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Synthesis, Characterization, Antimicrobial and Anticorrosion of Studies New 2-(5-(2-(5-hydrazinyl-1,3,4-thiadiazol-2-yl) hydrazinyl)-1,3,4-oxadiazol-2-yl) Phenol With Some Transition Metal Ions

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ABSTRACT

A new ligand2-(5-(2-(5-hydrazinyl-1,3,4-thiadiazol-2-yl) hydrazinyl)-1,3,4-oxadiazol-2-yl)phenol and its Cu(II) , Co(III) and Ni(II) complexes were synthesized . the new ligand and its complexes have been characterization on the basis their spectra of ^{1}HNMR, mass, Fourier transform infrared (FTIR), as well as magnetic susceptibility, atomic absorption, elemental analysis [C, H, N, S] and conductance measurements. The program of Hyperchem 7.51 have been used up for theoretical accounts using PM3 method [1] to study the electrostatic potential that Provided good information about the complexity site. A ligand and its complexes were tested for their antibacterial activity against two kinds of strains Escherichia coli (gram negative bacterial strains) and staphylococcus aureus (gram positive bacteria strains), This research showed excellent results in comparison with Ciprofloxacin as standard drug. The ligand was tested as a corrosion inhibitor for carbon steel in 1 M HCl solution. The corrosion inhibition efficiency was determined by using the weight loss methods

Keywords: ligand, complexes, characterization, Hyperchem, electrostatic potential, antibacterial

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INTRODUCTION

Heterocyclic compounds are the cyclic organic compounds which contain at least one heteroatom, the most common heteroatoms are the nitrogen, oxygen, and sulfur but heterocyclic rings containing other heteroatoms are also widely known [1]. The chemistry of heterocyclic compounds is an interesting field of study since a long time. There are two classes of heterocyclic very important include oxadiazole and thiadiazols, the Oxadiazole is a cyclic compound having one oxygen and two nitrogen atoms in a five-member ring [2]. The Oxadiazoles subsist in different isomeric forms such as 1,2,5-oxadiazoles , 1,2,4-oxadiazoles , 1,2,3-oxadiazoles and 1,3,4-oxadiazoles [3]


1,3,4-oxadiazole


1,2,4-oxadiazole


1,2,3-oxadiazole


1,2,5-oxadiazole

Oxadiazoles have occupied a specific place in the field of medicinal chemistry due to its wide range of activities[4]. From the existing literature we can see that 1,3,4-Oxadiazole nucleus has been possessing antimicrobial[5], antifungal[6], anti-inflammatory[7], anticonvulsant[8], antioxidant, analgesic[9], antitubercular[10], mutagenic activity[11], antioxidant[12] , corrosion inhibitor[13] . Thiadiazole is a heterocyclic compound featuring both two nitrogen atom and one sulfur atom as part of the aromatic five-membered ring , it was found that among the important pharmacophores responsible for various activities [14]. All review showed that the thiadiazole nuclei have antibacterial and antifungal, anti tubercular, anticonvulsant anti-leishmanial activities[15].

EXPERIMENTAL

Synthesis of 2-hydroxy benzohydrazide

A mixture of methyl benzoate (15.2ml, 0.1mol) and hydrazine monohydrate (7.5ml , 0.15mol) in ethanol absolute (25 ml) were refluxed for 6 hours , the mixture was evaporated to half volume , cooled , filtered and washed with ethanol absolute[16]. the solid was lighting white ,melting point 150 ^oC , yield 95%.

Synthesis of 2-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol

2-hydroxybenzohydrazide (15.2 gm , 0.1mol) , (5.6g, 0.1mol) of Potassium Hydroxide and carbon disulfide (7.6ml,0.1mol) were refluxed in ethanol absolute (50ml) .the solvent was evaporated and acidified with HCl (10%) then the precipitated was filtered and the result solid was recrystallized from ethanol absolute [17]. the solid was yellow ,melting point 200 ^oC , yield 72%.

Synthesis of 1,3,4-thiadiazole-2,5-dithiol

A mixture of (80%) hydrazine hydrate (0.1 mol, 5g), carbon disulfide (0.2 mol, 15g) and KOH (0.2 mol, 11g) was refluxing for 25 hrs. The reaction was follow by TLC. Then the excess solvent was distilled off, and the resulting solid was separated out by adding (10%) of hydrochloric acid. The mixture was filtered and dark yellow solid was recrystallized from ethanol. m.p = (162-164) ◦C, yield=78 % .[18]

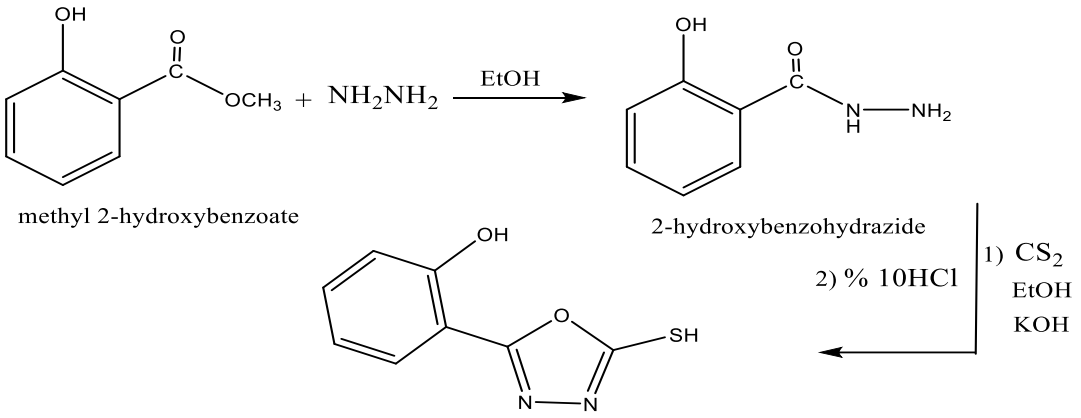
Synthesis 2,5-dihydrazinyl-1,3,4-thiadiazole

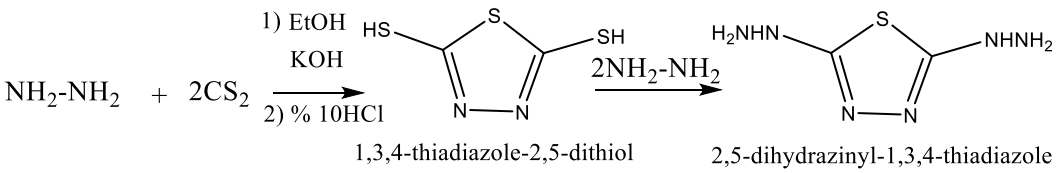
A mixture of compound (A1) (0.1 mol, 15g) in 50 ml of absolute ethanol and (80%) hydrazine hydrate (0.2 mol, 10g) was refluxing for 5 hrs. The reaction was following by TLC.

Then cooled to room temperature, poured in (100 ml) of ice water. The yellow solid result was filtered off, washed with water and recrystallized from ethanol, yield = 82 % [19].

Synthesis 2-(5-(2-(5-hydrazinyl-1,3,4-thiadiazol-2-yl)hydrazinyl)-1,3,4-oxadiazol-2-yl)phenol

The ligand was synthesized by condensation of 2,5-dihydrazinyl-1,3,4-thiadiazole (5gm , 0.034 mol) and 2-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol (6.65gm , 0.034mol) in 1:1 molar proportions in ethanol absolute (25ml) . then the mixture refluxed for 8 hours (monitored by TLC). the ligand was precipitated, filtered and recrystallized from ethanol absolute to get white ligand melting point 228-230 ^{o}C, yield 65%.





2.2. Preparation of complexes

The complexes were synthesized by mix (0.001mol) from ligand with salts ($CoCl_2.6H_2O$,$CuCl_2.6H2O$ and $NiCl_2.6H_2O$) both alone in (100ml) ethanol absolute and refluxed for 2 hrs. (monitored by TLC) .then the precipitate was filtered and wash several times with ethanol or aqueous ethanol to removed unreacted salts or ligand ,then precipitated complexes was dried[27].

2.3. Analysis and physical measurements

Physical properties and elemental microanalysis CHNS shown in table 1

Table 1: Physical properties, elemental microanalysis data of the ligand and its complexes

No	formula	Color	(C%)Ex. C% Cal.	(H%)Ex. H% Cal.	(N%)Ex. N% Cal.	(S%)Ex. S% Cal.	Atomic absor.	Λ Scm^2 mol-1	M.p °C	µeff B.M
1	$C_{10}H_{10}N_8O_2S$ (L)	brown	(39.51) 39.21	(3.78) 3.29	(36.12) 36.58	(10.11) 10.47		-----	200	-----
2	Cu(L)Cl_2	green					(15.91) 14.42	18	240	1.9
3	Co (L)Cl_2	Dark grey					(12.15) 13.51	22	270	4.8
4	Ni(L)Cl_2	yellow					(15.46) 13.46	12	262	0.48

DISCUSSION AND RESULT

3.1 FT-IR SPECTRAL

FT-IR of the synthesized ligand and its complexes were carried out using KBr disc to ligand and CsI for complexes. The free ligand (L) exhibited six major bands at (3448)cm^{-1}, (3348)cm^{-1}, (3026)cm^{-1}, (1625)cm^{-1}, (1527)cm^{-1}, (1442)cm^{-1}, (1327)cm^{-1}, (1273)cm^{-1} and (1081) [28]cm-1 Which are attributable to (υOH), (υNH), (υC-H)aro., (υC=N) , (υC=C), (υC-O-C)sym, (υ C-O-C)asy, (υC-S-C) and structure movement bands respectively. New bands were formed Attributed to the coordinated (M- N), and (M-Cl) bonds and appeared at the region (439-493) cm^{-1} and (262-316) cm-1 respectively. This indicates that the coordinate occurred through the (N), and (Cl) atoms. As shown in figures (14-17).

3.2 Nuclear Magnetic Resonance

The 1H-NMR spectra of the ligand showed signals at (11.82ppm, 1H) , (10.91ppm, 1H), (10.45ppm, 1H), (9.17ppm , 1H) and (3.17ppm , 1H), due to (N-H)c protons, (N-H)d protons, (O-H) protons, (N-H)b and (N-H_2)a respectively. signals at [(6.94-7.94)ppm , 4H] due to chemical shifts of aromatic ring protons linking the oxadiazole ring[21] as showed in the figure (9).

3.3 Mass spectra

The mass spectra of ligand appeared molecular ion peak at 306 m/z which is in conformity with the molecular formula $C_{10}H_{10}N_8O_2S$. Other peaks are due to the subsequent fragments like $[C_{10}H_8N_7O_2S]$+=290m/z, $[C_{10}H_9N_8OS]$+=289m/z, $[C_{10}H_7N_6O_2S]$+=275m/z , $[C_4H_5N_8OS]$+=213 m/z, $[C_8H_7N_4O_2]$+=191 m/z, $[C_8H_6N_3O_2]$+=176 m/z, $[C_2H_5N_6S]$+=145 m/z, $[C_2H_3N_4S]$+=115 m/z.

The mass spectral of the Cu(II) complexes showed molecular ion peaks at 440m/z corresponding to $[Cu(L)Cl_2]^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 405m/z, 369m/z, due to loss one and two chlorine atom respectively.

The mass spectral of the Co(II) complexes showed molecular ion peaks at 436m/z corresponding to $[Co(L)Cl_2]^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 400m/z, 365m/z due to loss one and two chlorine atom respectively. The mass spectral of the Ni(II) complexes showed molecular ion peaks at 435m/z corresponding to $[Ni(L)Cl_2]^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 400 m/z, 365m/z due to loss one and two chlorine atom respectively. as shown in figure (10-13)

3.4 magnetic susptibility

The magnetic momentum for each metal complexes is listed in table 1. these magnetic measurements give an idea about the electronic state of the transition metal ion of the complexes. The observed magnetic momentum value of Cu(II) complex was 1.9 BM , expected for Square planer geometry . the magnetic momentum value was 4.8 BM for Co(II) suggesting tetrahedral geometry . 0.48 BM for Ni(II) suggesting square planar geometry respectively[22] .

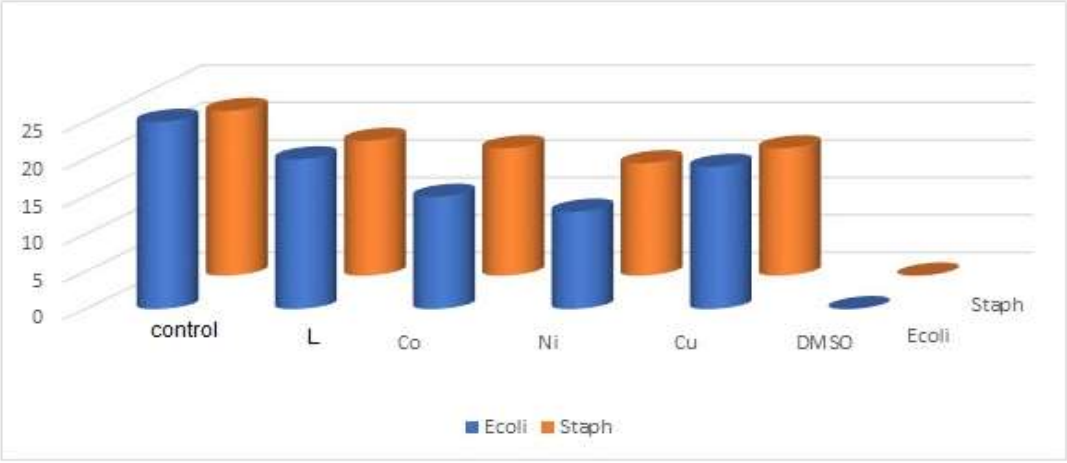
3.5 Biological Study

The antibacterial efficiency of ligand and its complexes were evaluated by using agar spread method. Two type of bacteria have been used, Gram Positive Bacteria as Staphylococcus Aureus and Gram-Negative Bacteria as Escherichia Coli (E. Coli), using Ampicillin as standard drug. The bacteria inhibition was calculated in millimeter. nutrient agar was used as culture medium. dimethyl Sulfoxide used as solvent. the concentration of all compounds in this solvent was 15mg/ml, using disc susceptibility test. This technique includes the exposure of the zone of inhibition toward the spread of bacteria on agar dish. The dishes were Put in the incubator for 24hr. at 37 °C [23]. From the observation of the results in table (2), Affirms that all compound shows good anti-bacterial activity

Table 2: Anti-bacterial data of ligand and its complexes

No.	compound	E. coli Inhibition zone (mm)	Staph. Inhibition zone (mm)
A	control	+++	+++
B	L	++	++
C	Co	+	+
D	Ni	+	+
E	Cu	+	+

+++= (20–30)mm highly active += (8-12) active ++= (13–19)mm more active,



Staph. Ecoli

Figure 1: Anti – bacterial of E.coli & Staph

3.6 Electrostatic potential (MEP). Molecular

Electrostatic potential is very important in finding the active site in the molecule system with a positive point charge. The species that have positive charge tend to attack a molecule where the electrostatic potential is strongly negative (electrophilic attack). Electrostatic potential of free ligand was measured and plotted as 2D contour to find the active site of molecule [24] as shown in figures [4-8].

3.7. Anticorrosion inhibitor

Study the effect of the prepared surfactant on the corrosion inhibition of carbon steel in 1 M HCl solution using weight loss methods. Steel specimens with dimensions of 2.5 cm × 5 cm × 0.5 cm were immersed in 1 M HCl in a closed beaker with and without the addition of different concentrations of inhibitor for (3-15) h at 30–60 °C. Triplicate specimens were exposed for each condition and the average weight losses were reported. The contents of. Steel specimens is shown in table (3)

Table 3: contents of. Steel specimens

Element	C	Si	Mn	P	S	Ni	Cr	Al	V	Ti	Cu	Fe
Weight (%)	0.19	0.05	0.94	0.009	0.004	0.014	0.009	0.034	0.016	0.003	0.022	Rest

The corrosion rate (k) was calculated from the following equation [25]:

$$K = \Delta W / St \quad mg/cm^2.h \qquad (1)$$

where ΔW is the average weight loss of three parallel steel sheets, S is the total area of the specimen and t is the immersion time. The corrosion inhibition efficiencies, IE% and the surfactant area were calculated according to the following equation [26]:

$$IE\% = (CR_{uninh}-CR_{inh}/ CR_{uninh}) * 100 \qquad (2)$$

$$\theta = (CR_{uninh}-CR_{inh}/ CR_{uninh}) \qquad (3)$$

Where IE% = inhibition efficiency, CR_{uninh}= Corrosion Rate without inhibitor

CR_{inh}= Corrosion Rate with inhibitor

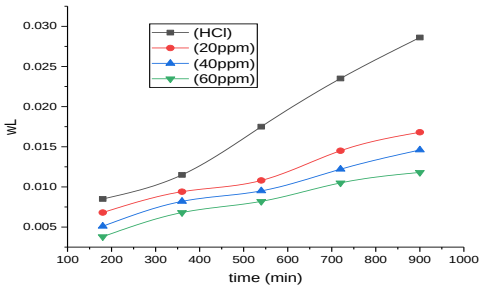
Weight loss data of carbon steel in 1 M HCl in the absence and presence of various concentrations of inhibitor are listed in Table 2 Data show that, the corrosion inhibition efficiency for the synthesized inhibitor increases with increasing the inhibitor concentration as shown in figure2. The influence of solution temperature on the inhibition efficiency was studied at 30, 40,50 and 60 °C. It was observed that the inhibition efficiency decreases with increasing temperature from 30 to 60 °C as shown in figure3. The reduction in inhibition efficiency with temperature may be attributed to desorption of the inhibitor molecules from the metal surface at higher temperatures.

A large number of organic compounds containing nitrogen, oxygen or sulphur have been used as inhibitors to control acid corrosion of iron and steel [27]. Compounds rich in heteroatoms can be regarded as environmentally friendly inhibitors because of their characteristic strong chemical activity and low toxicity [28]. The adsorption characteristics of organic molecules are also affected by sizes, electron density at the donor atoms and orbital character of donating electrons [29].

Table 4: Weight loss data of carbon steel corrosion in 1 M HCl in the absence and presence of different concentrations of the synthesized inhibitor at 30 °C.

Conc.	time	3h	6h	9h	12h	15h
(1M) HCl	ΔW(g)	0.0085	0.0115	0.0175	0.0235	0.0286
	Rc $mgcm^{-2}h^{-1}$	0.227	0.153	0.156	0.157	0.152
20 ppm	ΔW(g)	0.0068	0.0094	0.0108	0.0145	0.0168
	Rc $mgcm^{-2}h^{-1}$	0.181	0.125	0.096	0.097	0.089
	%IE	20	18	38	38.2	41
	(θ)	0.20	0.18	0.38	0.382	0.41

40 ppm	ΔW(g)	0.0051	0.0082	0.0095	0.0122	0.0146
	Rc $mgcm^{-2}h^{-1}$	0.136	0.109	0.084	0.081	0.078
	%IE	40	29	46	48	49
	(θ)	0.40	0.29	0.46	0.48	0.49
60 ppm	ΔW(g)	0.0038	0.0068	0.0082	0.0105	0.0118
	Rc $mgcm^{-2}h^{-1}$	0.101	0.091	0.073	0.07	0.063
	%IE	56	41	53	55	59
	(θ)	0.56	.41	0.53	0.55	.59



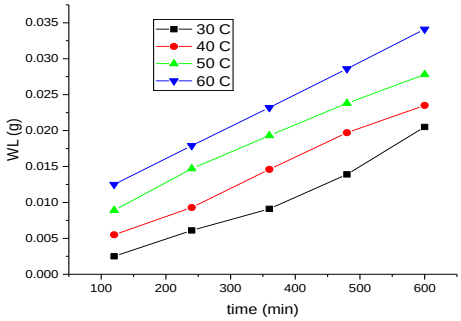


Figure 2: anticorrosion inhibitor of carbon steel in 1M HCl with different concentration of ligand at 30 ºC

Figure 3: anticorrosion inhibitor of carbon steel in 1M HCl with 20 ppm concentration of ligand at different temperature

CONCLUSION

The 1,3,4-oxdiazole derivative acts as a bidentate ligand. The spectroscopic data exhibit the involvement of NH and the azomethane of heterocyclic groups in coordination to the central transition metal ion. susceptibility magnetic technique has been used to characterization of transition metal complexes. a square plainer geometry for Ni(II) and Cu(II), tetrahedral geometry for Co(II) complex is proposed. The results of the electrostatic potential study were quite consistent with the practical results of the complexity sites.

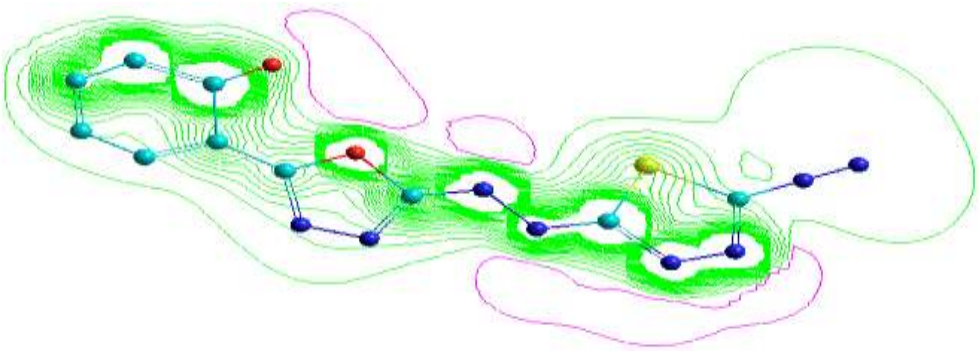


Figure 4: HOMO Electrostatic Potential as Contours for L

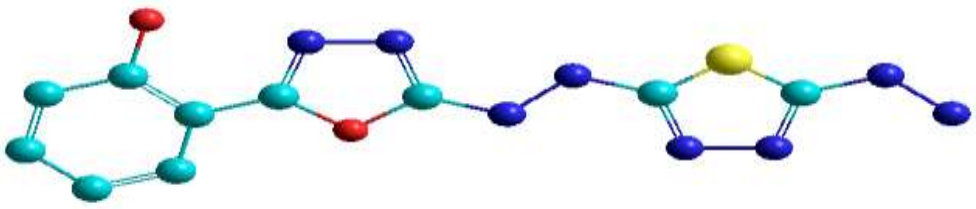


Figure 5: Graphical presentation of stereochemistry of the Ligand

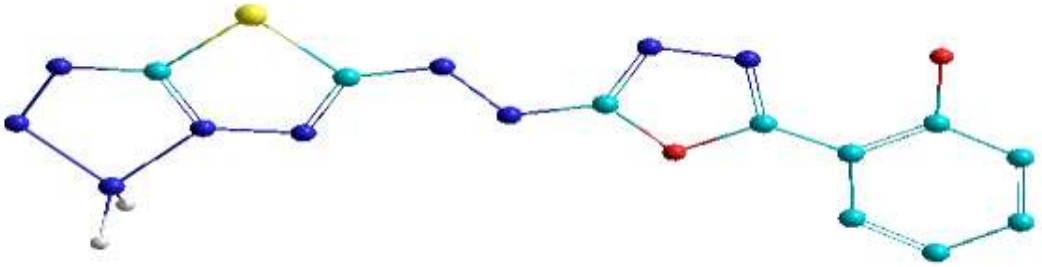


Figure 6: Graphical presentation of stereochemistry of the complex [Co(L1)Cl2]

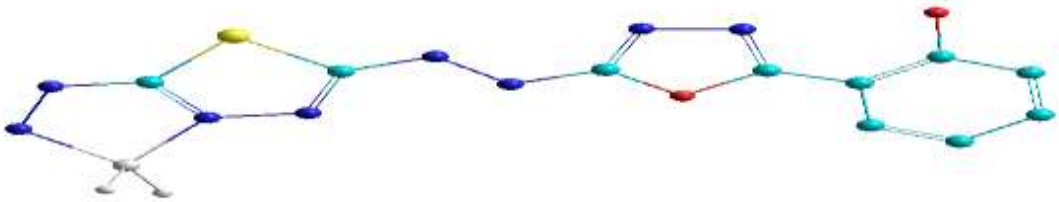


Figure 7: Graphical presentation of stereochemistry of the complex [Ni(L_1)Cl_2]

Figure 8: Graphical presentation of stereochemistry of the complex [Cu(L_1)Cl_2]

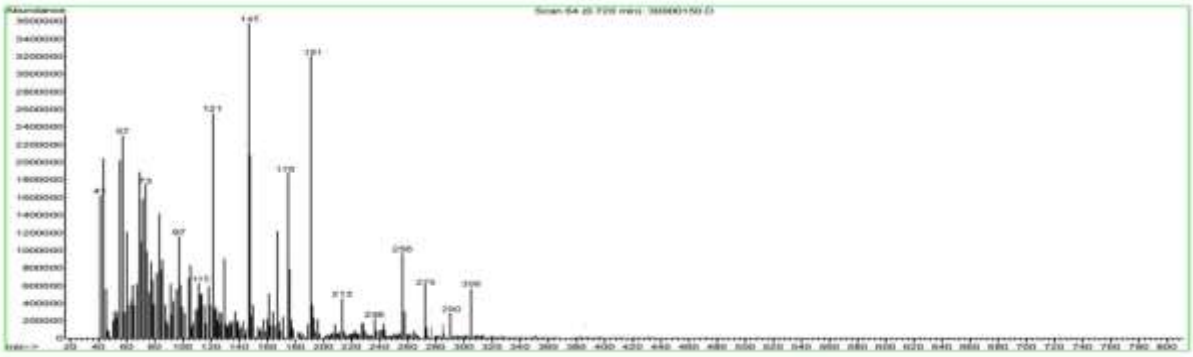


Figure 10: mass spectra of Ligand

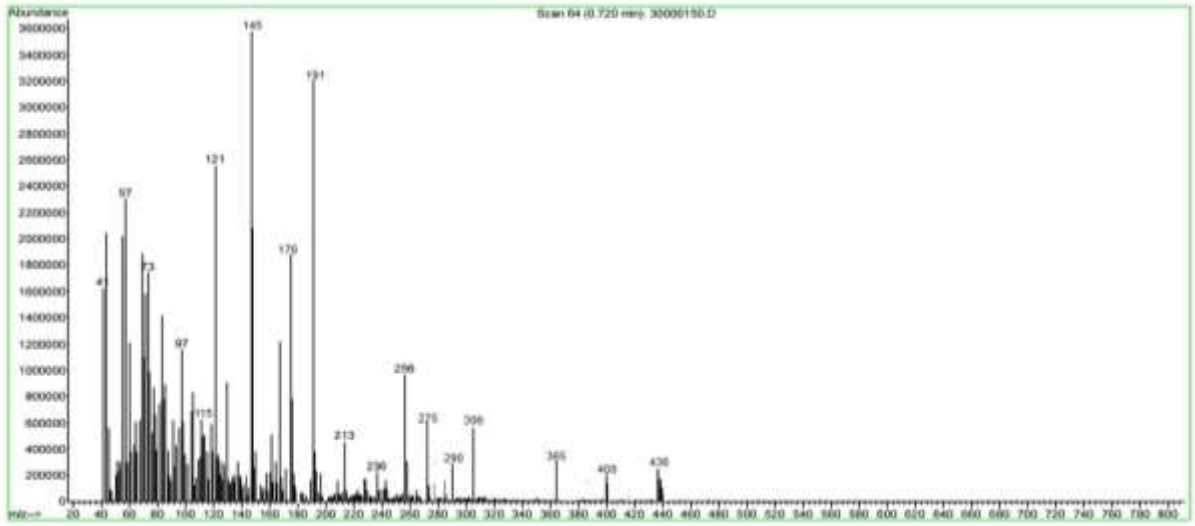


Figure 11: mass spectra of $[Co(L)Cl_2]$

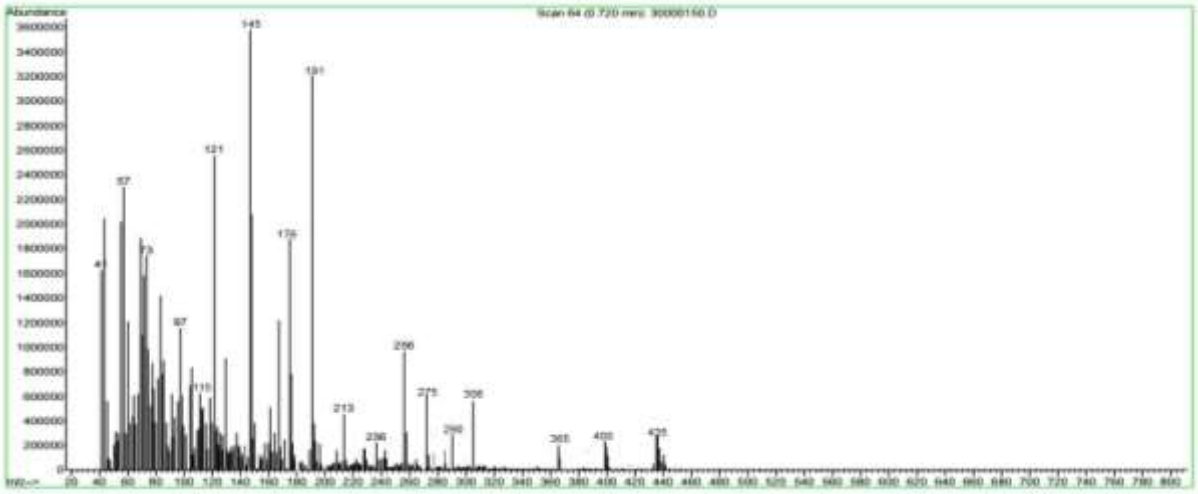


Figure 12: mass spectra of $[Ni(L)Cl_2]$

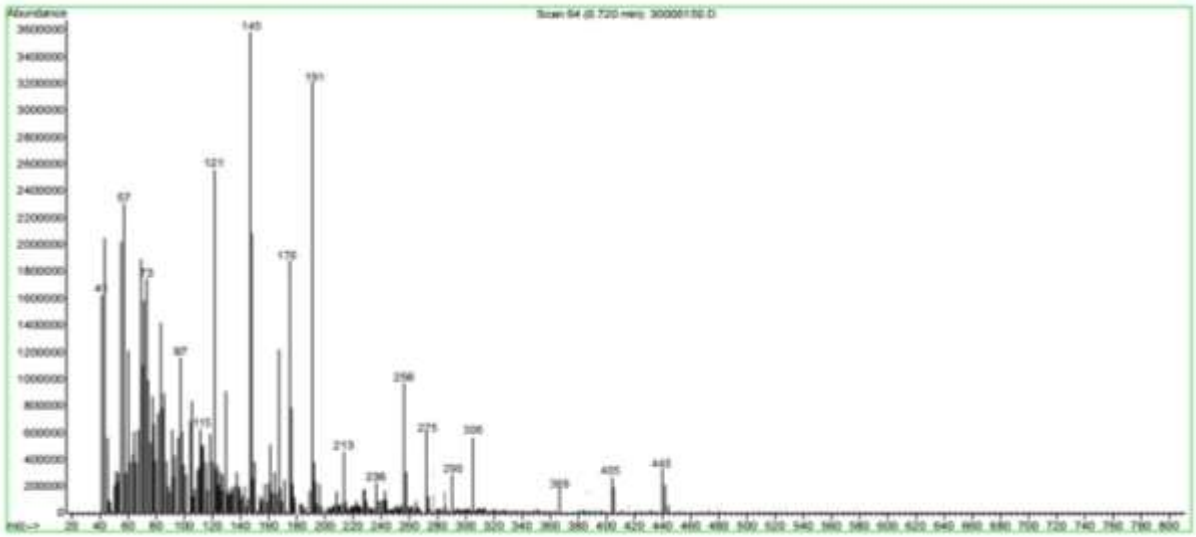


Figure 13: mass spectra of [Cu(L)Cl2]

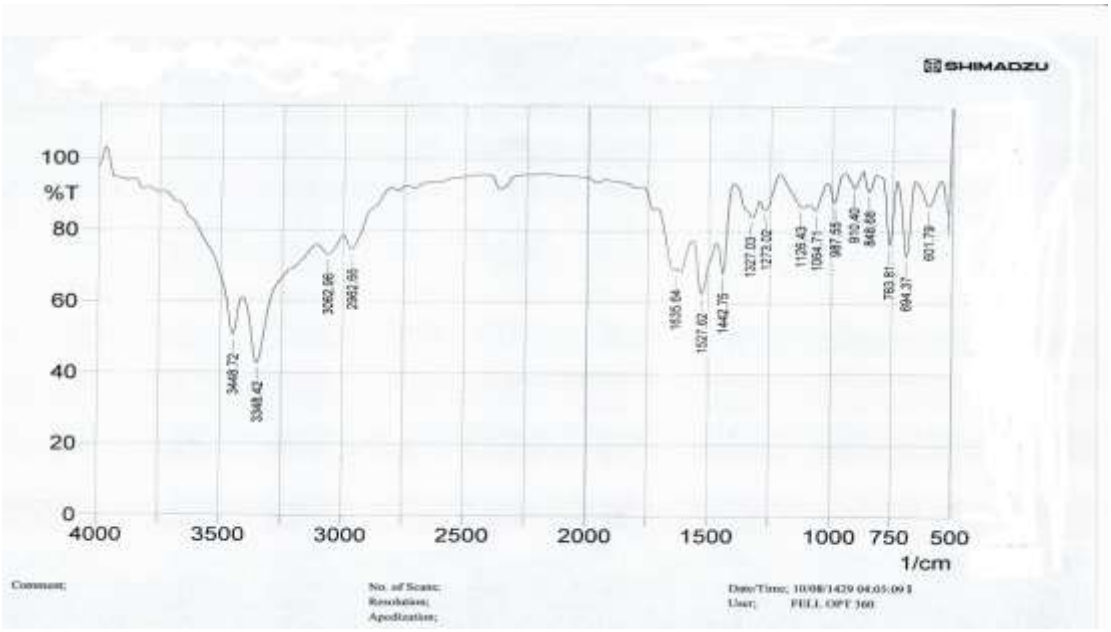


Figure 14: IR spectra of ligand

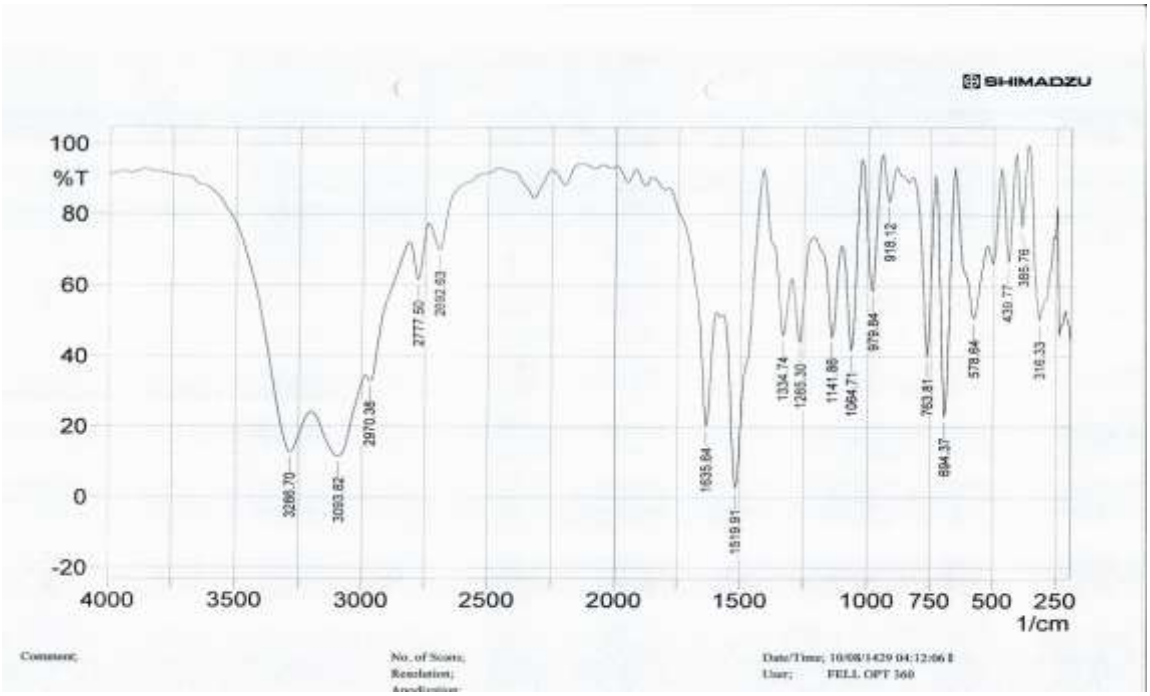


Figure 15: IR spectra of [Ni(L) Cl2]

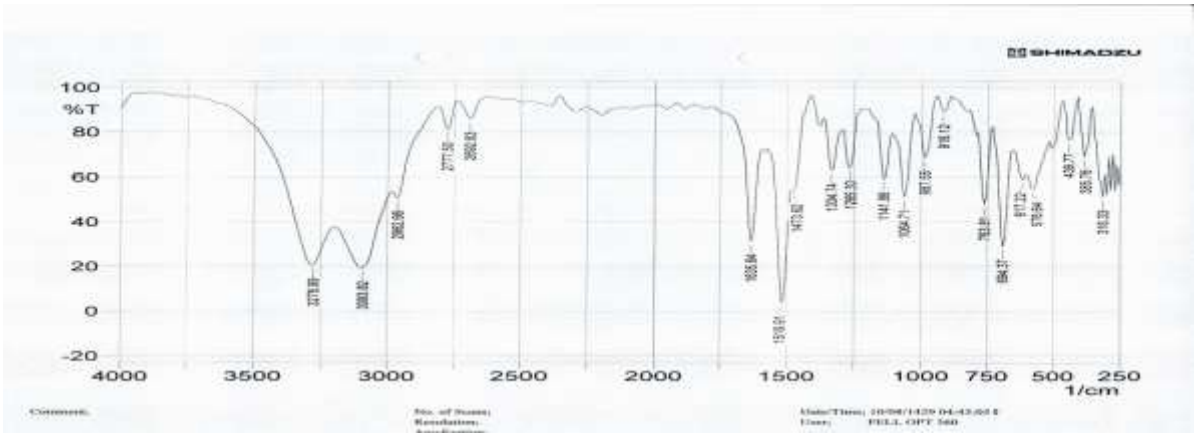


Figure 16: IR spectra of [Co(L) Cl2]

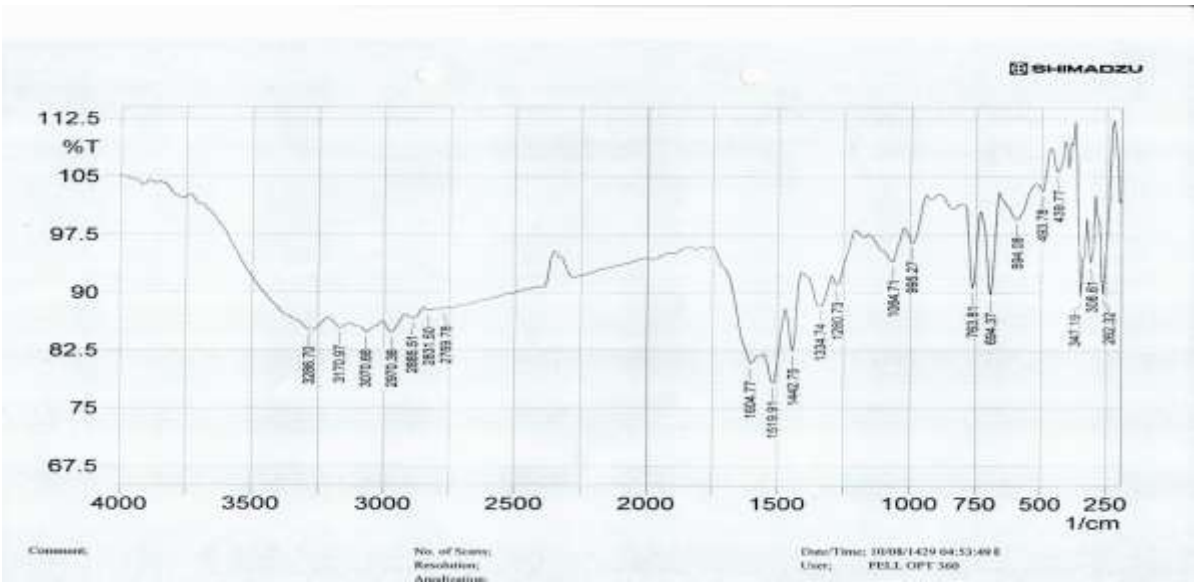


Figure 17: IR spectra of [Cu(L) Cl2]

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