RESEARCH ARTICLE

Synthesis, Characterization and Electrical Study of Ligand 5, 5'-(4H-1, 2, 4- triazole - 3, 5- diyl) bis (azaneylylidene)) bis (methaneylylidene)) bis (4,1-phenylene)) bis (methaneylylidene)) bis (azaneylylidene)) bis (4H-1, 2, 4- triazol- 3- amine with Some its Transition Metal ion Complexes

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Abstract

A new series of some transition metal ion (Cr (III), Co (III), Fe (III) and Ni (II) complexes of new ligand {5, 5'- (4H-1, 2, 4-triazole- 3,5-diyl)bis(azaneylylidene))bis(methaneylylidene)) bis(4,1- phenylene)bis(methaneylylidene)) bis (azaneylylidene)) bis(4H-1,2,4-triazol-3-amine}(L) were synthesized .A ligand was synthesized by reaction 2,5-di amino-1,2,4-triazole and two mole for Terephthalaldehyde in presence of ethanol and drops for acetic acid to give 4,4'-((1Z,1Z')-((4H-1,2,4-Triazole-3,5,-diyl)bis(azaneylylidene))bis(methanelidene))dibenzaldehyde. Followed by reaction with two mole for 2, 5-diamino-1, 2, 4-triazole to give (L). The newly synthesized compounds were characterized by Fourier transform infrared (FTIR) Spectroscopy, Nuclear Magnetic Resonance Spectroscopy (^{1}H NMR), elemental analyses (C, H, N), Mass Spectra data, magnetic susceptibility data, Molar electrical conductivity and atomic absorption. The Hyperchem 0.75 program have been used to draw the ligand geometry optimization and then study the electrostatic potential that given right data about the active site. It has been shown through all the above diagnostic measures that ligand is formed with the (Fe, Co and Cr) ions tetrahedral complexes and with the nickel formed square planer complex, Electrical insulation properties were studied for L and their complexes with (Fe, Co and Cr) , The measured electrical conductivity results are measured at room temperature with the applicable field frequency, Note when frequency increase the applied field of (50 Hz -1MHz) Connectivity increases due to the direct relationship between frequency and electrical conductivity. During the study, both ligands and their complexes were I_2-doping showed that iodine increased the conductivity of Ligand, reduced the conductivity of complexes.

Keyword: *1, 2, 4-Triazole, Terephthalaldehyde. Electrical conductivity, Doping, Magnetic susceptibility.*

Introduction

A cyclic organic compound containing all carbon atoms in ring formation is referred to as a *carbocyclic compound.* If at least one atom other than carbon forms a part of the ring system designated as a *heterocyclic* compound.

Nitrogen, oxygen and sulfur are the most common hetero atoms [1, 2].Triazoles are five member heterocyclic compounds having three nitrogen atoms.

They are on two types Figure (1) and (2). A literature survey revealed triazole derivatives belonging to an important group of heterocyclic compounds that have been the subject of extensive study in the recent past. Diverse biological activities, such as antibacterial [3], antifungal [4], anticancer [5], anti-inflammatory [6, 7] antihypertensive, and antiviral, have been associated with 1,2,4-triazole derivatives [8, 9].
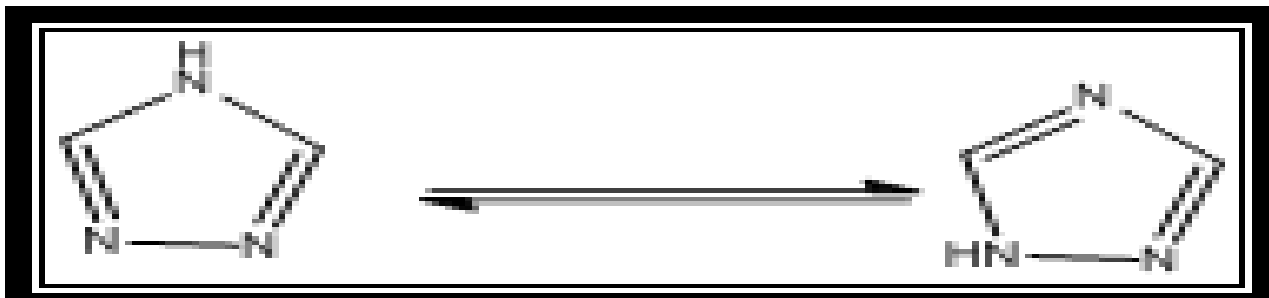
Figure 1: 1, 2, 4-triazole


Figure 2: 1, 2, 4-triazole

Experimental

Preparation of the Ligand (L)

Synthesis of 4, 4'-((1Z, 1'Z)-((4H-1, 2, 4-triazole-3, 5-diyl) bis (azaneylylidene)) bis (methaneylylidene)) dibenzaldehyde

Synthesis compound (A₂) was started with (0.02 mol, 2.68 g) terephthalic aldehyde, which was mixed with 15 ml ethanol and drops of ice acetic acid. After this (0.01 mol, 0.99 g) of 3, 5-diamino-1, 2, 4-triazole was added to this alcoholic solution. Mixture was allowed to reflux for 5 hours. After reflux, cold water was added and solid yellow product was obtained. Product was filter and dried for further use [10].

Synthesis of 5, 5'- (4H-1, 2, 4-triazole-3, 5-diyl) bis (azaneylylidene)) bis (methaneylylidene)) bis (4, 1-phenylene)) bis (methaneylylidene)) bis (azaneylylidene)) bis (4H-1, 2, 4-triazol-3-amine)

To a solution of compound A₂ (0.01 mol ,3.3g) in 50 ml of absolute ethanol, drops of ice acetic acid and 3,5-diamino-1,2,4-triazole(0.02 mol 1.98g) was added drop wise to the solution. The reaction mixture was refluxed for 4-5 hrs. Then cooled to room temperature, poured in 100 ml of ice water. The precipitated was filtered off, washed with water and recrystallized from ethanol M.P 299 D, yield 89% as the Scheme (1) [11].
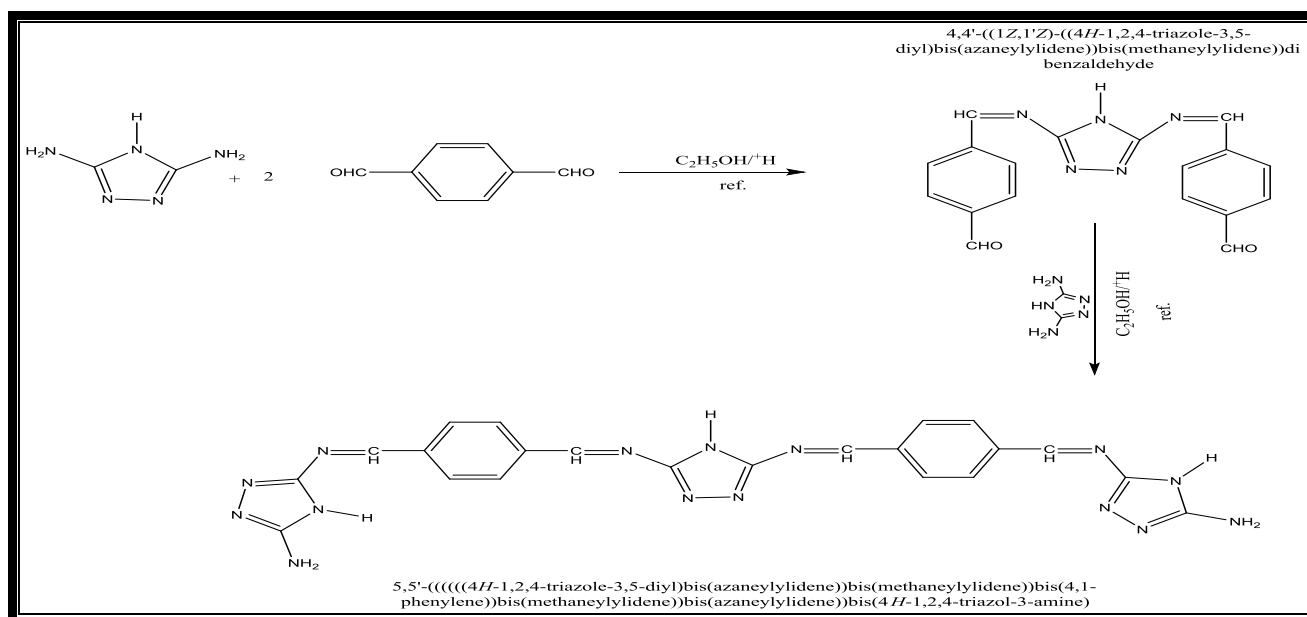
Scheme 1: Sequence of steps synthesis ligand

Complexes of L

The Cr (III), Co (III), Ni (II), and Fe (III) complexes were prepared by refluxing the respective hydrated metal chloride (0.001mol) in 15 ml ethanol with 50 ml of an ethanolic solution of the ligand (0.001 mol, 0.49g) for 3 hrs. The separated solids were filtered and washed with ethanol to removed unreacted salts or ligand, and then precipitated complexes dried in air. Melting point, physical properties of all compounds studied are tabulated in Table (1) [10].

Table 1: Melting point, physical properties of all compounds

No.	Compound	M. Wt	Color	M.P	Yield%
1	Ligand	493.00	yellow	299	89.0
2	$[Cr(L) Cl_2]Cl$	651.49	green	288-290	81.7
3	$[Co(L) Cl_2]Cl$	658.43	violate	212-214	78.3
4	$[Fe(L) Cl_2]Cl$	655.35	Dark brown	276-278	90.0
5	$[Ni(L) Cl_2]$	622.69	light green	234-236	73.8

Doping the Ligand and Complexes by Iodine

Mixed %10 of the ligands (L_3) and complexes with iodine through taking 0.1 from ligand or complexes with 0.01 from iodine, grind well to get a homogeneous mix to initialize the forming stage[12].

Preparation of Samples for Electrical Measurements

Samples were formed in a semi-dry form to reduce porosity .Where the mixture was taken by weight (0.25 g) of each mixture It was compressed with a hydraulic piston using a mold made of stainless steel thickness (0.68 cm) and diameter (1.72 cm), The mold is lubricated to reduce friction with powder minutes and mold walls and compress pressure (16.9 MPa) depending on the search program, take the time of pressing (3Min) to ensure flow. Form templates for presence and absence of iodine have been formed for testing Electrical insulation properties were studied using a device (LCR meter) at different frequencies ranging from (50 Hz -1MHz).

Experimental Section

Infrared absorption spectra were recorded on FTIR spectrophotometer on a model (Shimadzu FT-IR Spectrometer) in the range (200-4000) cm^{-1}. ^{1}H-NMR spectra were recorded at 500 MHZ utilize a model Bruker DRX, TMS use as standard, DMSO-d^6 used as solvent. Mass spectra (MS) were registered in the range (0-800) m/e on 5973 network mass selective detector. Elemental C, H, N and S analysis were carried out on a LECO elemental analyzer/CHNS-932. Melting points were specified in open capillary tubes using an electro thermal melting point/SMP31 device, LCR-8105G-LCR Meter, Bench, 5MHz, 100 kHz, 1F, 100 Mohm, LCR-8000G Series.

The ratio of the metal elements in the prepared complexes was determined using the flame atomic absorption technique using a type of device AA-6300 Shimadzu. University of Thi. Qar, College of Science. The molar electrical conductivity of the prepared complexes was measured using a type of device (Inolabcond 720) the polarization of platinum was the amount of constant cell 1.52 cm^{-1} with concentration (1*10^{-3}), University of Thi. Qar, College of Science.

Result and Discussion

The purity of 5,5'-(4H-1,2,4-triazole-3,5-diyl) bis (azaneylylidene)) bis (methaneylylidene)) bis (4,1-phenylene)) bis (methaneylylidene)) bis (azaneylylidene))bis (4H-1,2,4-triazol-3-amine and its complexes were checked by TLC .Elemental analysis (C, H, N) tabulated in Table (2).The theoretical values were in a good accordance with the experimental values.

Table 2: Elemental analysis (C, H, N, S)

Comp.	Elemental analysis theoretical				Elemental analysis Experimental			
	H%	C%	N%	S%	H%	C%	N%	S%
Ligand	3.85	53.55	42.59	---------	3.47	53.91	42.98	---------

FTIR Spectra of L₃ and Complexes

Infrared spectra are used to limitation of the electron donor atoms that contribute in the complex formation and identify the change compared with the ligand spectra. The characteristic bands in the Table (3).The free ligand showed five major bands at (3317.56) cm-1, (3140.11) cm-1, (1651.07) cm-1, (1519.91) cm-1, and (1350.17) cm-1, which are assigned to (υNH$_2$), (υC=H) aromatic, (υCH=N), (υC=N of triazole) and (υ C-N-C) bands respectively. The FTIR spectrum of compound (L) showed a medium intensity band at 1064.71 cm^{-1} that could corresponds with structure movement bond. The bands stretching of υNH$_2$ and υC=N (of triazole) in ligands shows shift to lower and higher wave number, this indicates that the coordinate occurred through the υC=N. New bands were formed due to the coordinated The stretching frequency at (516-indicating of 540) of complexes band corresponds to M-N group, and stretching frequency at (200-295) cm $^{-1}$ corresponds to M-Cl group[13, 16]. As showed in Figure (1)-(5).

Table 3: FT-IR absorption bands of the prepared L and their complexes

NO.	Compound	υNH2 cm-1	υCH aromatic cm-1	υCH= N cm-1	υ C-N-C cm-1	υC=N endocyclic cm-1	Structural movement cm-1	υM-N cm-1	υM-Cl cm-1
1	Ligand	3317.56	3140.11	1651.07	1350.17	1519.91	1064.71	--------	---------
2	[Cr(L)Cl2]Cl	3394.72	3132.40	1635.64	1381.03	1578.64	1056.99	540.07	295.26
3	[Co(L)Cl2]Cl	3302.13	3155.54	1620.21	1350.17	1512.19	1049.28	516.92	200.60
4	[Fe(L)Cl2]Cl	3318.89	3140.11	1620.21	1332.46	1512.19	1064.71	524.64	277.75
5	[Ni(L)Cl2]	3302.13	3129.46	1620.21	1303.88	1512.19	1049.28	532.35	231.46

Nuclear Magnetic Resonance Spectra (¹H-NMR)

The ¹H NMR spectra of free ligands were recorded in DMSO-2.51 and HDO-3.34.

(1H-NMR) of Ligand

¹H NMR spectrum of ligand shows the signals (ppm) at 6.16 (, 4H, 2NH$_2$), 8.06 (s, 8H, Ar-H), 9.08 (s, 2H, 2 N=CH), 10.12 (s, 2H, 2 N=CH), 12.12 (s, 3H, 3NH endocyclic) Figure (6).

Mass Spectra

Mass Spectra of Ligand

Mass spectrum of ligand shows the molecular ion peak at m/z 493 and other packs list in Table (4) which confirms the structural formula of L₃ as in the following Figure (7). The mechanics of break-up are embedded in the Scheme (2).
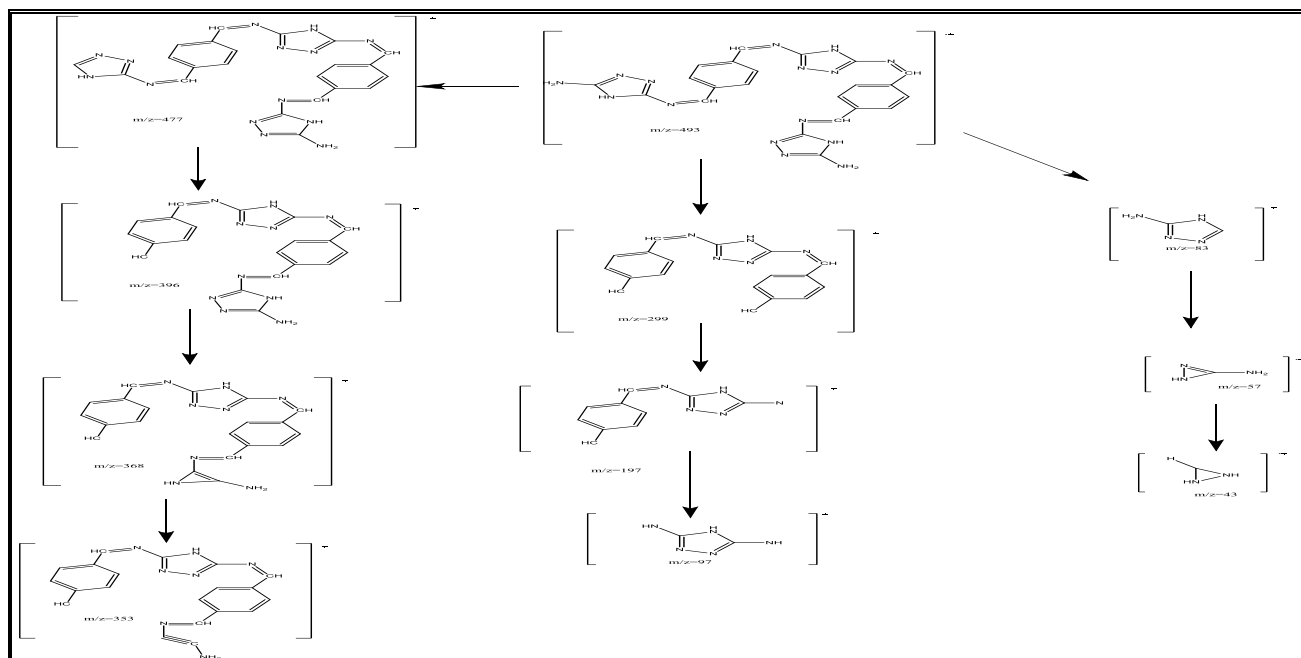
Table 4: The most important fractures in the Mass spectrum for Ligand

NO.	Molecular Ione	m/z
1	[C$_{22}$H$_{19}$N$_{15}$]$^{+\cdot}$	493
2	[C$_{22}$H$_{17}$N$_{14}$]$^{+\cdot}$	477
3	[C$_{18}$H$_{13}$N$_5$]$^{+\cdot}$	299
4	[C$_{20}$H$_{16}$N$_{10}$]$^{+\cdot}$	396
5	[C$_{10}$H$_7$N$_5$]$^{+\cdot}$	197
6	[C$_2$H$_3$N$_5$]$^{+\cdot}$	97
7	[C$_{20}$H$_{16}$N$_8$]$^{+\cdot}$	369
8	[C$_2$H$_3$N$_4$]$^{+\cdot}$	83
9	[CH$_3$N$_3$]$^{+\cdot}$	57
10	[CH$_3$N$_2$]$^{+\cdot}$	43
11	[C$_{20}$H$_{15}$N$_7$]$^{+\cdot}$	354

Mass Spectra of Complex [Cr (L) Cl₂] Cl

Mass spectrum of these complex shows the molecular ion peak at 651 m/z and other packs list in table (5) correspond with lose chloride atom. This confirms the structural formula of complex as in the following Figure (8).

Scheme 2: fragmentation proposed for ligand

Table 5: the most important fractures in the Mass spectrum for Cr-L Complex

NO.	Molecular Ione	m/z
1	$[Cr(L)Cl_2]Cl$	651.7
2	$[Cr(L)Cl_2]^{+\cdot}$	616.3
3	$[Cr(L)Cl]^{+\cdot}$	580.1
4	$[Cr(L)]^{+\cdot}$	545.6
5	$[Cr(C_9H_8N_5)]^{+\cdot}$	237.3
6	$[C_{10}H_7N_5]^{+\cdot}$	197.2

Mass Spectra of Complex [Co (L) Cl₂] Cl

Mass spectrum of these complex shows the molecular ion peak at 658 m/z and other packs list in table (6) correspond with lose chloride atom. This confirms the structural formula of complex as in the following Figure (9).

Table 6: the most important fractures in the Mass spectrum for Co-Complex

NO.	Molecular Ione	m/z
1	$[Co(L)Cl_2]Cl$	658.3
2	$[Co(L)Cl_2]^{+\cdot}$	622.7
3	$[Co(L)Cl]^{+\cdot}$	587.3
4	$[Co(L)]^{+\cdot}$	551.3
5	$[C_{11}H_8N_7]^{+\cdot}$	238.5
6	$[C_9H_7N_4]^{+\cdot}$	171.2

Mass Spectra of Complex [Fe (L) Cl₂] Cl

Mass spectrum of these complex shows the molecular ion peak at 655.7 m/z and other packs list in table (7) correspond with lose chloride atom. This confirms the structural formula of complex as in the following Figure (10).

Table 7: the most important fractures in the Mass spectrum for Fe-L Complex

NO.	Molecular Ione	m/z
1	$[Fe(L)Cl2]Cl$	655.7
2	$[Fe(L)Cl2]^{+\cdot}$	619.5
3	$[Fe(L)Cl]^{+\cdot}$	584.3
4	$[Fe(L)]^{+\cdot}$	548.6
5	$[Fe(C_{10}H_9N_5)]^{+\cdot}$	254.7
6	$[C_2HN_5]^{+\cdot}$	95.1

Mass Spectra of complex [Ni (L) Cl₂]

Mass spectrum of these complex shows the molecular ion peak at 622.4 m/z and other packs list in Table (8) correspond with lose chloride atom. This confirms the structural formula of complex as in the following Figure (11).

Table 8: The most important fractures in the Mass spectrum for Ni-L Complex

NO.	Molecular Ione	m/z
1	[Ni(L)Cl2]	622.4
2	[Ni(L)Cl]$^{+.}$	587.5
3	[Ni(L)]$^{+.}$	551.6
4	[Ni(C$_{10}$H$_9$N$_5$)]$^{+.}$	257.3
5	[C$_9$H$_6$N$_{14}$]$^{+.}$	170.2
6	[C$_{12}$H$_8$N$_7$]$^{+.}$	238.3

Magnetic Susceptibility

Magnetic susceptibility (µef) is a very important method to predict the structure of the complex. This measurement gives information about the type of bond and strength of ligand field of complexes by providing information about the number of unpaired electrons. The values of (µef) of complexes have shown in the Table (11).

As evidenced by the results that the ligand with iron (III), cobalt (III) and chromium (III) ions are produced tetrahedral complexes. Where iron has the value of µeff (5.8) they are identical to the electronic arrangement (eg^2 T$_2$g^3) with no orbital contribution, while the value of the µeff for cobalt equal to (4.7) it match the electronic arrangement (eg^3 T$_2$g^3) with no orbital contribution, as for the electronic arrangement of chromium is (eg^2 T$_2$g^1) So the value of the µeff (3.7) while the value of the µeff for Nickel equal to (2.8) and the electronic arrangement (T$_2$g^6 eg^2) Thus, the nickel is a square planer complex with the ligand (L) [17].

Molar Electrical Conductivity

The values of the molar conductivity of the complex depend on the number of ions outside the coordinate ball and that these values can be Provides us with good information about the complex formula of the complex, showing the molecular conductivity values of the complex Preparedness Some of these complexities behave like neutral compounds (Non-electrolytic) because of lack of Ions are outside the coordinate ball, while others are ionic cell salts and have varying conductivity Among them depending on the number of chloride ions outside the coordinate ball as accompanying ions central Ion (Counter Ions).

For the purpose of ascertaining the presence of chlorine ions outside the coordinate ball, water solution (AgNO$_3$) is added to the complex solution dissolved in (DMSO) where there was observed turbidity in the presence of chlorine out of the coordinating ball the turbidity increases with the number of chlorine atoms outside the coordinate ball and that the results are obtained were adapted to the proposed molecular and phylogenetic formulas of prepared complexes[18].

Flame Atomic Absorption

The proportion of metallic elements in their complexes was calculated using the atomic absorption technique; the results of the analysis were included in Table (11). When comparing the values obtained in practice with those calculated theoretically, the convergence between them is shown, which confirms the validity of the formulas proposed for the prepared complexes [19].

Table 11: Percentage of metal ions under study measured by atomic flame absorption, Molecular conductivity values Λ$_M$ for ligand complexes (L) in a solvent (DMSO) with concentration (1*10^{-3}) when the temperature (298K^o) and Active magnetic momentum values for the prepared complexes

No.	Complex	M% theoretical	M% experimental	Λm (S .cm^2 .mole^{-1})	Percentage of electrolyte	Electronic order	Meff
1	[Cr(L)Cl$_2$]Cl	7.99	8.31	38.8	1:1	eg^2 T$_2$g^1	3.7
2	[Co(L)Cl$_2$]Cl	8.96	8.62	37	1:1	eg^3 T$_2$g^3	4.7
3	[Fe(L)Cl$_2$]Cl	8.53	8.37	43	1:1	eg^2 T$_2$g^3	5.8
4	[Ni(L)Cl$_2$]	9.44	10.35	17	Non electrolyte	T$_2$g^6 eg^2	2.8

Results and Discussion of Electric Conductivity

The measured electrical conductivity results are measured at room temperature with the applicable field frequency, Note when frequency increase the applied field of (50 Hz -1MHz) Connectivity increases due to the direct relationship between frequency and electrical conductivity.

$$\sigma_{a.c} = 2\pi f \epsilon' r \epsilon_o$$

Figure (12) shows the linear relationship between the applied frequency and the electrical conductivity of the prepared ligand (L_3) , The conductivity of the ligand can be attributed to the repeat units and conductivity for some common conjugated Ligand [20]. The conjugated structure with alternating single and double bonds or conjugated segments coupled with atoms providing *p*-orbitals for a continuous orbital overlap (*e.g.* N) seems to be necessary for compound to become intrinsically conducting.

This is because just as metals have high conductivity due to the free movement of electrons through their structure; in order for ligand to be electronically, conductive they must possess not only charge carriers but also an orbital system that allows the charge carriers to move. The conjugated structure can meet the second requirement through a continuous overlapping of orbitals along the compound backbone.

The existence of exchange along, Due to its simple conjugated molecular structure and fascinating electronic properties. The conductivity of the prepared ligand with the conductivity of their complexes was compared as shown in Fig. (13), the comparison shows that the complexity of L_3 can increase the conductivity. In fact, the insulator-to-metal transition in conjugated compound is not so simple, and the way in which charges can be stabilized on the compound and the nature of the charge transport process are still a matter of debate.

Nevertheless, the simple band theory can provide some useful information about the doping induced changes in electronic structure. The energy bands of insulators and semiconductors, however, are either completely full or completely empty. For instance, most conventional compounds have full valence bands and empty conduction bands, which are separated from each other by a wide energy gap In contrast, conjugated compounds have narrower band gaps and doping can change their band structures by either taking electrons from the valence band (*p*-doping) or adding electrons to the conduction band *(n*-doping) [21].

All of this shows that the metals transition elements behave like (*n*-doping) as it works to add electrons to the conduction bands and thus significantly increase the value of the conductivity of the ligand. As shown by the Figure (13) that the electrical conductivity for complexes as follows Fe > Co > Cr this can be explained by the fact that the complexes have octahedral shape and the ligand is weak so the secondary cover electrons are based on the base of Hund, therefore iron has five single electrons, cobalt has four single electrons and chromium has three single electrons.

During the study, the ligand and their complexes were I_2-doping. Figure (14) and (17) showed that iodine increased the conductivity of Ligand; this can be attributed to iodine behavior as *p*-doping [21]. On the other hand, iodine has reduced the electrical conductivity of the complexes, although it remains higher than in the free ligand. This can be attributed to the fact that iodine will interact with the transition metals, the metal iodine component and thus reduce the conductivity of the complexes [22]. The following Table (12) shows the electrical conductivity values of ligands at (1 MHz) and their complexes with the presence and absence of iodine.

Table 12: Comparisons of the electrical conductivity of ligands and their complexes with the presence and absence of iodine

NO.	Comp.	The electric conductivity(S/m) absence iodine at 1MHz	The electric conductivity(S/m) presence iodine at 1MHz
1	L	0.04	0.21
2	Fe-L-Comp.	0.40	0.29
3	Co-L-Comp.	0.28	0.10
4	Cr-L-Comp.	0.23	0.11

Study the Electronic Density of the Ligand using the Program Hyperchem 0.75

A program was used hyperchem 0.75 to draw the prepared ligand and the distribution of the electronic density to find potential consistency positions using a method PM_3.

The study of the electrical voltage of the molecule is important to find effective sites in the molecular system, the program was also used to draw the stereotaxic body of the complexes Prepare and find out which formulas are most likely by comparing the energy content of these complexities as Shown below.
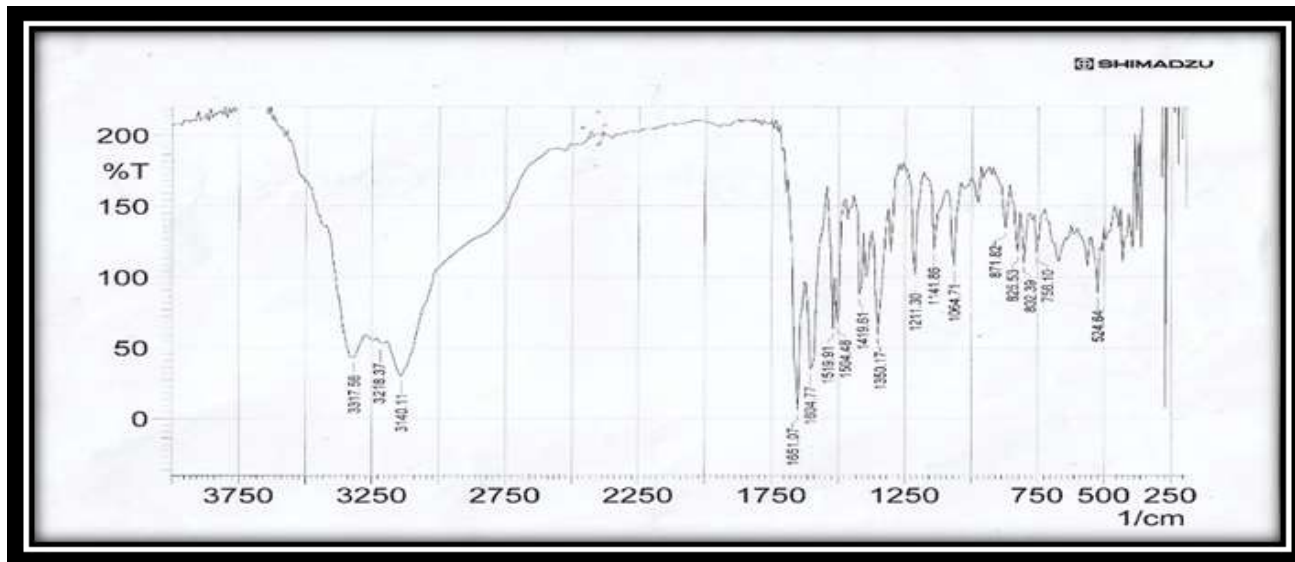
Figure 1: FTIR Spectrum of 5,5'- (4H-1,2,4-triazole-3,5-diyl)bis(azaneylylidene))bis(methaneylylidene))bis(4,1-phenylene))bis(methaneylylidene))bis(azaneylylidene))bis(4H-1,2,4-triazol-3-amine
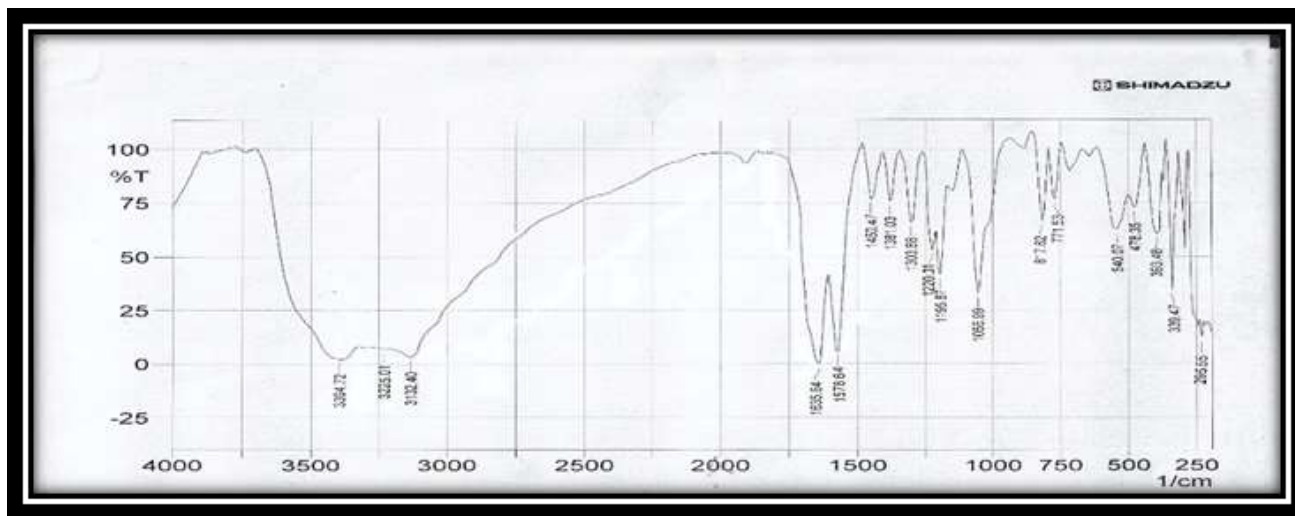
Figure 2: FTIR Spectrum of [Cr (L) Cl₂] Cl
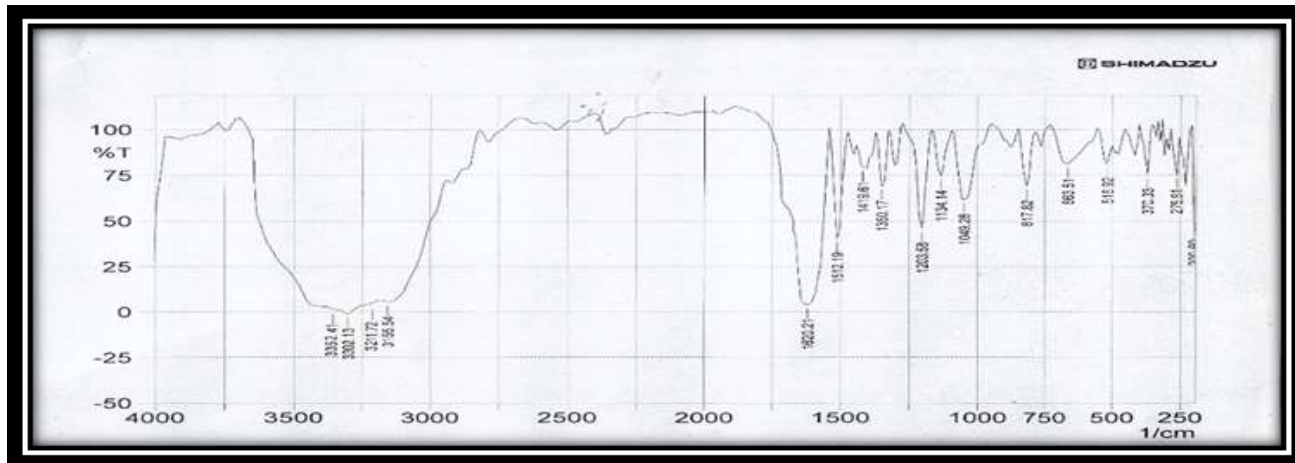
Figure 3: FTIR Spectrum of [Co (L) Cl₂] Cl
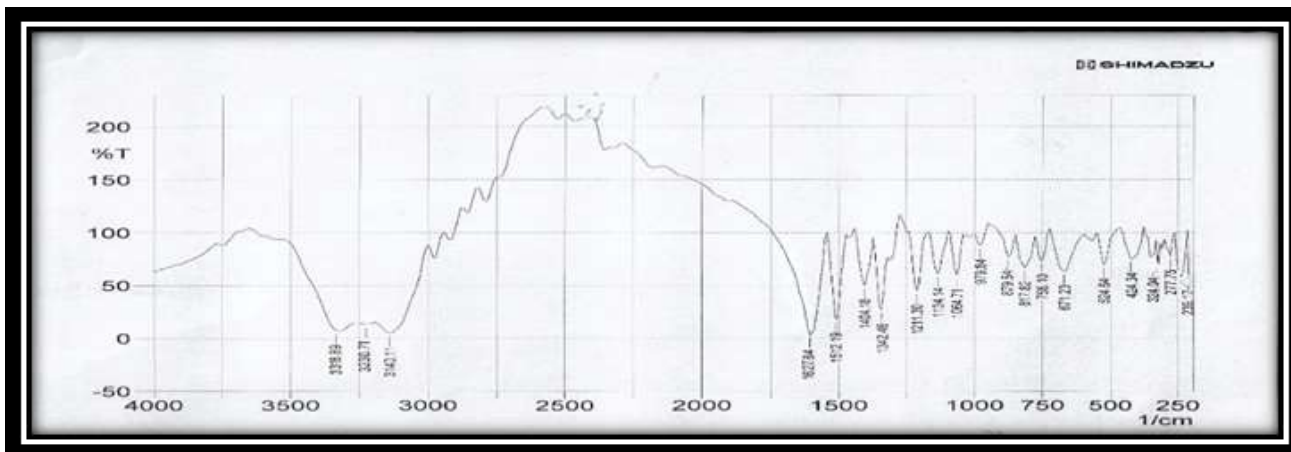
Figure 4: FTIR Spectrum of [Fe (L) Cl₂] Cl
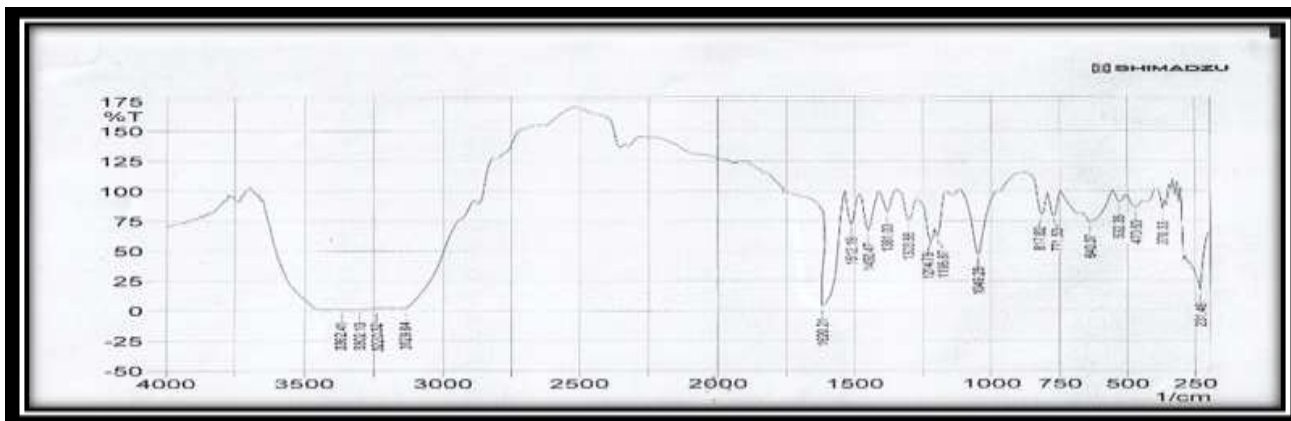
Figure 5: FTIR Spectrum of [Ni (L) Cl₂]
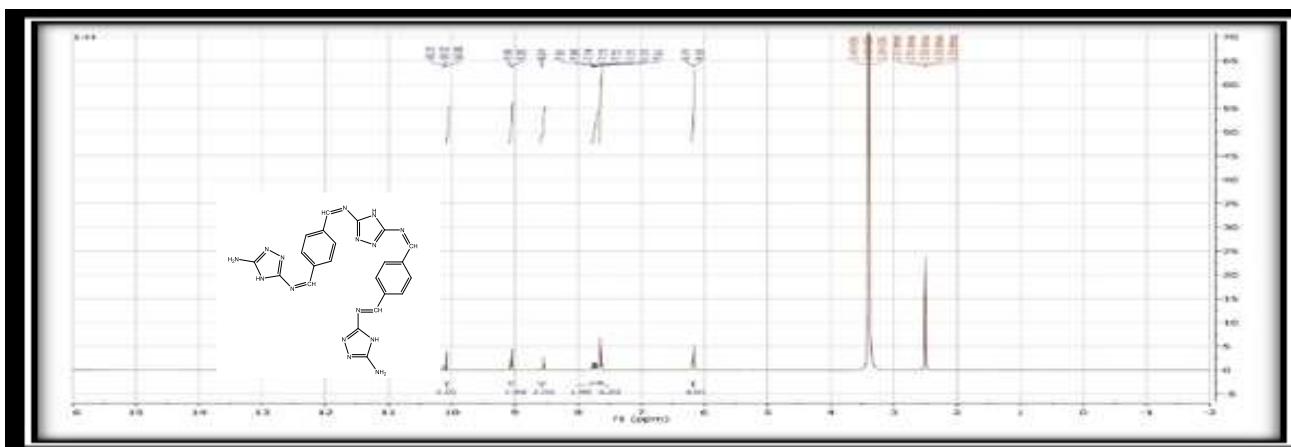
Figure 6: ¹HNMR Spectrum of Ligand
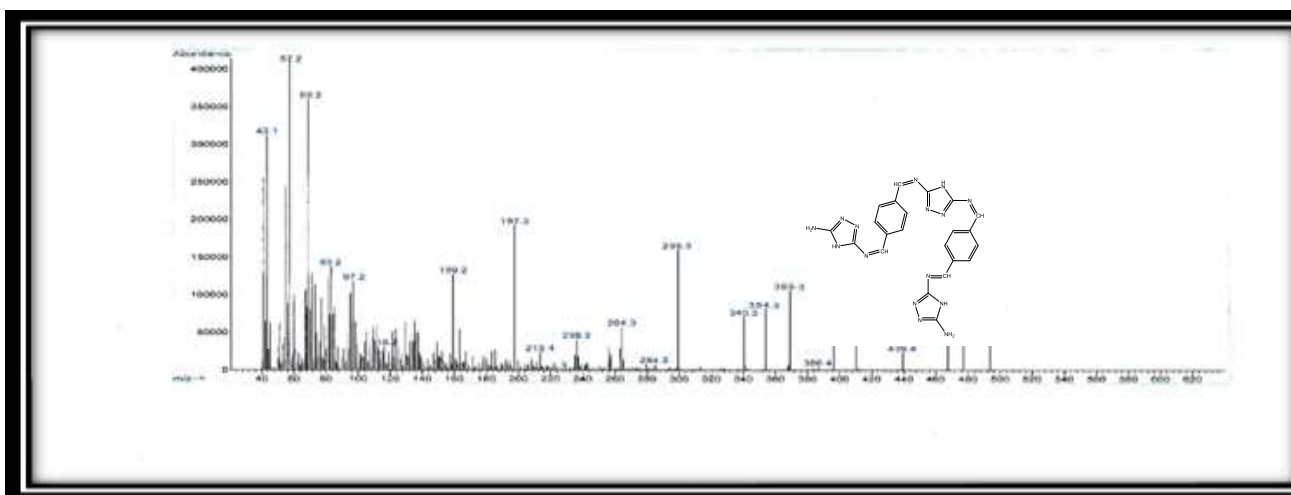
Figure 7: Mass Spectrum of Ligand
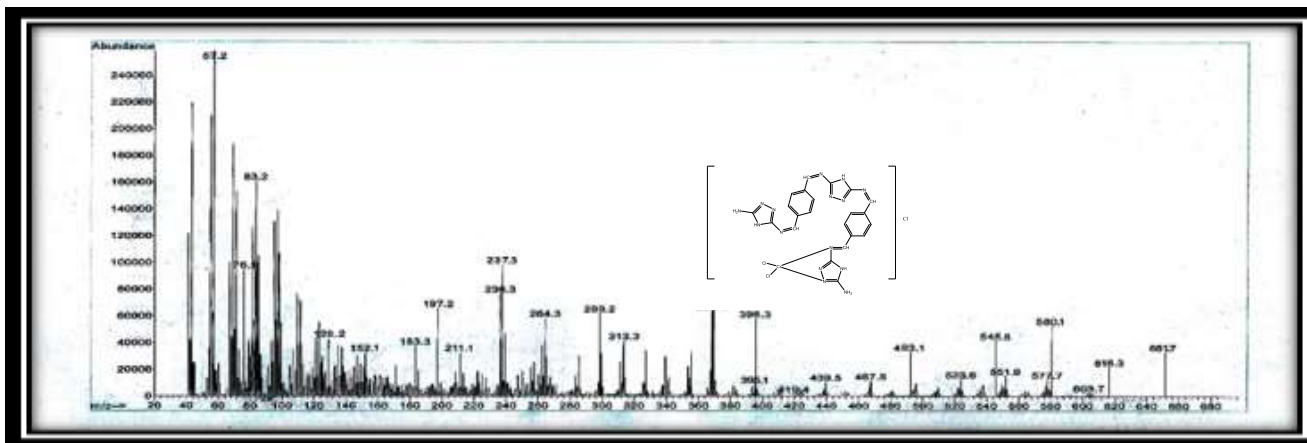
Figure 8: Mass Spectrum of [Cr (L) Cl₂] Cl
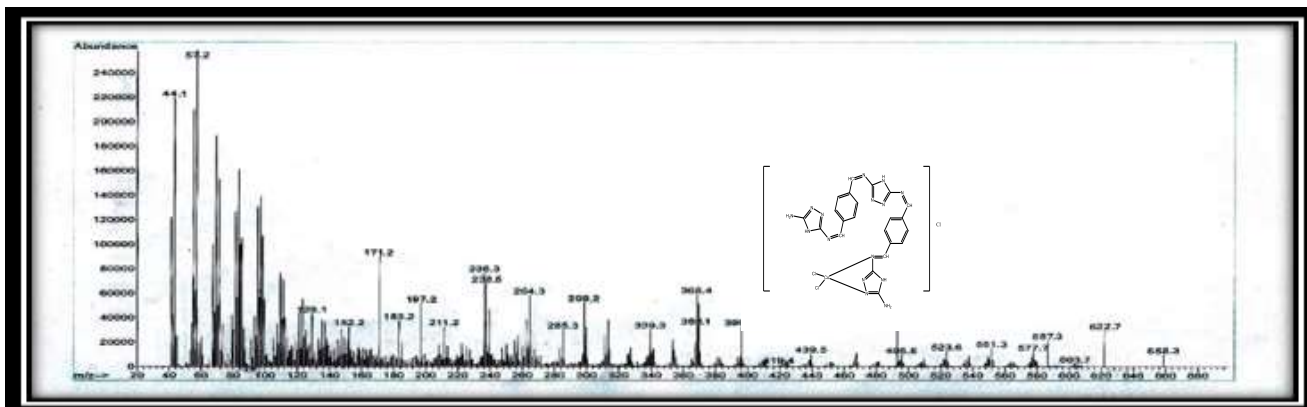
Figure 9: Mass Spectrum of [Co (L) Cl₂] Cl
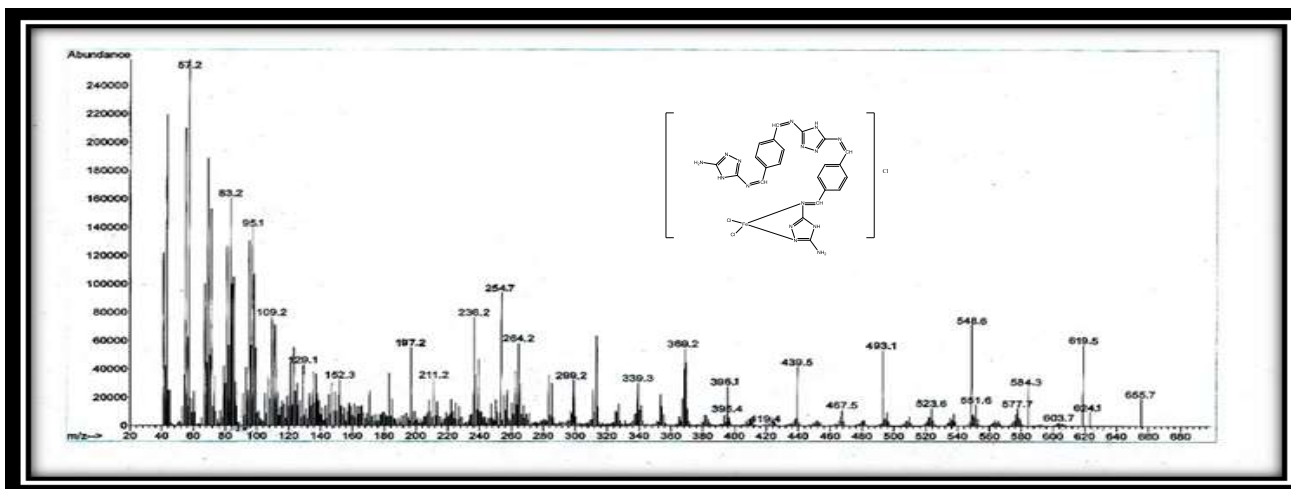
Figure 10: Mass Spectrum of [Fe (L) Cl₂] Cl
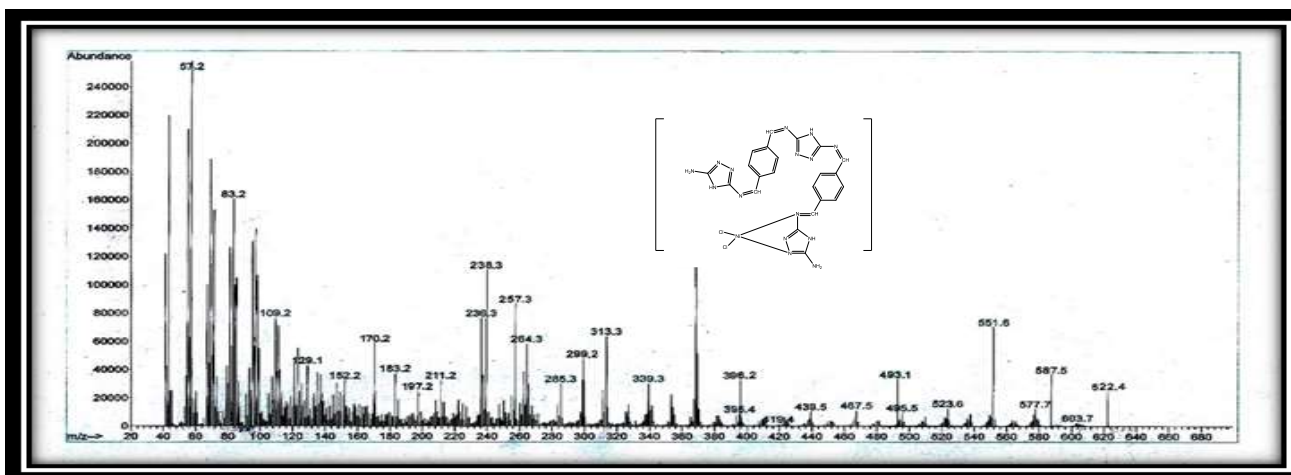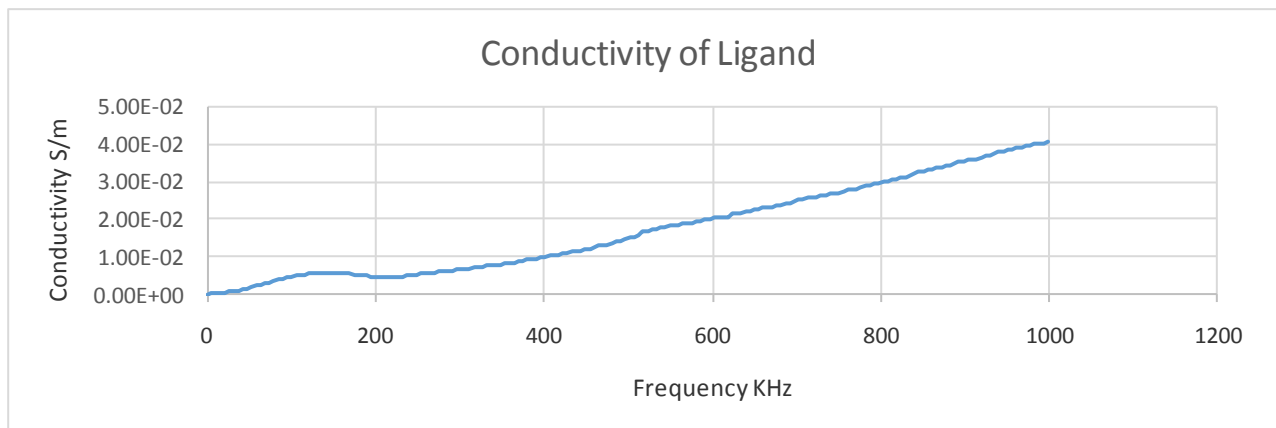

Figure 11: Mass Spectrum of [Ni (L) Cl₂]

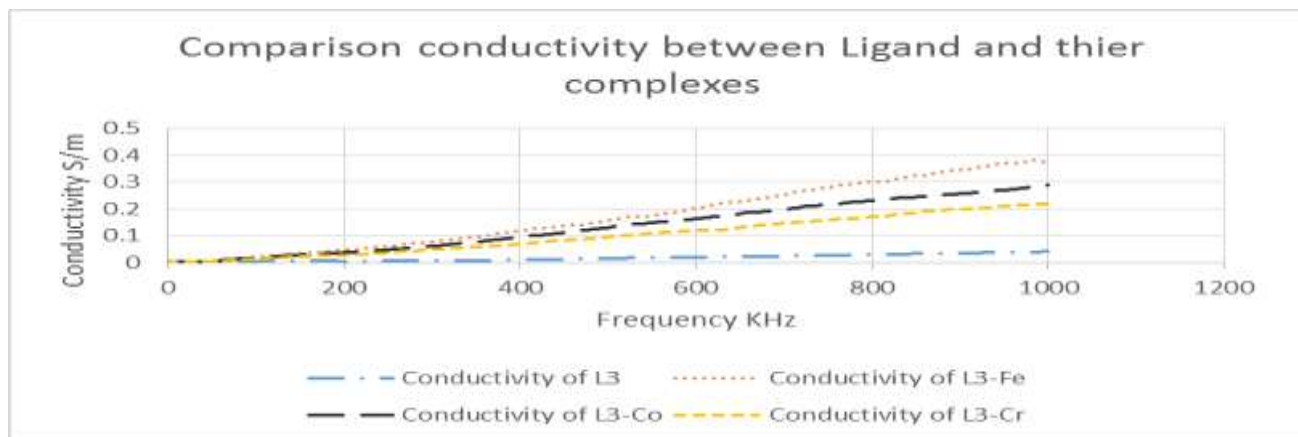**Figure 12: relationship between conductivity for L and frequency**

Figure 13: comparison conductivity between L and their complexes
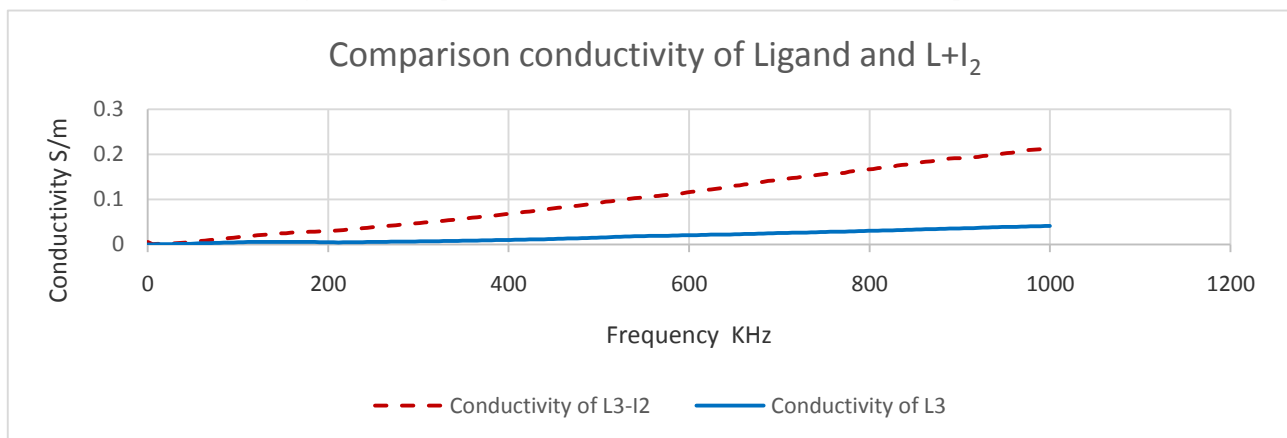
Figure 14: comparison conductivity of Land L-I₂
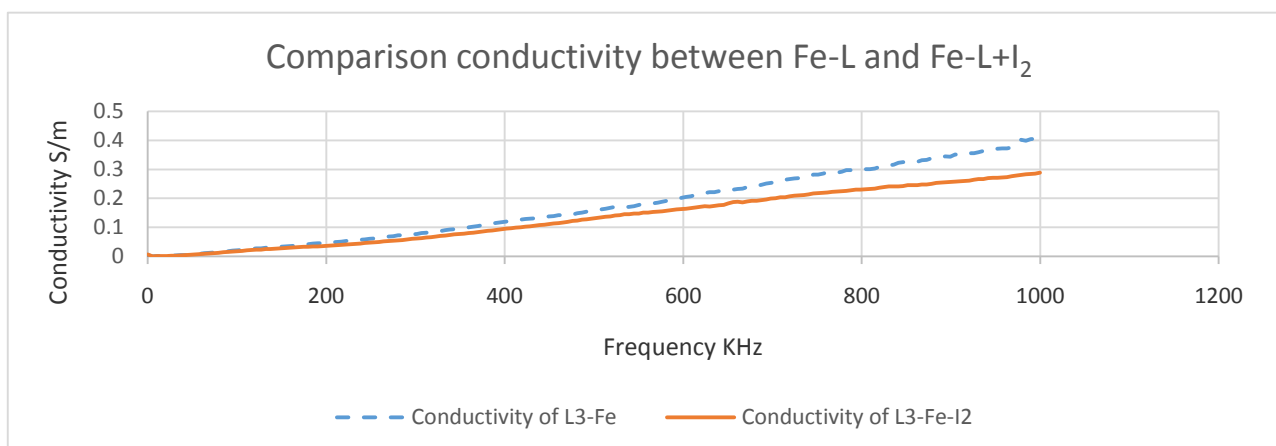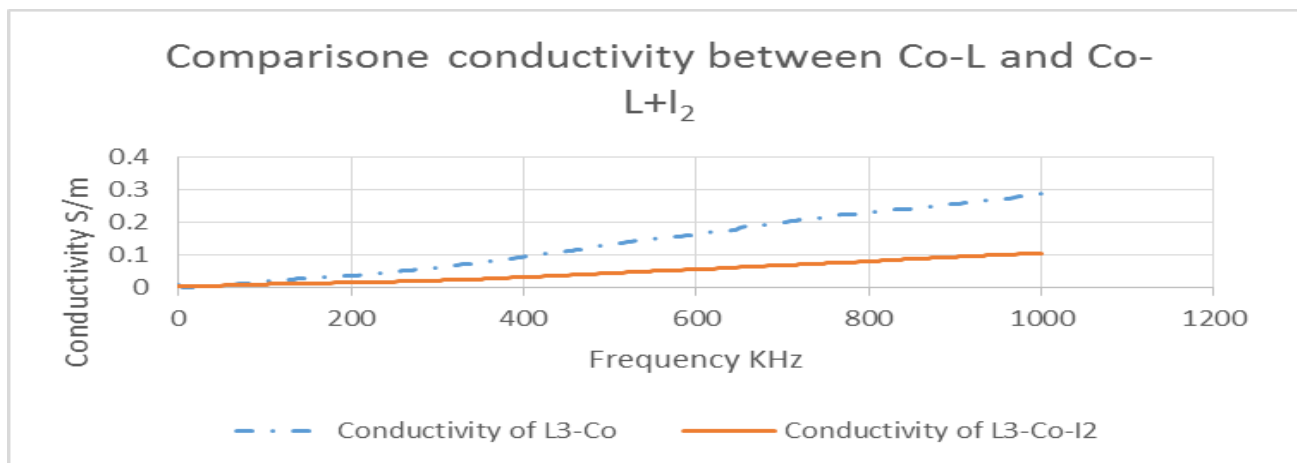

Figure 15: comparison conductivity between Fe-L and Fe-L-I₂

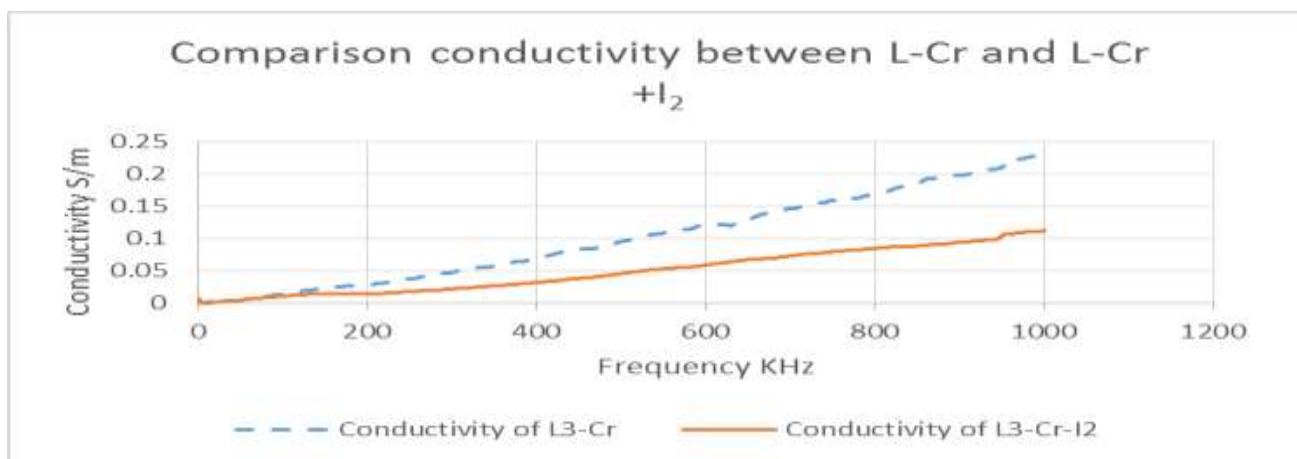**Figure 16: comparison conductivity between Co-L and Co-L-I$_2$**

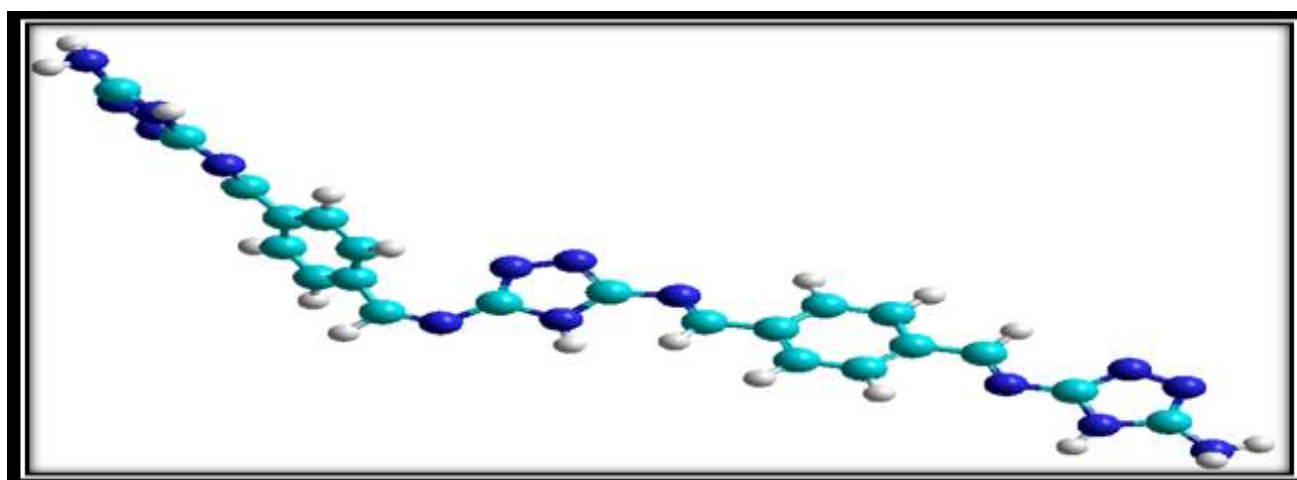**Figure 17: comparison conductivity between Cr-L and Cr-L-I$_2$**

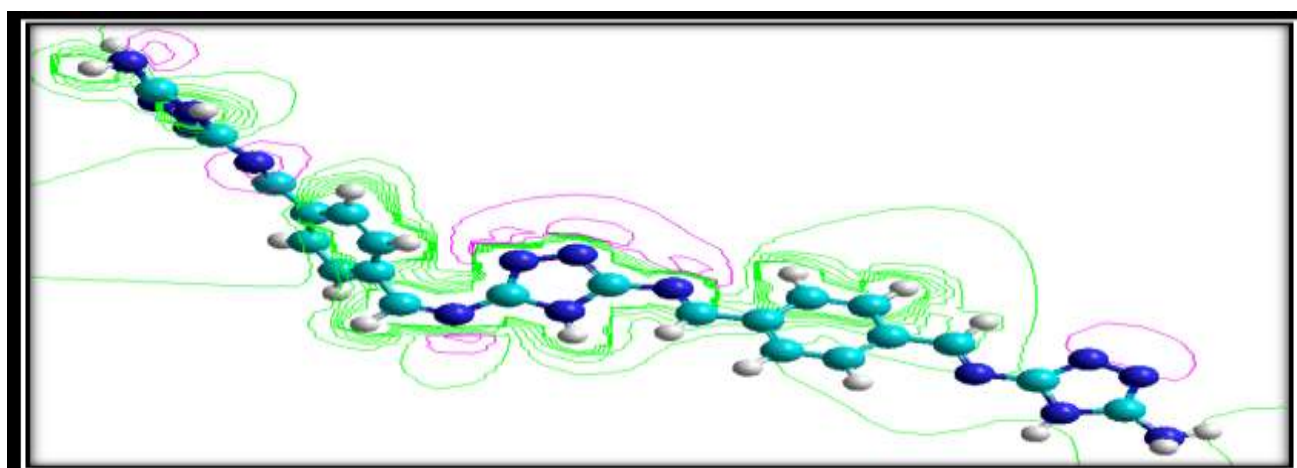**Figure 18: Ligand (C$_{22}$H$_{19}$N$_{15}$)**

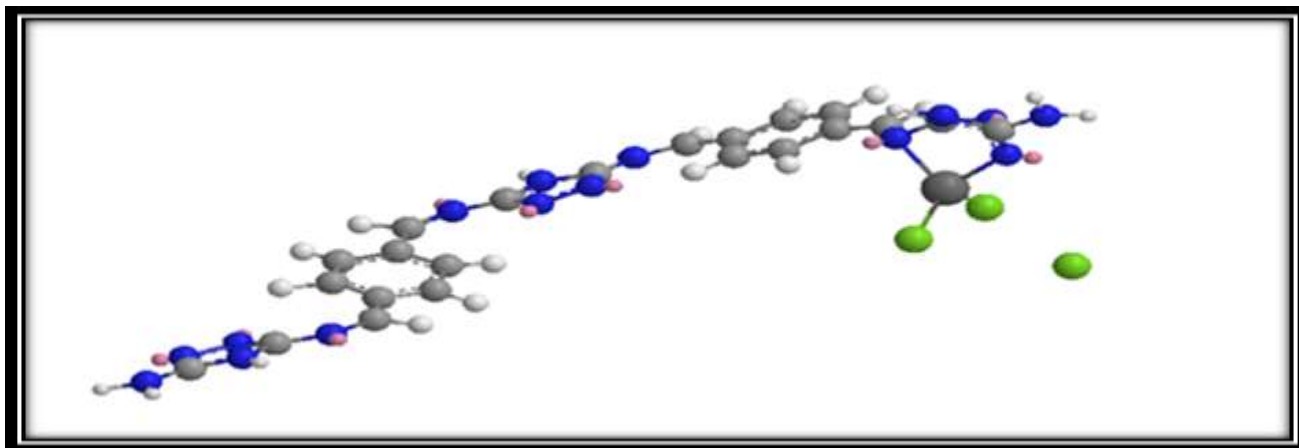**Figure 19: Electrostatic potential for (L)**

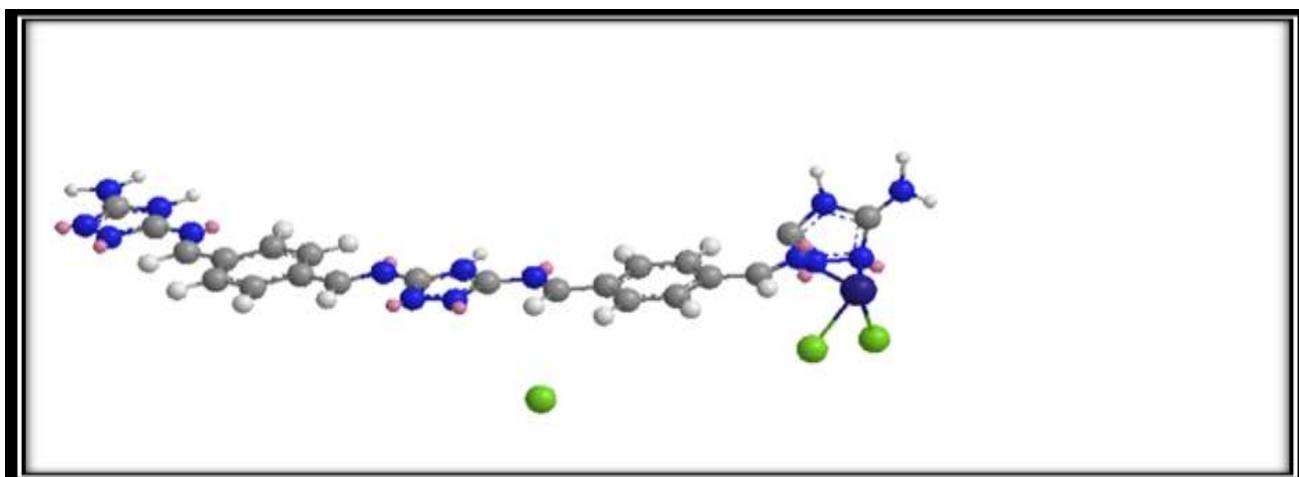**Figure 20: [Cr (L) Cl₂] Cl**
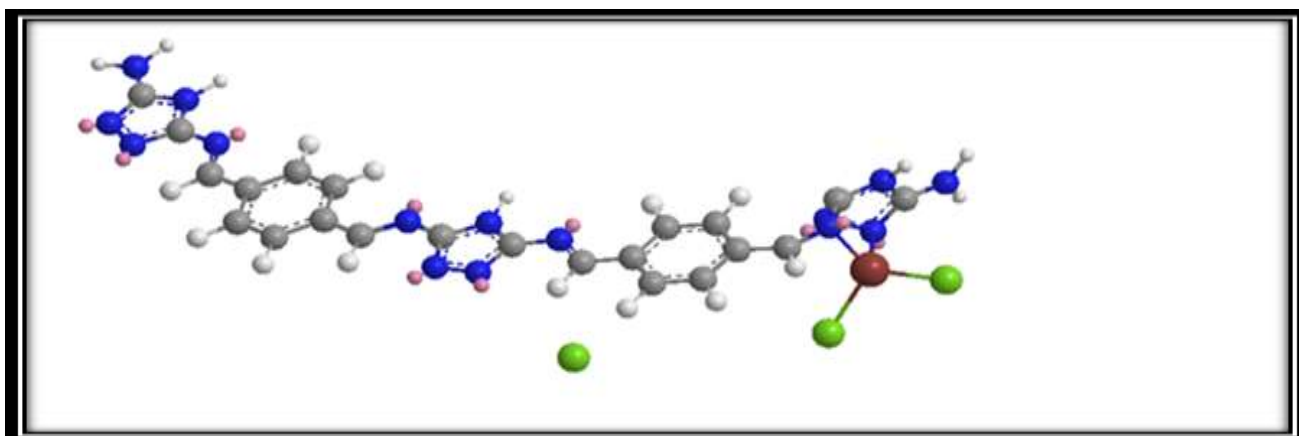

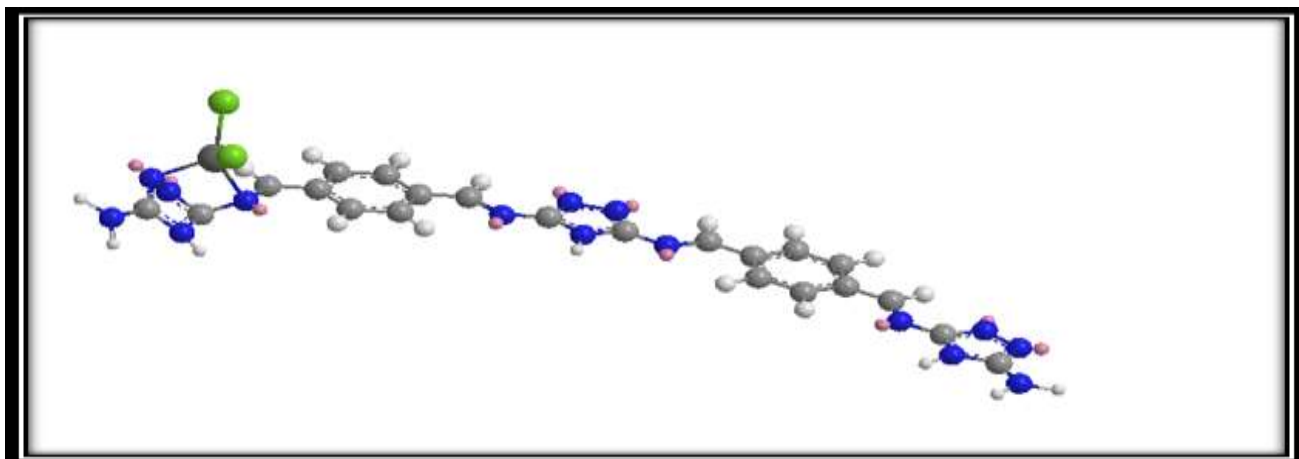**Figure 21: [Co (L) Cl₂]Cl**

Figure 22: [Fe (L) Cl₂] Cl

Figure 23: [Ni (L) Cl₂]

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