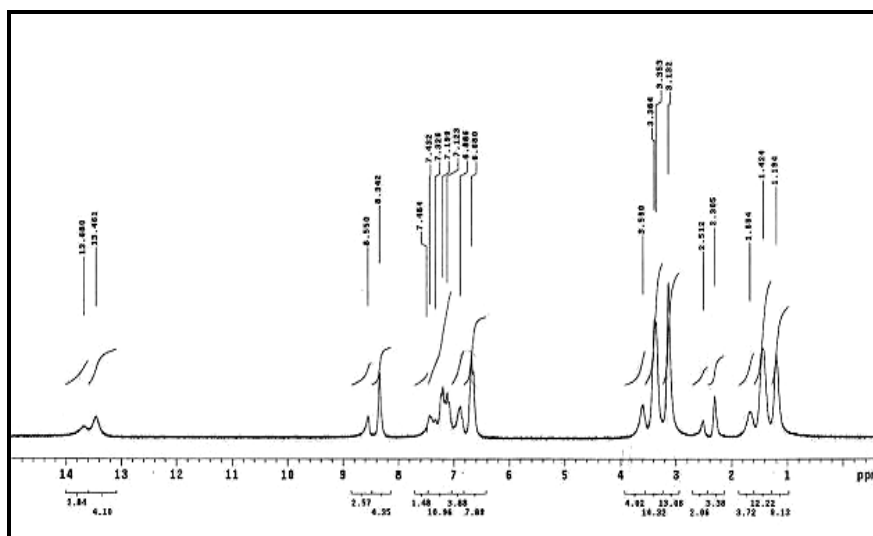

Fig. 2. ^1H- NMR spectrum of ligand.

Mass spectra

The mass spectrum of the free ligand confirmed the proposed formula of ligand by showing a peak at 324 due to molecular ion (parent peak). The series of peaks in the range of 65, 76,77, 91, 93, 94, 107, 123, 121, 121, 119, 120, 134, 148, 162,

176, 190, 204, 230, 231, may correspond to various fragments and their intensity gives an idea of stability fragments (Fig. 3) mechanism (Scheme 2).
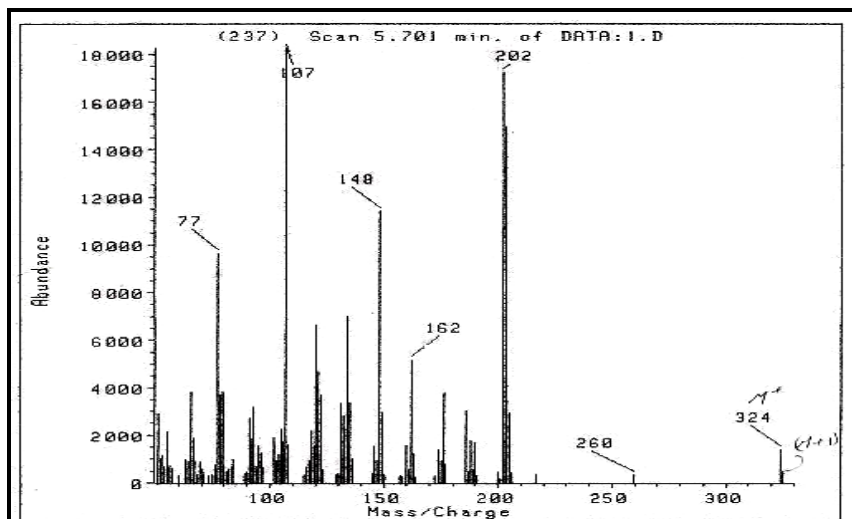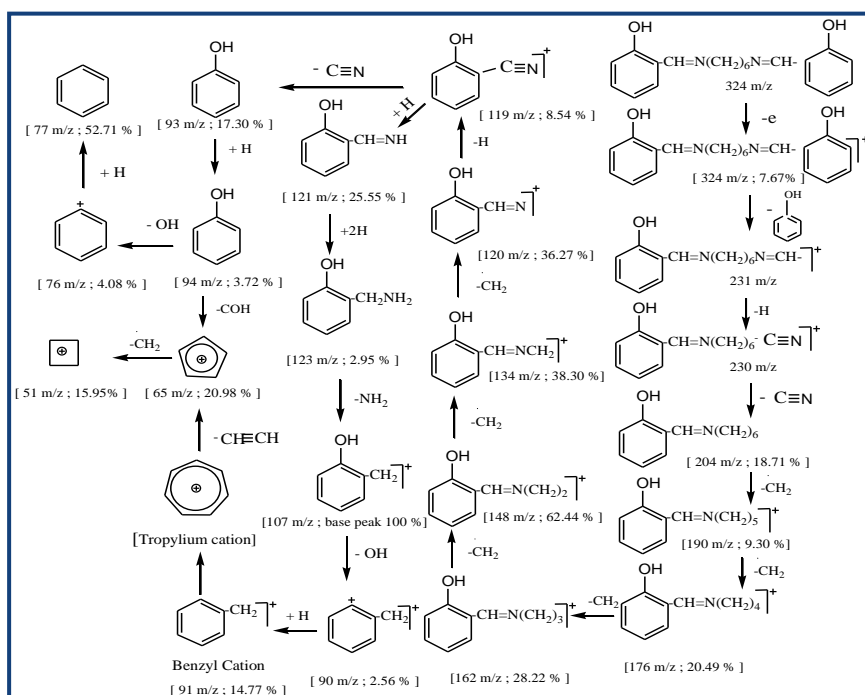

Fig. 3. Mass spectrum of ligand .

Scheme 2. Mass fragmentation of the ligand.

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Thermogravimetric analysis

TGA is used to confirm the structure of the complexes. TGA curve of $[Cu_2(L)(Cl)_2(H_2O)_2]$ shows four stages . The first one at ($35 - 226\ ^{o}C$) with a loss of two coordinated water molecules (Calc./ Found % ; 6.47 / 4.52 %) of total weight of the complex. The second stage at ($226 - 265.4\ ^{0}C$) corresponds to the loss of two chloride (Calc./ Found %; 13.65 / 12.42 %). The third stage at ($265 - 400^{0}C$) corresponds to the loss of $C_6H_{14}N_2O_2$ (calc./ Found % ; 32.96 / 31.47 %). The later stage was at ($400 - 650\ ^{0}C$) corresponds to loss of C_6H_5 (Calc./ Found ; 25.41, 24.42%).

TGA curve of $[Cu_2(L)_2(CH_3COO)_2(H_2O)_2]$ complex shows three stages. the first one is at ($35–258\ ^{o}C$) with loss of physisorbed and coordinated water molecules (Calc. / Found %; 3.88 / 3.38 %) of total weight of complex. The second stage at ($258-296\ ^{o}C$) corresponds to the loss of acetate group and $C_6H_{14}N_2O_2$ (Calc. / Found %; 29.68; 29.11 %). The third stage at ($296 - 650\ ^{o}C$) is due to the gradual decomposition of the complex corresponding to the loss of $C_8H_{14}N_2O_2$ & C_6H_5 (Calc. / Found %; 41.61/ 41.63%). Figure 4(a,b) shows the TGA curve of $[Cu_2(L_1)(Cl)_2(H2O)_2]$. $[Cu_2(L_1)_2(CH_3COO)_2(H_2O)_2]$. The total loss accompanying the complete decomposition of the ligand heated in air at temperature reaching to 600 ^{o}C attained (78.49, 72.97 %) this value is close to the calculated value on the bases of the complete decomposition of the ligand (75.17, 74.12 %), respectively. This finding indicates the absence of any contaminants in the ligand.

Dielectric constant study

The dielectric constant, ε', as a function of temperature was investigated at three spot frequencies (1, 10 and 100 kHz) and illustrated graphically in Fig. 5a for the ligand as a representative example. Two trends are shown clearly in the figure. The first one in the range of temperature ranging between room temperature about 300 Kand 345 K. The first trend shows that there is no effect neither of the temperature nor the frequency on the dielectric constant at least over these investigated ranges of both. This may reflect an expected case of stability due to the fact that, the bonding of the benzene rings to the carbon-nitrogen bond is very strong and the small thermal energy did not affect such bonds. In other words there is no polarization effect or migration of charge carriers throughout this range of temperature. The other range of temperature ranging from 345 to 400 K shows an increase of ε' with increasing temperature. The rate of ε' variation as a function of temperature is very high at the lower frequency 1 kHz and decreases in increasing frequency. This is due to the effect of electrical conductivity of the librated charge carriers at relatively higher temperatures (higher thermal energies). Further electrical investigations should be done to characterize the kind and the mechanism of the conductivity in such kinds of materials. On the other hand, the strength of the metal oxygen and metal nitrogen bonds is too high to be dissociate by increasing the thermal energy due to heating the sample in such ranges. The above mentioned bonds will increase in length as a result of increasing the degree of freedom of the

dipoles. The electric field accompanied by the applied frequency aligned them in its direction. The dielectric constant increases, which is the feature of the second region of temperature. This is of course in quite well agreement with the universal dispersion step known for the dielectric materials.
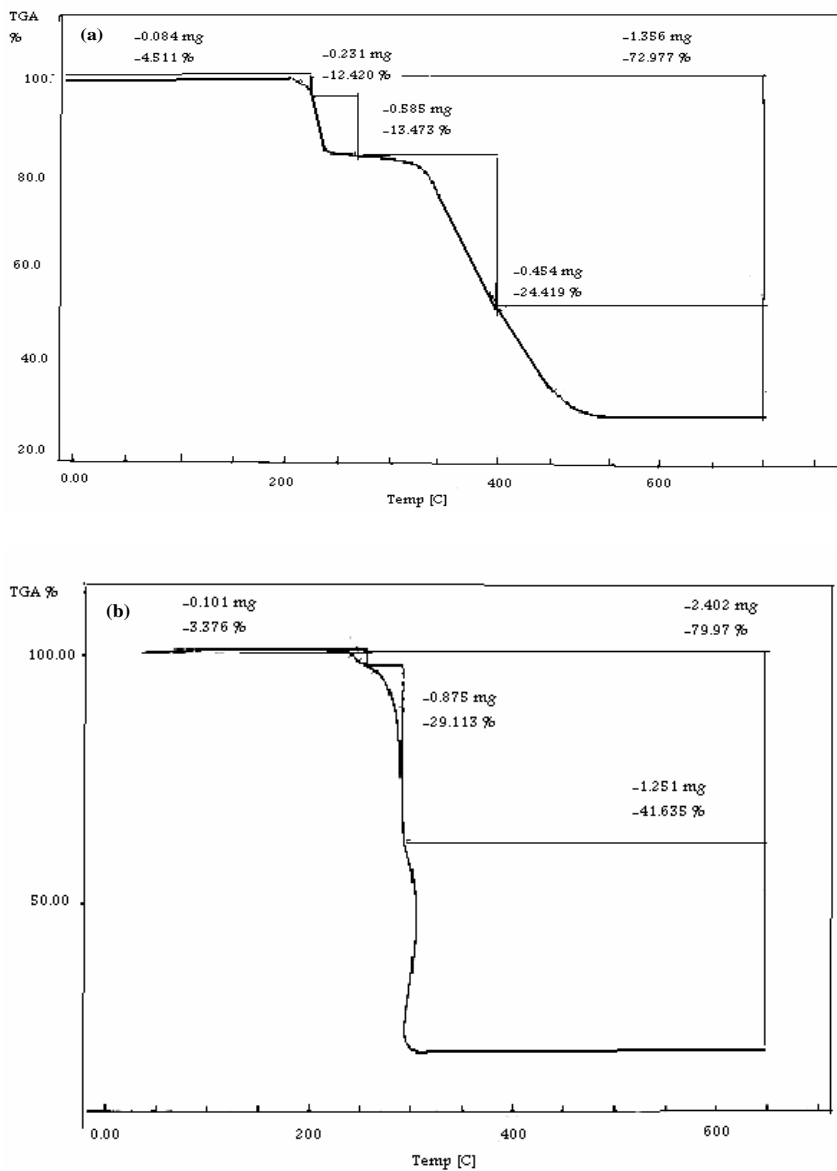

Fig. 4 (a,b). TGA curves of CuCl$_2$. 2H$_2$O and Cu(OAc)$_2$. 2H$_2$O Schiff base complexes.
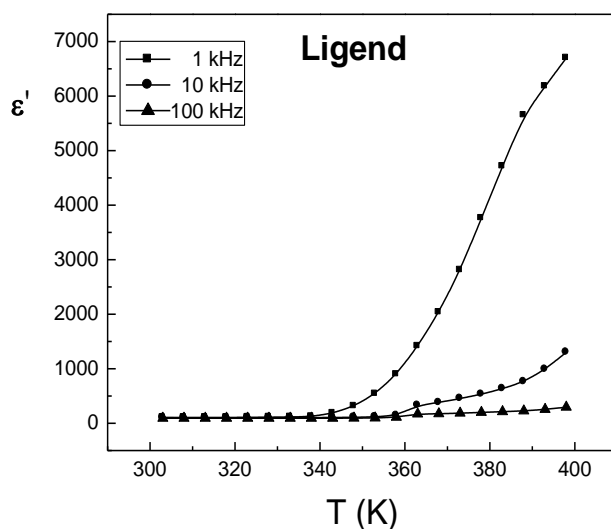
Fig.5. Dielectric constant έ as a function of temperature.

The imaginary part of the dielectric function ε'' is plotted versus temperature for three different samples of different anions namely, (Cl⁻, Br⁻ and NO_3^-) in Fig. 6a. From the figure it is clear that ε'' behaves in a plateau like trend in the lower temperature. The values of the dielectric loss ε'' increase by increasing the anion radius (Cl, Br, NO_3) over the range of temperature investigated here. The higher range of temperature shows a wing of some electric relaxation peak for all samples under investigation. This may support the need for further electric measurements especially the effect of temperature and frequency (broad band) on the complex conductivity. The real part of the dielectric function ε' shows a peak like behavior for the Cl⁻ anion sample as shown in Fig. 6b. The peak seems to be broader and shifted towards low temperature at the lower frequency. The increase of ε' in decreasing temperature at lower temperatures seems to be a wing of some relaxation peak whereas the increase in increasing temperatures at higher temperatures is due to the migration of charge carriers librated by heating.

The feature of dielectric change like the negativity of this anion, is shown from Fig. 8 but the ligand suddenly changed due to the variation of ionic radii. The values of activation energy at low temperature were calculated from experimental results (Fig. 9). From the data, it is clear that the activation energy decreases gradually with increasing the ionic radius, also this variation depends on the electronic configuration and on ionization potential.
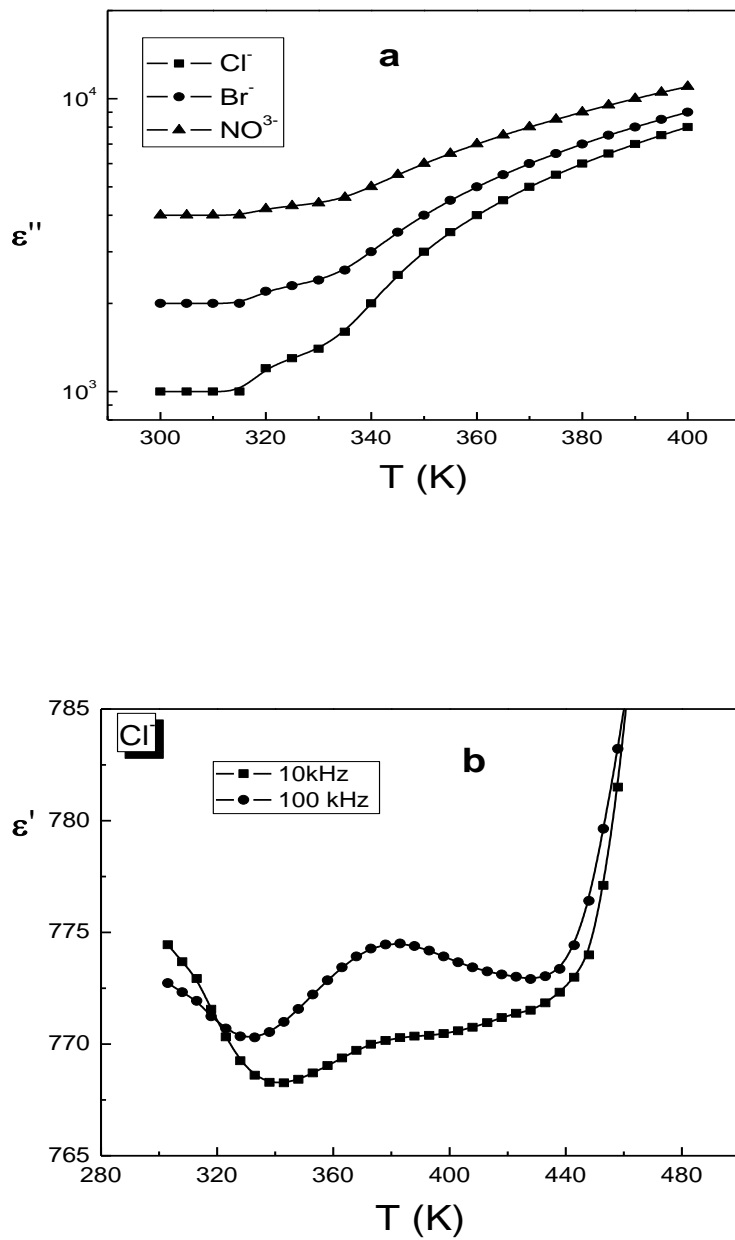
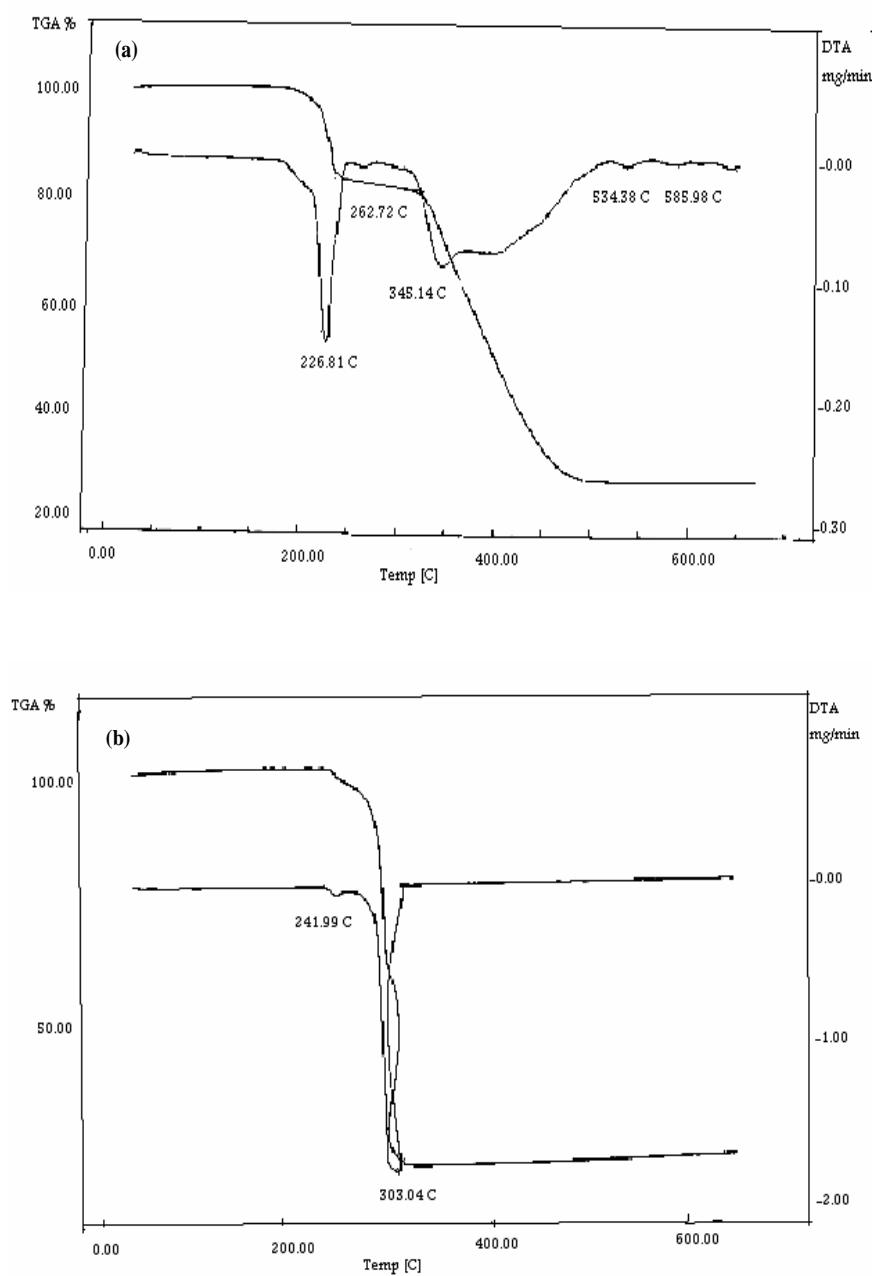**Fig.6 (a,b). Dielectric constant έ as a function of temperature.**

Fig. 7 (a,b). DTA curves of CuCl$_2$.2H$_2$O and Cu(OAC)$_2$.2H$_2$O Schiff base complexes.
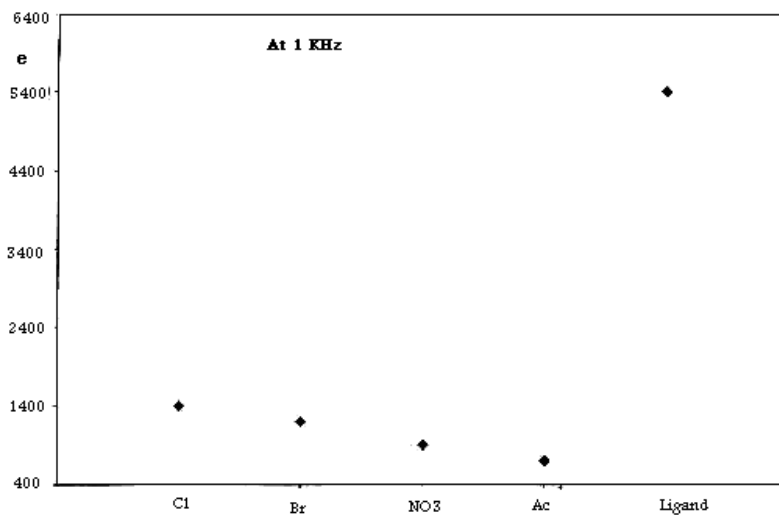
Fig. 8. Relation between e and different anions at 1 KHz.
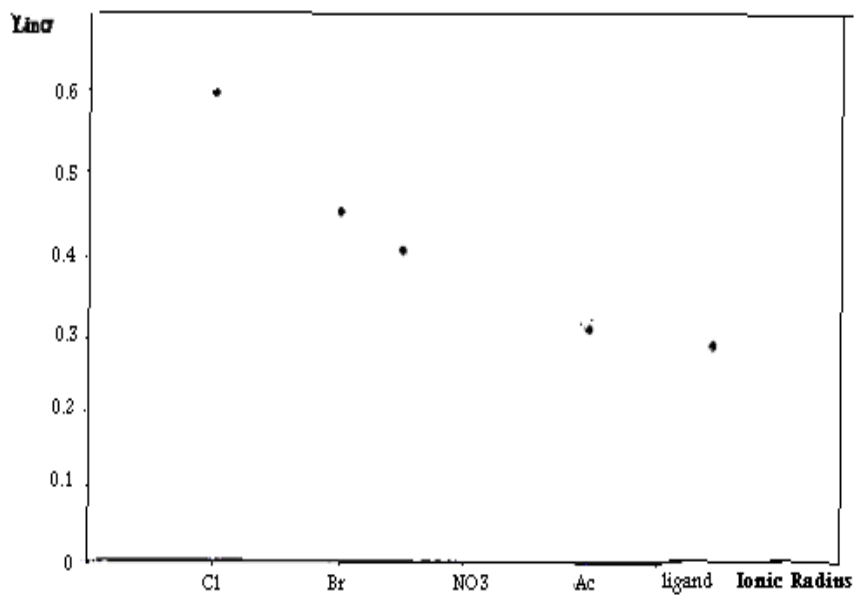
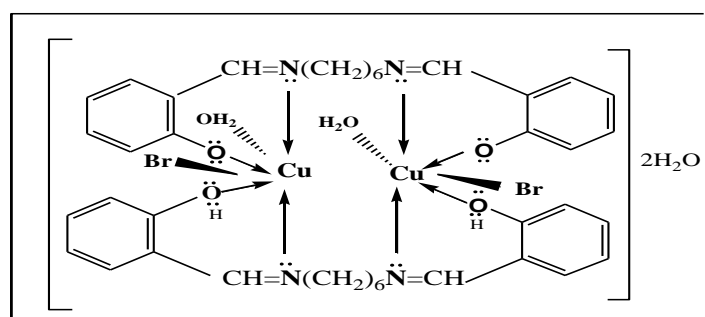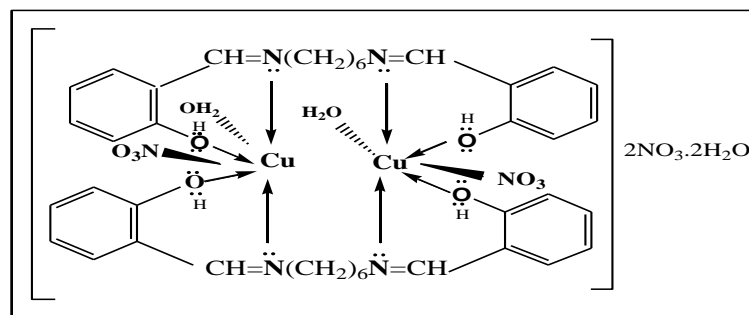**Fig. 9. Relation between ionic radius and lin σ.**

CH=N(CH₂)₆N=CH

L M
1 : 2

L M
1 : 1
2 : 2

CH=N(CH₂)₆N=CH

2H₂O

CH=N(CH₂)₆N=CH

L M
1 : 1
2 : 2

CH=N(CH₂)₆N=CH

2NO₃.2H₂O

L M
1 : 1
2 : 2

CH=N(CH₂)₆N=CH

CH=N(CH₂)₆N=CH
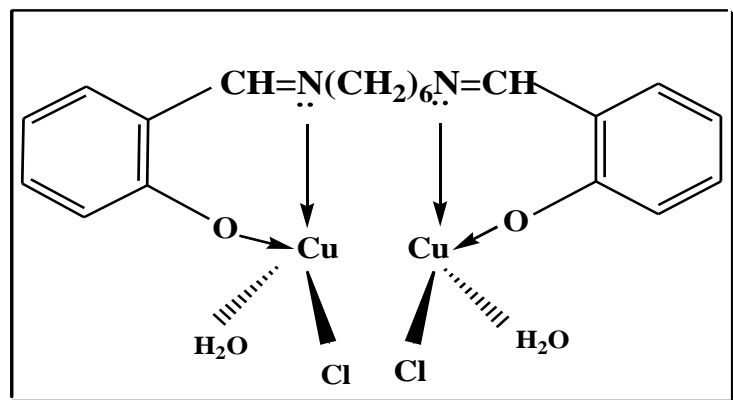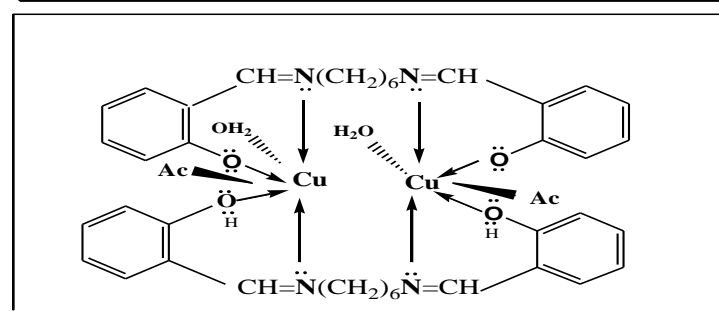
Representative structures of Cu(II) conplexes with different anions.

Conclusion

In conclusion, the Schiff base derived from condensation of hexamethylenediamine and salicylaldeyde is bonded to the Cu (II) ions as tetradentate ligand. The two bonding sites are the oxygen of the deprotonated hydroxyl group and the lone pair of nitrogen of azomethine group which lead to stable six membered chelating ring.

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دراسات طيفية وتوصيلية لمتراكبات النحاس باستخدام ثنائى الامين هكسان

علــى مصــطفى علــى حســن ، هــدى عبــد الحكــم بيــومى [*] **، مصــطفى ابــراهيم الدسوقى** [**] **، حمدى السيد حماد و منارة احمد ايوب** [*]

قسم الكيمياء- كلية العلوم ـ جامعة الازهر ، [*]قسم الكيمياء ـ كلية البنات ـ جامعة عين شمس و [**]قسم الفيزياء ـ كلية العلوم ـ جامعة الازهر ـ القاهرة ـ مصر .

تـــــــم أثـــبـــاتـــها بـــــوســـطـــة التـــحـــلـــبـــل العـــنـــصـــرى والاشـــعة الفـــوق بنفسـجية والمرئيـة والاشـعة تحـت الحمـراء والـرنين النـووى المغناطيسى و مطياف الكتلة والتحليل الوزنى الحرارى و قياسات مغناطيسية وتيار متردد للتوصيل الكهربى. اللجند تم اثباتها بواسطة الاشعة تحت الحمراء وطيف الكتلة وطيف الرنين النووى المغناطيسى وتم أثبات وجود مجموعتين الأزوميثين والهيدروكسيل الكحولية. واضحت النتائج ان متراكب النحاس يأخذ الشكل الرباعى الهرمى وشكل ثمانى الاوجه وشكل رباعى الاوجه مبنى على الاطياف الكترونية والعزم المغناطيسى. وأثبت الدراسة أيضا ان المركبات لها سلوك أشباه موصلات.