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Synthesis ,characterization and antibacterial study 0f new 2-ethyl-5-{[(3-phenyl-5- sulfanyl-4H-1,2,4-triazol-4-yl)imino]methyl}benzene-1,4-diol and their transition metal complexes

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Abstract

Transition metal complexes of new legend 2-ethyl-5-{[(3-phenyl-5- sulfanyl-4H-1,2,4-triazol-4-yl)imino]methyl}benzene-1,4-diol such as Cr^{3+},Fe^{3+}, Co^{3+}, Ni^{2+},Cu^{2+} were synthesis ,characterization by magnetic susceptibility measurements, conductance , elemental analyses, 1HNMR , IR , and mass spectra. The electrolytic behavior were confirmed from their conductance data. spectral study of transition metal complexes suggest octahedral geometry for Cr^{3+},$Fe^{3+}$and Co^{3+} ion , square planer geometry for $Ni^{2+}$and Cu^{2+} ion. The complexes and ligand were tested against two types of bacteria (Staphylococcus aureus, Escherichia coli).all prepared complexes showed good biological activity.

Keywords: - Triazole, Transitions metal complexes, biological activity.

1- Introduction

Introduction1. 1,2,4 triazole which is six –electron aromatic system of five –member heterocyclic ring.1,2,4 triazole and its derivatives have one of the most promising ligand ,and show with transition metal ion widely applied in luminescence ,magnetic and ion precursor properties[1-3].The derivatives of 1,2,4 –triazole has attractive whispered attention due to biological activity such as antimicrobial ,anti-inflammatory ,analgesic , antitumor, [4-10],anti HIV [11] , anti-cancer [12-13] and materials field [14]. The ability of transition metals to assume a wide range of oxidation states and coordination geometries provides unique opportunities for catalysis .1,2,4- triazole derivatives play excellent role as anti-corrosion iron [15]. The effectiveness of these compounds as corrosion inhibitors mainly depends on its functional groups, steric effects, electronic density of donor atoms, and p-orbital character of donating electrons [16-19]. Complexes of 1, 2, 4-triazoles are being developed for potential use such as magnetic material and photo chemical chemically driven molecular devices [20].

2.Experimental

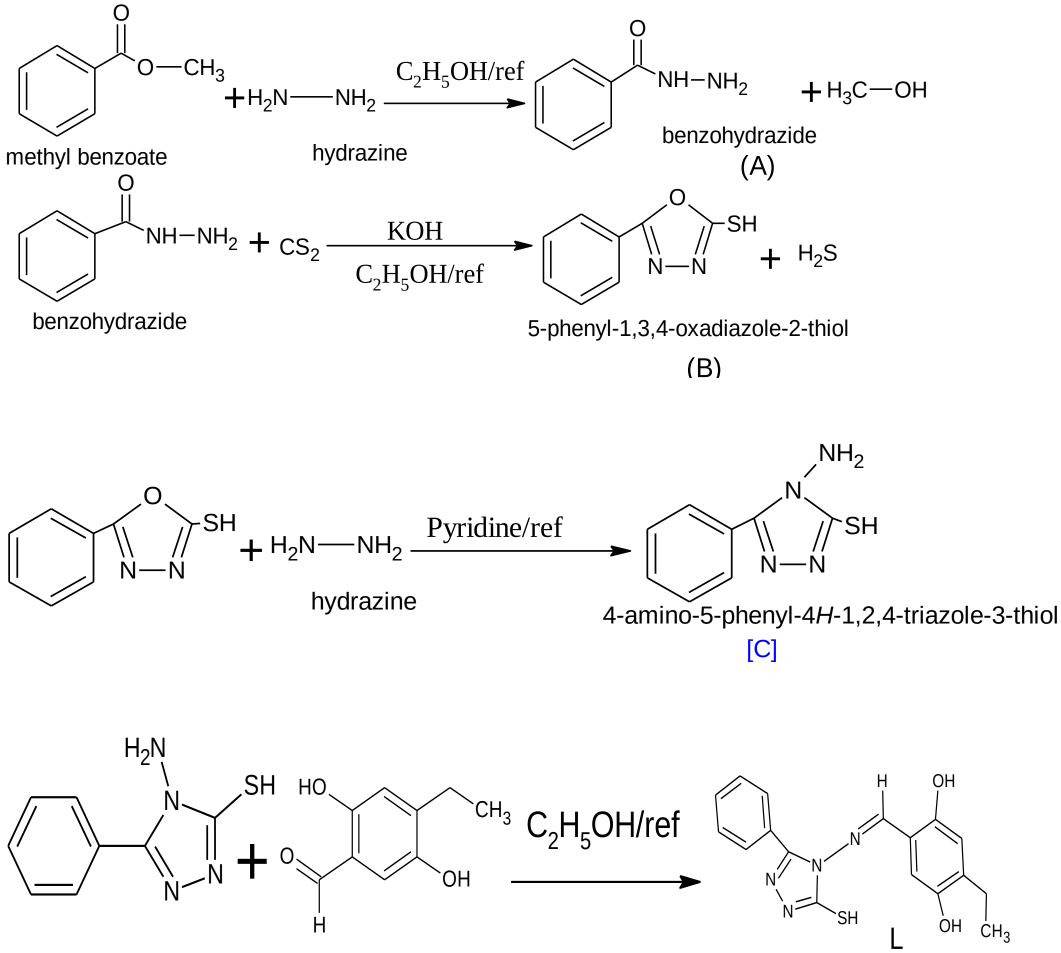
2.1 Preparation of the ligand

Novel ligand 2-ethyl-5-{[(3-phenyl-5-sulfanyl-4H-1,2,4-triazol-4-yl)imino]methyl}benzene-1,4-diol (Scheme 1) was prepared as follows:-

A mixture of (13.6 g, 0.1mol) of methyl benzoate and (10ml, 0.2mol) of hydrazine was dissolved in (100ml) ethanol. The resulting mixture was heated under reflux for (6hours).The resultant mixture was concentrated , The crude product was filtered(size of 6 µm) and washed with ethanol to give the desired product benzo hydrazide(A) as White needles, yield(91%), Melting point(m.p.) (138-140) °C [21,22] Amixture(A)(13.6 gm .1mol) and (5.6,g.1mol)of Potassium Hydroxide dissolved in(100 ml) absolute ethanol of this solution (7.5 ml, 0.1mol) of carbon disulfide was added. The resulting mixture was mixing by shaking and heated at (80) °C under reflux for (20hours).The resultant mixture was concentrated , and carefully acidified with hydrochloric acid HCl(5%) to give paleWhite precipitate. The crude product was filtered and washed with cold water rand ethanol to give the desired product 5-phenyl-1,3,4-oxadiazole-2-thiol (B), as White solid, yield(90.5%),m.p.
(220-222) ° C[21,22].

Amixture(B) (17.8gm .0.1 mol,) and access of hydrazine (0.15mol, 7ml) dissolved in Pyridine (100ml) was refluxed on a water bath (80) °C for(20) hrs, . The resultant mixture was concentrated , and carefully acidified with hydrochloric acid(5%) HCl to give pale light Brown precipitate. The crude product was filtered and washed with cold water, recrystallized from ethanol to give the desired product(4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol)(C)as Brown crystal ,yield(90%), m.p.(189-191)°C.

Amixture(C) (0.01mol,1.92gm) and (4-ethyl-2,5-dihydroxybenzaldehyde) (0.01mol,2ml) dissolved in (50ml) ethanol, And was refluxed for (3hrs) the solution is evaporated to half and failed cooling output when nominated and returned crystallized using absolute ethanol to give crystals of Light brown color of the output [ligand]. Yield: (73 %), m.p. (202-204)°C.(scheme1)



Scheme (1)

2.2 Preparation of complexes

The metal complexes were obtained by reflex(1 mmol) of transition metal chloride (1mmol)Cr(III), Co(III), Fe(III), Ni(II) and Cu(II) in 50 ml ethanol with the ligand (1mmol, 0.2 gm) for 2 hrs. The resultant solids which separated were filtered, washed with ethanol and dried in air.

2.3 Measurements

Elemental C, H and N analysis were carried out on a Thermofinigan flash analyzer, the FTIR spectra in the range (200-4000) cm^{-1} were recorded as CsI discs using a Shimadzu FTIR spectrophotometer, molar conductance measurements were made in anhydrous DMSO at 25 ^{o}C using Inolabcond

720.The ^{1}H NMR spectra were recorded on a Mercury-300BB NMR 300 spectrometer, relative to the internal standard tetramethylsilane (TMS) , DOSO-d6 used as solvent. Melting points were determined in open capillary tubes using an electro thermal melting point /SMP3 apparatus. Mass spectra were recorded in the range (0-800) m/e on a 5973 network mass selective detector. Balance Magnetic susceptibility.

3. RESULTS & DISCUSSION

The purity of the ligand and its complexes were checked by TLC using silica gel-G as adsorbent., elemental analysis tabulated in Table (1), Melting point, physical properties and molar conductance of all the compounds studied are tabulated in Table (2). The calculated values were in a good agreement with the experimental values.

Table 1.Elemental analysis for the ligand

Experimental			Theoretical		
C%	H%	N%	C%	H%	N%
60.08	4.72	16.61	59.98	4.74	16.46

Table 2. conductance, physical properties data of the ligand and its complexes

No	Compound	Molecular formula	Color	Λ Scm$_2$mol^{-1}	Melting Point °C	μeff B.M.
1	Ligand	$C_{17}H_{16}N_4SO_2$	Light brown	-------	202-204	-
2	$[Cr(L)_2Cl_2]Cl$	$Cr(C_{17}H_{16}N_4SO_2)_2Cl_3$	yellow	34	228-230	3.9
3	$[Fe(L)_2Cl_2]Cl$	$Fe(C_{17}H_{16}N_4SO_2)_2Cl_3$	Dark brown	36	221-223	2.3
4	$[Co(L)_2Cl_2]Cl$	$Co(C_{17}H_{16}N_4SO_2)_2$	Dark Green	31	210d*	0.42

		Cl_3				
5	$[Ni(L)Cl_2]$	$Cu(C_{17}H_{16}N_4SO_2)Cl_2$	Light yellow	18	188-191	0.16
6	$[Cu(L)Cl_2]$	$Ni(C_{17}H_{16}N_4SO_2)Cl_2$	Dark brown	15.6	192-194	1.8

d*=decomposition

3.1 magnetic susptibility :

The value of The effective magnetic moment of the complexes is tabulated in table[2] by using Balance Magnetic susceptibility .from the Results are shown in the table above, all the complexes given low value of effective magnetic moment. chromium ion Cr^{+3} shows the highest value(3.9)M.B due to the presence of three single electrons compare the low value(0.42) for nickel ion Ni^{+2} because there is no single electron for nickel ion Ni^{+2} . we conclude that the ligand works as strong ligand (strong field).

3.2 Infra-Red Spectroscopy

Infrared spectroscopy is one of the most commonly used tools for the detection of functional groups in pure compounds and mixtures. The spectra for L shows a characteristic stretching absorption bands at (3115,2740 ,1633,2940 and 1404) cm^{-1} assigned to υ(C-H.Ar.) ,υ(S–H) [23] ,υ (C=N),υ(C-H) , and symmetrical C-N-C stretching respectively [24]. The C=N and S-H stretching are important to predict the bonding mode of the ligand ,these bands shift higher wavenumber in the spectra of complexes compare with ligand, observed changes are the evidences of complexation had happened . The IR data of the ligand and complexes are shown in Table (3) and figure(8),(9) and(10) . The Table lists the stretching frequency (υ) for some of the characteristics groups exhibited by the ligand and complexes.

Table 3: Characteristic absorption bands of ligand and its complexes

NO	**υO_H**	**υC – H.alf**	**υC – H.ar**	**υSH**	**υC =N**	**υ C-N-C**	**Stru. Moveme**	**υM -N**	**υM- S**	**υM- Cl**

			o				nt			
L	3302	2940	3115	2740	1633	1477	1076	_	_	_
1	3310	2933	3120	2788	1639	1494	1072	686	401	308
2	3303	2941	3116	2830	1635	1479	1074	688	402	343
3	3300	2977	3133	2814	1637	1492	1074	680	433	312
4	3292	2945	3122	2801	1620	1489	1078	692	398	300
5	3304	2970	3128	2744	1637	1499	1074	688	405	341

3.3 Nuclear Magnetic Resonance

The 1H NMR spectral data for the 2-ethyl-5-{[(3-phenyl-5- sulfanyl-4H-1,2,4-triazol-4- yl) imino]methyl}benzene-1,4-diol displayed good solubility in DMSO. The proton nuclear magnetic resonance spectral data gave additional support for the suggested structure of the ligand. The spectra exhibit a triplet at (1.6)ppm due to methyl group, another singlet peaks exhibit at (2.5)ppm due to CH_2 , The 1H NMR spectrums of ligand shows multiplets peak were observed around (7.5-8.06)are due to phenyl protons .the spectra exhibit also beaks at(3.8) , ,(8.9) and (9.95) due to SH[25], C-H.Ar , C=N-H and OH respectively[26,27]. the proton NMR of the ligand shown in figure(11) .

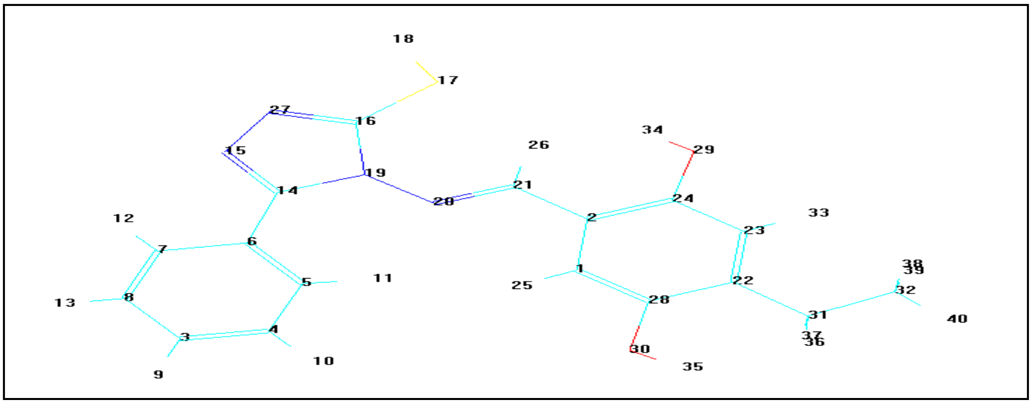
3.4 Mass spectra :

The mass spectra of the ligand and its transition metal complex were recorded at room temperature ,The mass spectrum of the ligand shows a molecular ion peak [M0] at m/z =340, The fragmentation pathways of ligand give the peaks at different mass numbers at m/z = (295, 281,267,252,238,175,165 ,143, 117,103,99,66, and 65)due to $[C_{16}H_{15}N_4O_2]^{+\cdot}$, $[C_{16}H_{15}N_3O_2]^{+\cdot}$, $[C_{16}H_{15}N_2O_2]^{+\cdot}$, $[C_{15}H_{12}N_2O_2]^{+\cdot}$, $[C_{14}H_{10}N_2O_2]^{+\cdot}$, $[C_8H_5N_3S]^{+\cdot}$, $[C_9H_{11}NO_2]^{+\cdot}$ $[C_8H_5N_3]^{+\cdot}$, $[C_7H_5N_2]^{+\cdot}$, $[C_7H_5N]^{+\cdot}$, $[C_2HN_3S]^{+\cdot}$, $[C_2N_3]^{+\cdot}$ $[C_5H_5]^{+\cdot}$ Respectively. The intensity of these peaks reflects the stability and abundance of the ions. It is also supported by the mass spectra of the other complexes. as shown in Figure(12-17)and(Scheme 2). the complex $[Cr(L)Cl_2]Cl$ showed a molecular ion peak at m/z [M0]= (840)which is equivalent to molecular mass of the complex. This complex shows another a fragmentation peak with loss of chlorine atom at m/z(804). the complex spectrum shows fragment ion peak with loss two chlorine atom at m/z (768,733) due to$[Cr(L)_2Cl]^{+\cdot}$ and $[Cr(L)_2]^{+\cdot}$ respectively.

The mass spectrum of the complex $[Fe(L)_2Cl_2]Cl$ shows a molecular ion peak at m/z [M0] (843)which is equivalent to molecular mass of the complex. This complex shows another a fragment ion peak with loss of chlorine atom at m/z(808). the complex spectrum shows fragment ion peak with loss two chlorine atom at m/z (772, 737) due to $[Fe(L)_2Cl]^{+\cdot}$and $[Fe(L)_2]^{+\cdot}$ respectively.

The mass spectrum of the complex $[Co(L)_2Cl_2]Cl$ shows a molecular ion peak at m/z [M0] =(845)which is equivalent to molecular mass of the complex. This complex shows another a fragment ion peak with loss of chlorine atom at m/z(811). the complex spectrum shows fragment ion peak with loss two chlorine atom at m/z (775, 740) due to $[Co(L)_2Cl]^{+\cdot}$and $[Co(L)_2]^{+\cdot}$ respectively.

The mass spectrum of the complex $[NiLCl_2]$ shows a molecular ion peak at m/z [M0](470), This complex shows another a fragment ion peak with loss of chlorine atom at m/z (435)and (399). The mass spectrum of the complex $[CuLCl_2]$ shows a molecular ion peak at m/z [M0] (475), This complex shows another a fragment ion peak with loss of chlorine atom at m/z (439)and (404).the mass spectra of the complexes shown in figure(13),(14),(15)(16)(17).



Figure(1):The stereochemistry of the Ligand

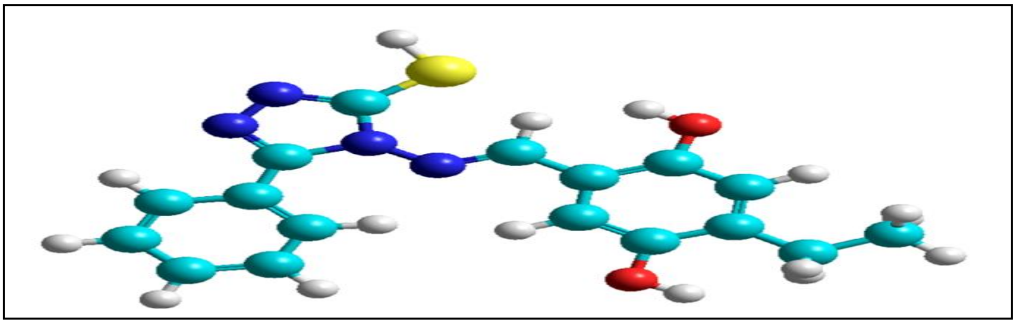


Figure (2): Graphical presentation of stereochemistry of the Ligand ($C_{16}H_{14}N_4SO$)

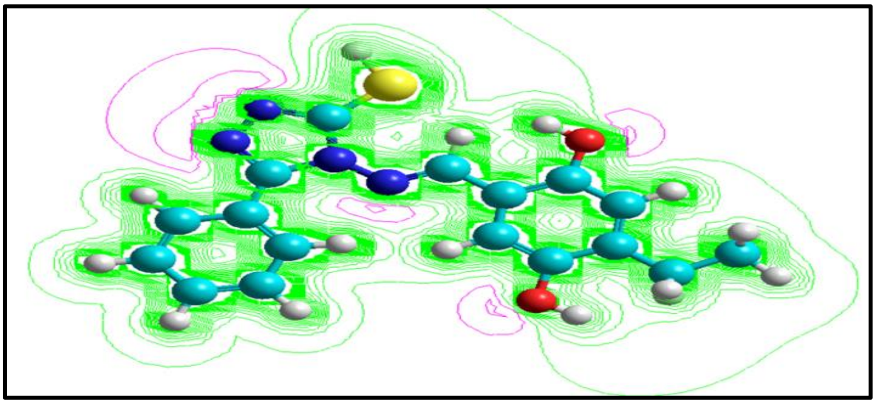


Figure (3): Electrostatic potential 2D for Ligand

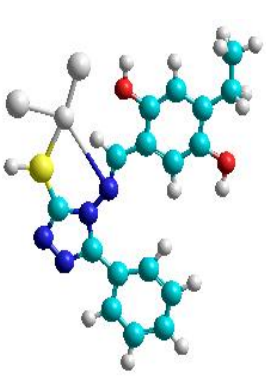


Figure 4:$[Ni(L)Cl_2]$

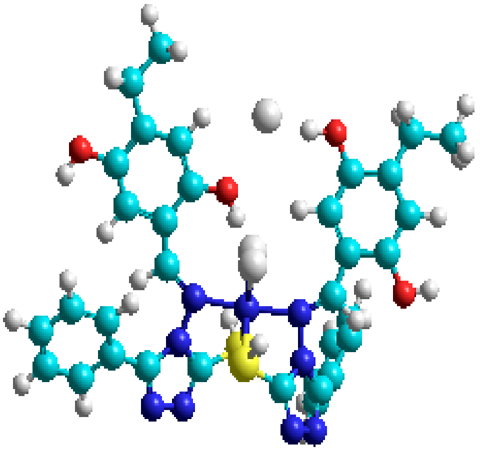


Figure 5:$[Co(L)_2Cl_2]Cl$

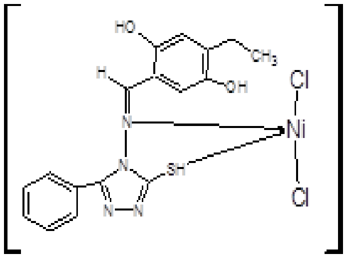


Figure 6

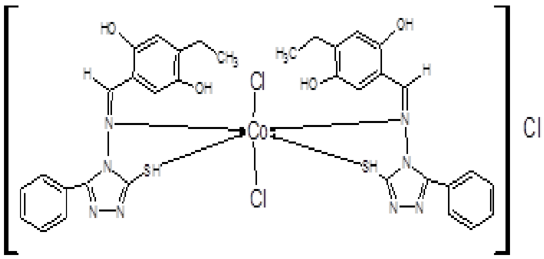


Figure 7

Analytical and spectral data (^{1}H NMR,IR, mass spectra) of all synthesized compounds were in full agreement with the proposed structure.

3-4 – Biological Study

The ligand and its transition metal complexes were evaluated for antimicrobial activity against gram positive bacteria such as Staphylococcus aureus and gram negative bacteria Escherichia coli, by using agar well diffusion method. All the microbial cultures were adjusted to 0.5 McFarland standard, dimethyl sulphoxide (DMSO) were used to prepared all the test solution .The area of inhibition was measured in millimeter. nutrient agar used as culture medium[28] , the values of the investigated compounds are tabulated in Table.4.The observe result showed transition metal complexes enhanced antimicrobial activity than that of free ligand. This result can be due to the greater lipophilic nature of the complexes and favors its permeation through the lipoid layers of the bacterial membranes. The activity of transition metal complexes can be expound on the basis of Overton's concept [29] and Chelation theory [30]

Table 4 . Antibacterial screening data of the ligand and its metal complexes

Compound	Escherichia coli Inhibition zone(mm)	Staphylococcus Aurens Inhibition zone(mm)
L= $C_{17}H_{16}N_4SO_2$	10++	15+++
$[Cr(L)_2Cl_2]Cl$	18++++	15+++
$[Fe(L)_2Cl_2]Cl$	20+++++	10++
$[Co(L)_2Cl_2]Cl$	18++++	10++
$[Ni(L)Cl_2]$	13+++	10++
[Cu(L)CL2]	15+++	10++

****(+ = Weak Inhibition , ++ =Medium Inhibition , +++ = Good Inhibition ,++++ =Very good**

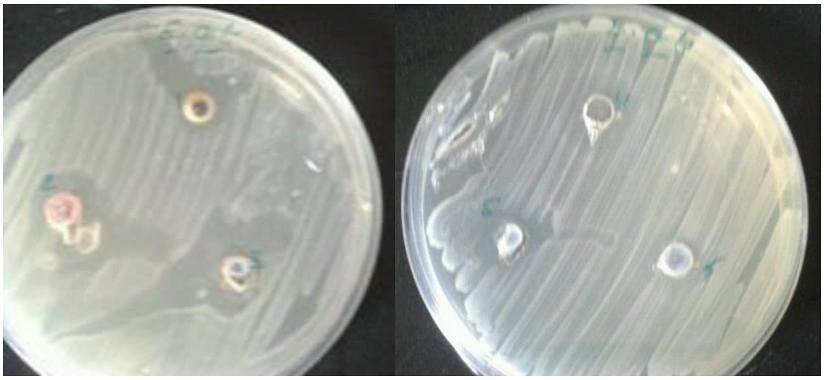
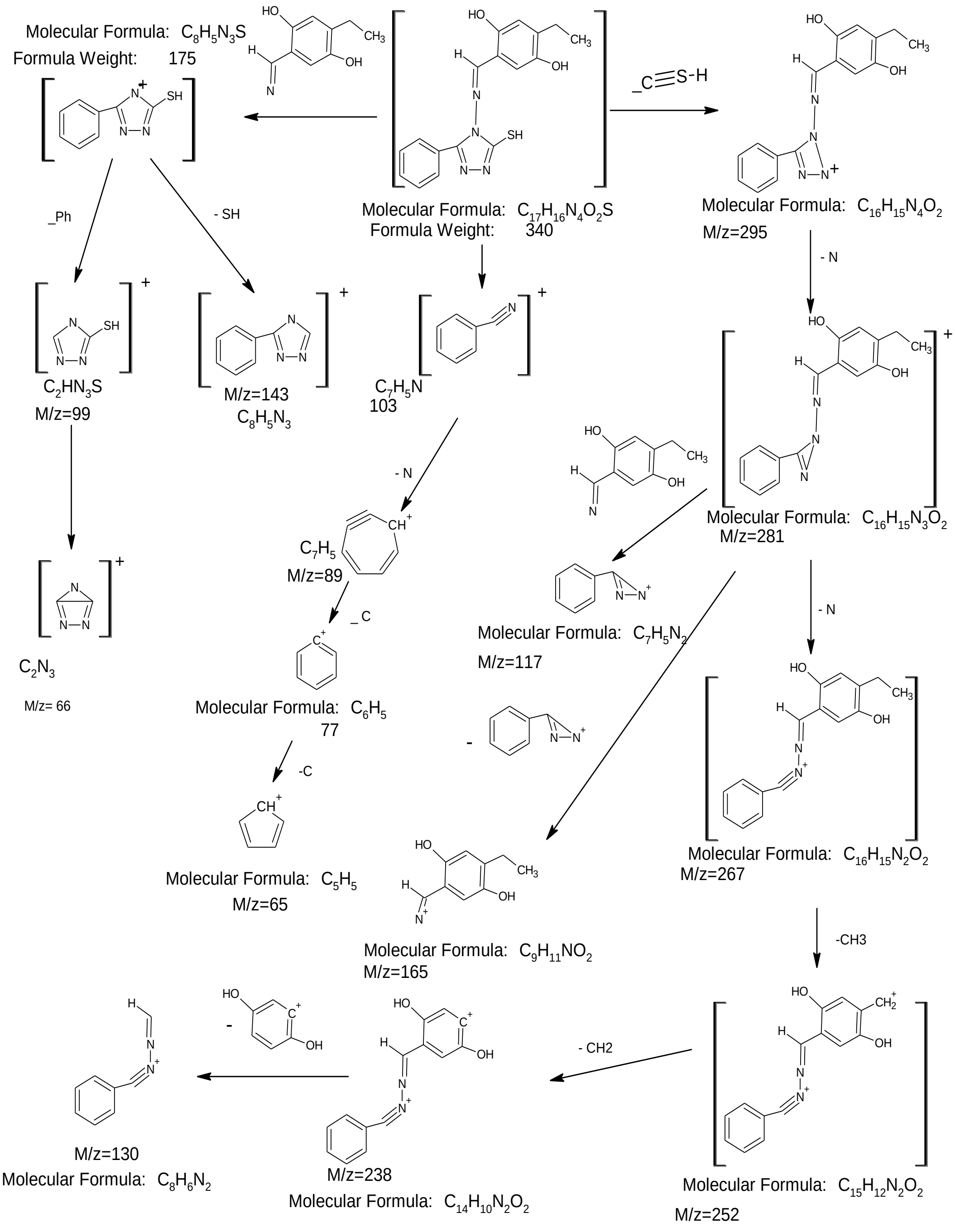


Figure (8) biological study

4. Conclusion:

The ligand4-{[(*Z*)-(2-methoxyphenyl) methylidene] amino}-5-phenyl-4*H*-1,2,4- triazole -3-thiol. was successfully synthesized. The elemental analysis, , IR, ^{1}H NMR and mass spectral observations suggest the octahedral geometry for the Cr(III), Co(III),Fe(III). tetrahedral geometry was proposed for Ni(II),Cu(II) .


Molecular Formula: $C_8H_5N_3S$
Formula Weight: 175
Molecular Formula: $C_{17}H_{16}N_4O_2S$
Formula Weight: 340
_C≡S-H
Molecular Formula: $C_{16}H_{15}N_4O_2$
M/z=295
_Ph
- SH
- N
C_2HN_3S
M/z=99
M/z=143
$C_8H_5N_3$
C_7H_5N
103
Molecular Formula: $C_{16}H_{15}N_3O_2$
M/z=281
- N
C_7H_5
M/z=89
_ C
C_2N_3
M/z= 66
Molecular Formula: $C_7H_5N_2$
M/z=117
- N
Molecular Formula: C_6H_5
77
-C
Molecular Formula: $C_{16}H_{15}N_2O_2$
M/z=267
Molecular Formula: C_5H_5
M/z=65
Molecular Formula: $C_9H_{11}NO_2$
M/z=165
-CH3
- CH2
M/z=130
Molecular Formula: $C_8H_6N_2$
M/z=238
Molecular Formula: $C_{14}H_{10}N_2O_2$
Molecular Formula: $C_{15}H_{12}N_2O_2$
M/z=252

Scheme2

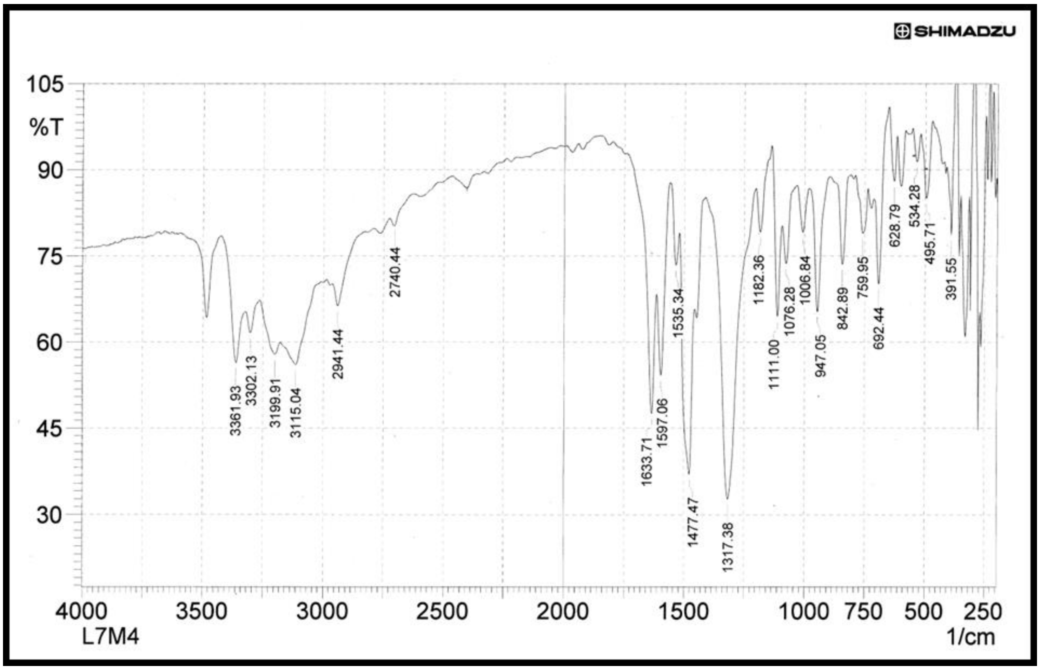


Figure 8: IR spectra of Ligand ($C_{17}H_{16}N_4SO_2$)

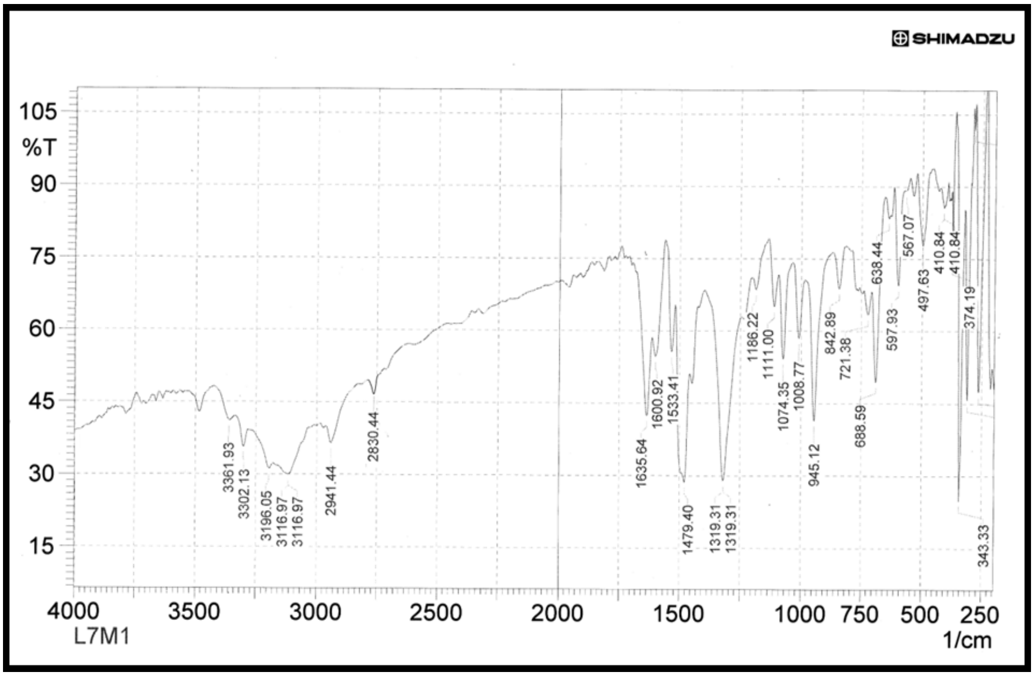


Figure 9: IR spectra of [$Fe(L)_2Cl_2$]Cl

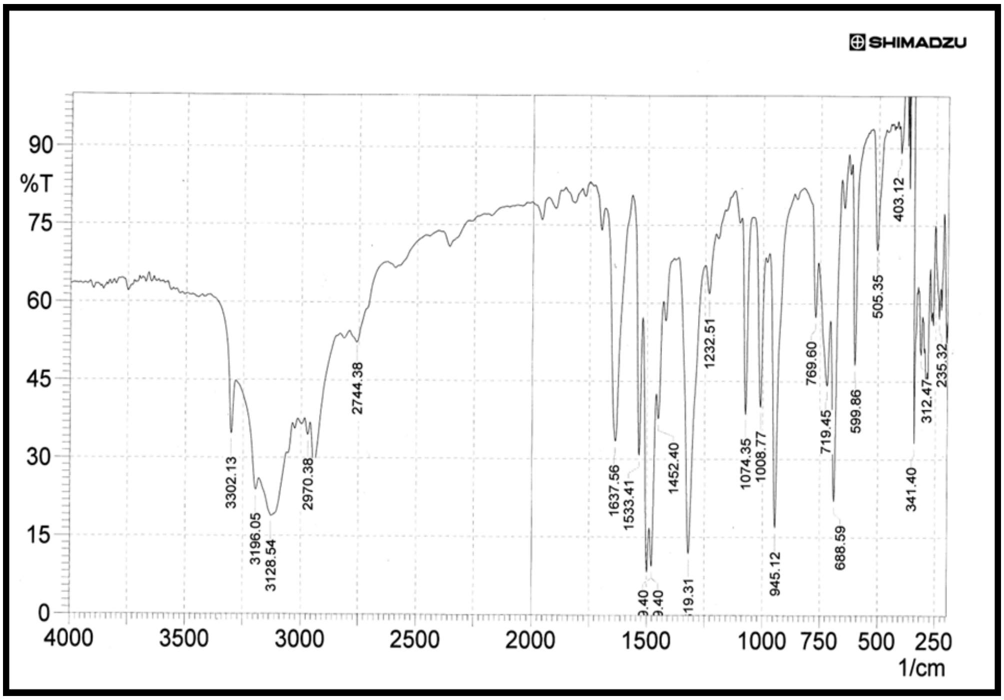


Figure 10: IR spectra of $[Cu(L)_2Cl_2]$

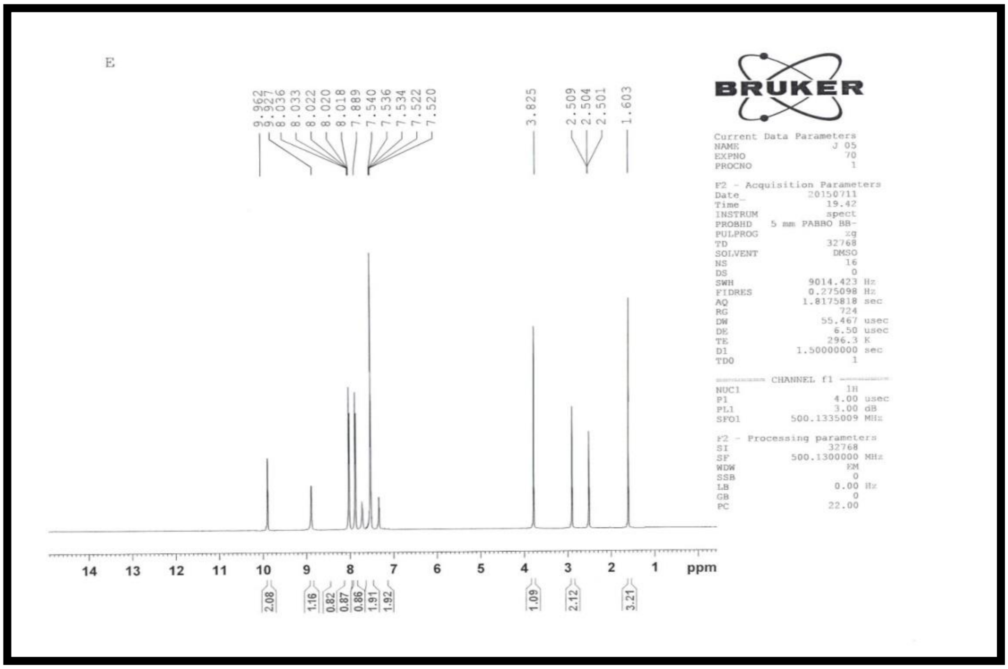


Figure 11 : NMR spectra of the ligand($C_{17}H_{16}N_4SO_2$)

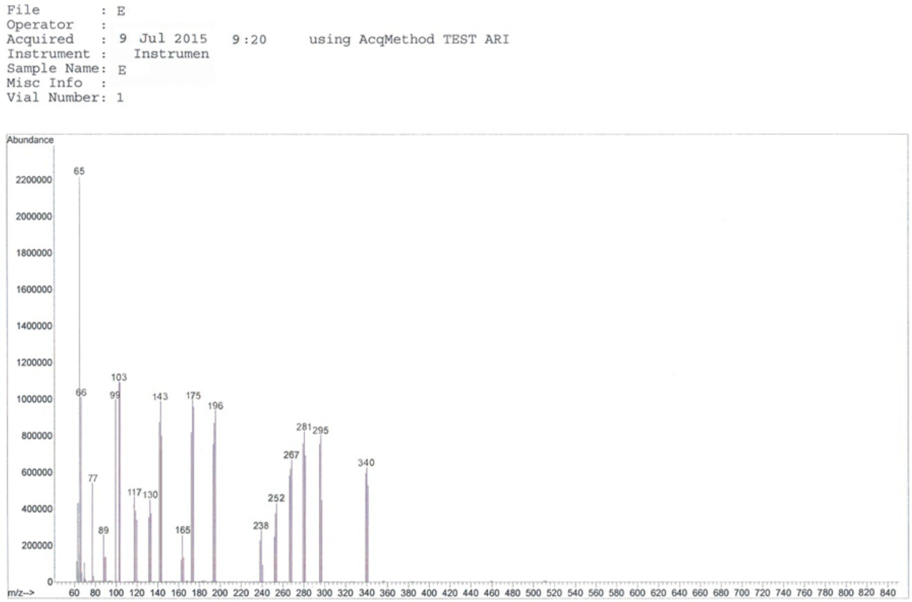


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Figure 12: mass spectra of ligand

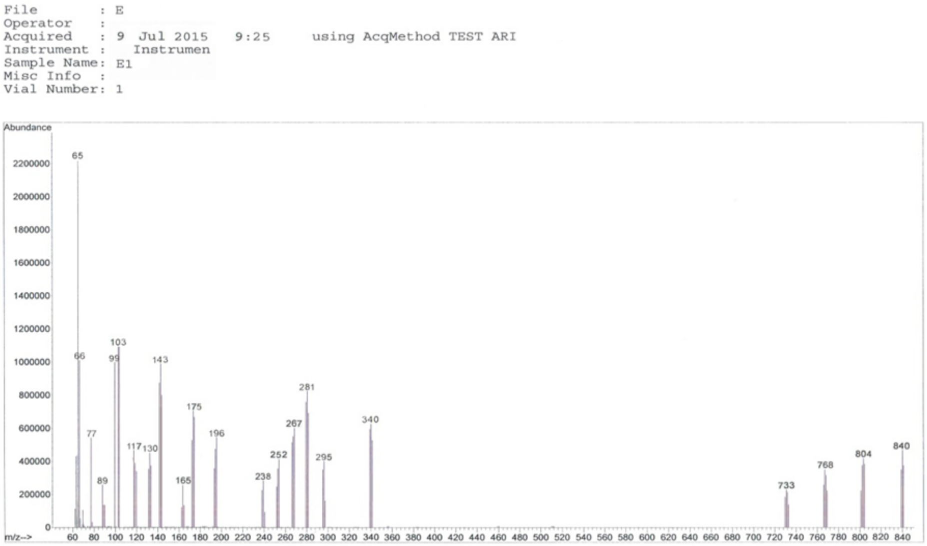

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Figure 13: mass spectra of $[Cr(L)_2Cl_2]Cl$

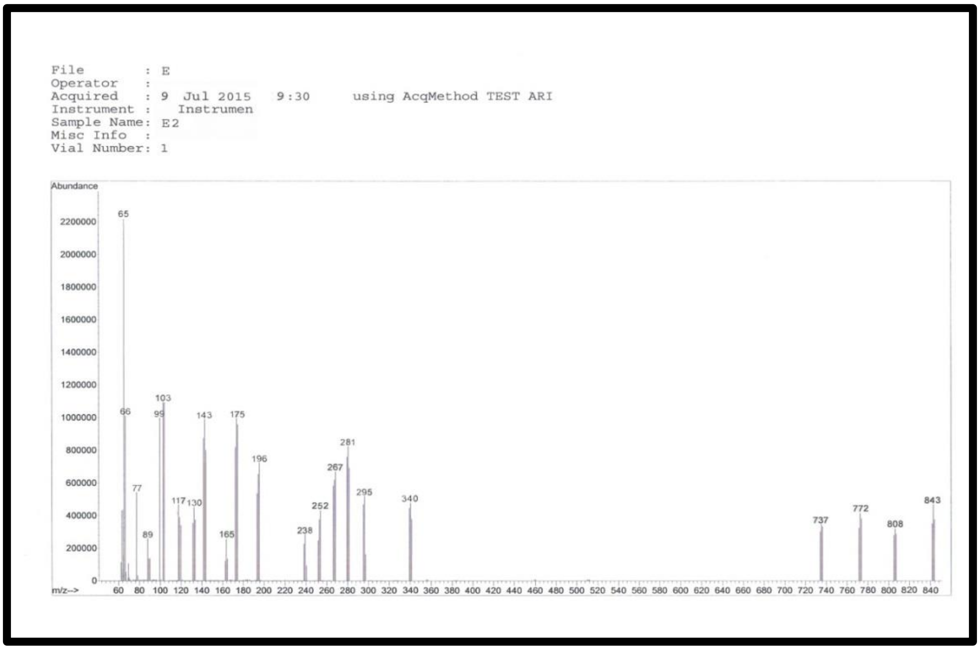

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Figure 14: mass spectra of $[Fe(L)_2Cl_2]Cl$

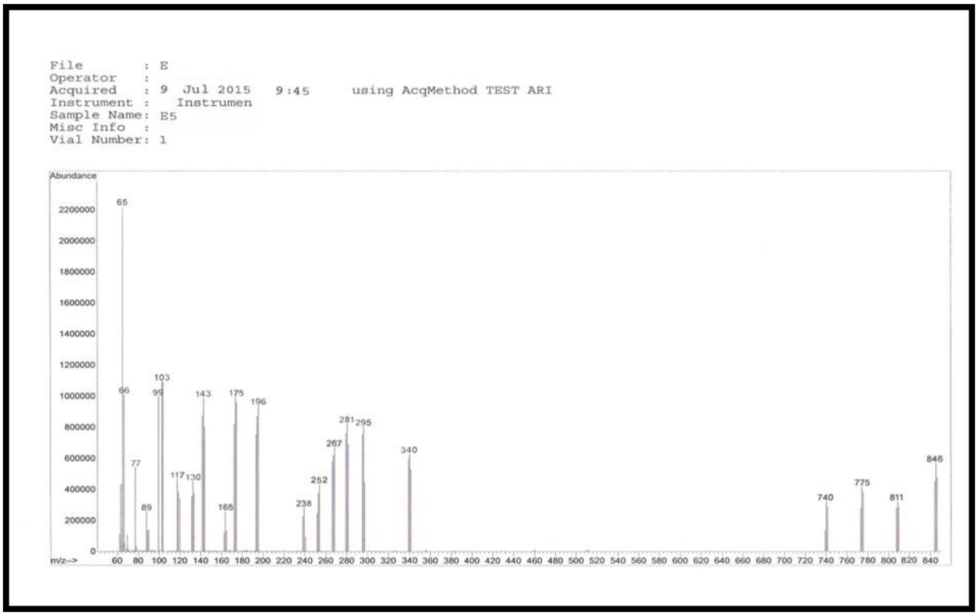

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Figure 15: mass spectra of $[Co(L)_2Cl_2]Cl$

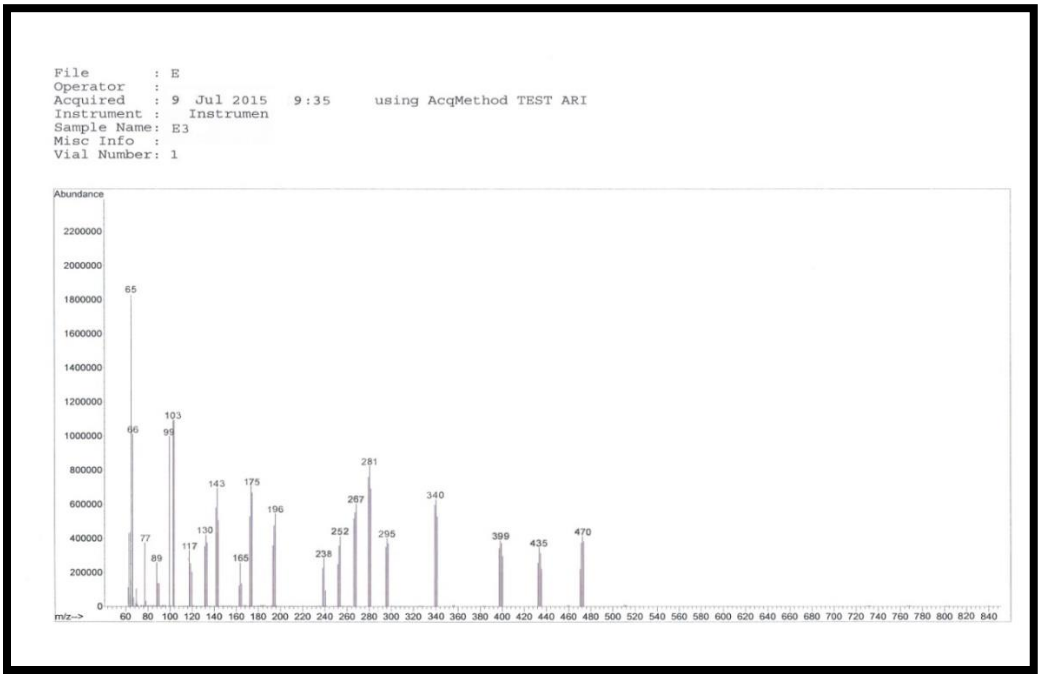

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Figure 16: mass spectra of [$Ni(L)Cl_2$]

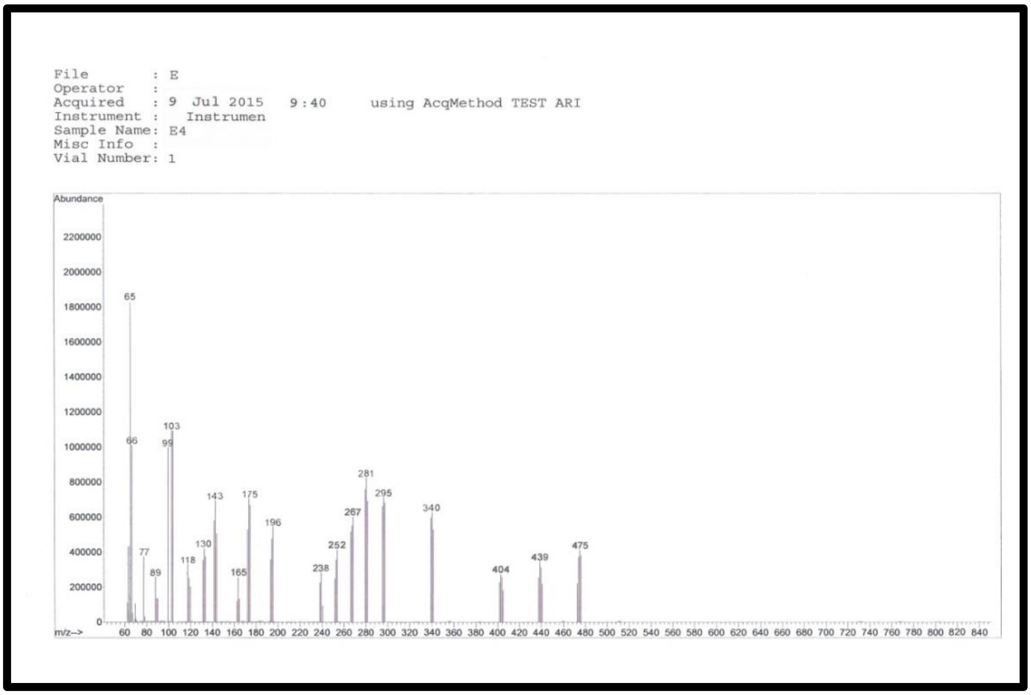

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Figure 17: mass spectra of [Cu(L) Cl_2]

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تحضير وتشخيص ودراسة الفعالية البايولوجية لليكاند مع بعض معقدات العناصر الانتقالية

2-ethyl-5-{[(3-phenyl-5-sulfanyl-4H-1,2,4-triazol-4-yl)imino]methyl}benzene-1,4-diol

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الخلاصة

تم تحضير سلسلة جديدة من معقدات العناصر الانتقالية(Cu(II) و Cr(III),Fe(III),Co(III),Ni(II)مع الليكاند 2-ethyl-5-{[(3-phenyl-5-sulfanyl-4H-1,2,4-triazol-4-yl)imino]methyl}benzene-1,4-diol تم تشخيص الليكاندات والمعقدات المحضرة باستخدام تحليل العناصر الدقيق ,التوصيلية المولارية, اطياف الاشعة تحت الحمراء, وبروتون الرنين النووي المغناطيسي وطيف الكتلة والحساسية المغناطيسية. السلوك الالكتروليتي للمعقدات تم تاكيده باستخدام تقنية التوصيل المولاري .وتم الاستنتاج بان الليكاند يتناسق من خلال ذرتي النتروجين والكبريت كما موضح في الجدول (3) , في جميع المعقدات وجد ان الليكاند يعمل كليكاند ثنائي السن ويتناسق من خلال [S] و [N] وهذه الرؤية تضيف دعما اخر من خلال ظهور حزمة تقابل حزمة امتصاص M-N بحدود cm^{-1} (680-692).

من النتائج التي تم الحصول عليها اقترح الشكل مربع مستوي للـNi(II) ورباعي السطوح (Cu(II)بينما لمعقدات Cr(III),Fe(III),Co(III) شخصت على انها ثمانية السطوح.

وتم اختبار الفعالية البايولوجية لليكاند المحضر ومعقداته باستخدام طريقة الانتشار وذلك بقياس منطقة التثبيط وباستخدام مذيب ثنائي مثيل اوكسيد الكبريت (DMSO) , وأعطت اختبارات الفعالية نتائج ايجابية حيث لوحظ ظهور تثبيط جيد ضد بكتريا E-coli و Staphylococcus .