International Journal of Psychosocial Rehabilitation, Vol. 24, Issue 05, 2020
ISSN: 1475-7192

Synthesis, Characterization, Antibacterial, Antioxidant Studies of New 2-(5-(2-((1E,2E)-3-(2-methoxyphenyl)allylidene)hydrazinyl)-1,3,4-oxadiazol-2-yl)Phenol with the Some Transition Metalions

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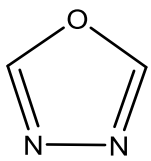
Abstract--- *A new ligand 2-(5-(2-((1E,2E)-3-(2-methoxyphenyl)allylidene)hydrazinyl)-1,3,4-oxadiazol-2-yl)phenol and its complexes with Cr(III), Co(II), Ni(II), Cu(II) ions were synthesized. The new ligand and its complexes have been characterization on the basis their spectra of H^1NMR, mas, Fourier transform infrared (FTIR), as well as magnetic susceptibillty, elemental analysis (CHN) and conductance measurements. The program of Hyperchem 8 have been used for theoretical accounts using PM3 method to study the electrostatic potential that provided good information about the complexity site. Of the result obtained we can suggested octahedral geometries for Cr(III), tetrahedral geometry for Co(III) and square planer geometry for Ni(II) and Cu(II). The antioxidant activity of ligand was evaluated by DPPH scavenger and the ligand was showed high antioxidant activity. The ligand and its complex gave a good activity antibacterial.*

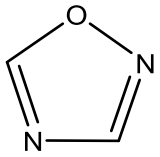
Keywords--- *Ligand, Complexes, Characterization, Electrostatic Potential, Antibacterial.*

I. INTRODUCTION

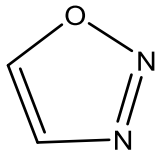
Oxadiazole can regard as simple five membered heterocyclic, aromatic heterocycles that contain three heteroatoms on the same ring have one oxygen and two nitrogen atoms. there are four different isomeric forms such as 1,2,5-oxadiazole, 1,2,4-oxadiazole, 1,2,3-oxadiazole and 1,3,4-oxadiazole [1]. The isomer 1,2,3-oxadiazole reverts to the diazoketone tautomer (unstable) [2].



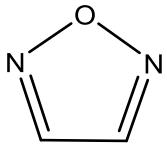
1,3,4-oxadiazole



1,2,4-oxadiazole



1,2,3-oxadiazole



1,2,5-oxadiazole

Oxadiazole which has been found to exhibit various pharmacological activities [3] and so that the compounds containing 1,3,4-oxadiazole moiety play an important application in the field of biological activities as antibacterial [4,5], antifungal [6], anti-inflammatory [7], anti-cancer[8], anticonvulsant[9], antiviral[10], anti-HIV[11], anti-diabetic[12], anti-tubercular[13], lipid peroxidation inhibitor[14]. other application of 1,3,4-oxadiazole as

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DOI: 10.37200/IJPR/V24I5/PR201817
Received: 13 Feb 2020 | Revised: 07 Mar 2020 | Accepted: 23 Mar 2020

insecticidal [15], antioxidant [16], corrosion inhibitor [17], fluorescent and colorimetric chemosensors [18], dyes [19], polymers material [20], light emitting diodes [21].

II. EXPERIMENTAL

All the chemicals and solvents used were of chemically pure grade, and commercially available. All metal salts were used as chloride.

2.1. Physical Measurements

The melting point or the decomposition temperature of all the prepared ligand and metal complexes were determined in an electro thermal melting point apparatus model (Melting SMP31). The FTIR spectra were recorded as KBr disc for ligand and CsI for complexes using a Shimadzu FTIR spectrophotometer (Model: IR- affinity, Shimadzu). Nuclear Magnetic Resonance Spectra were obtained using Bruker DXR System AL500 (500 MHz). Mass Spectra were obtained using (Network Mass Selective Detector 5973).

2.2. Preparation of the ligand

2.2.1. Synthesis of 2-hydroxy benzohydrazide (A)

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[22]. the solid (A) was lighting white, melting point 150 ^{0}C, yield 95%.

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

2-hydroxybenzohydrazide (A) (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 [23]. the solid (B) was yellow, melting point 200 ^{0}C, yield 72%.

2.2.3. Synthesis of 2-(5-hydrazinyl-1,3,4-oxadiazol-2-yl)phenol (C)

2-(5-mercapto-1,3,4-oxadiazol-2-yl)phenol (B) (19.4gm, 0.1mol) and hydrazine monohydrate (5ml, 0.1mol) in ethanol absolute as solvent (50 ml) were refluxed for 15 hours. white precipitate appeared in round bottom [24] was filtered and recrystallized from ethanol absolute. melting point 220 ^{0}C, yield 60%

2.2.4. Synthesis 2-(5-(2-((E)-3-(2-methoxyphenyl)allylidene)hydrazinyl)-1,3,4-oxadiazol-2-yl)phenol

The ligand was synthesized by condensation of 2-(5-hydrazinyl-1,3,4-oxadiazol-2-yl)phenol (C)(9.6gm, 0.05 mol) and o-methoxy cinnamaldehyde (8.1gm, 0.05mol) in 1:1 molar proportions in ethanol absolute (25ml). then the mixture refluxed for 8 hours (monitored by TLC) [25,26]. the ligand was precipitated, filtered and recrystallized from ethanol absolute to get yellow ligand melting point 228-230 ^{0}C, yield 75%.as shown in the scheme.

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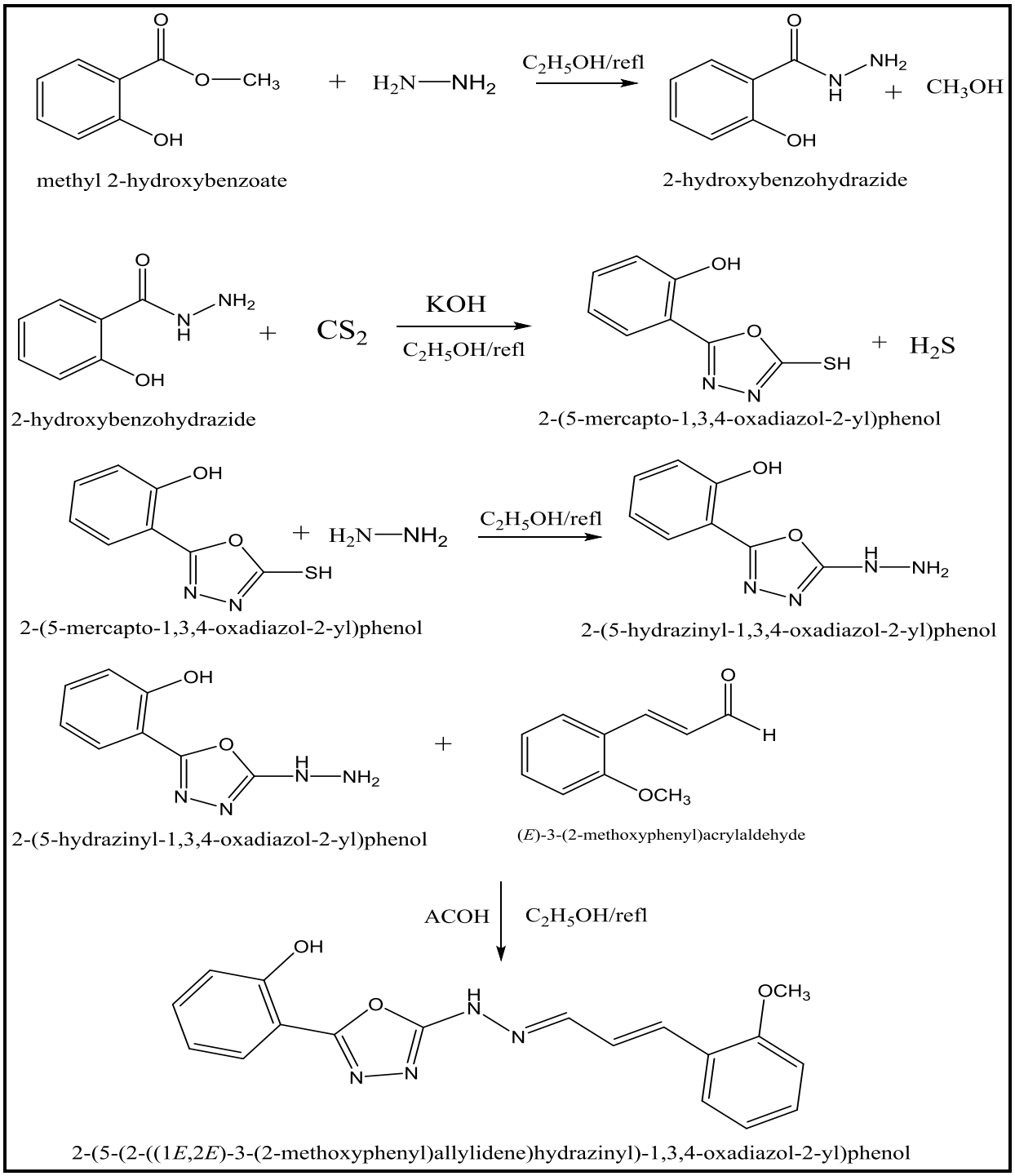

2.3. Preparation of complexes

The complexes were synthesized by mix (0.001mol) from ligand with salts ($CrCl_3.6H_2O$, $CoCl_2.6H_2O$, and $NiCl_2.6H_2O$, $CuCl_2H_2O$) both alone in (15ml) 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].

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III. RESULT AND DISCUSSION

The physical properties of the prepared derivatives 1,3,4- oxadiazole bearing Schiff base which included molecular formula and melting points and elemental analyses (CHNS) as shown in Tables (3 -1). The structures formula of the newly synthesized compound is assigned by the mass spectra, ^{1}HNMR, FT- IR and CHNS elemental analyses. The results obtained are in good agreement with those calculated for the suggested formula.

3.1. Analysis and physical measurements

Table 1: Physical properties, elemental microanalysis and (CHN), percentage of metal and the magnetic momentum data of the ligand and its complexes

No.	formula	colour	C %	H %	N %	M%	ʌ $scm^2 mol^{-1}$	M.p ^{0}C	µeff. B.M
1	$C_{18}H_{16}N_4O_3$(L)	yellow	65.97exp. 66.66cal.	5.02exp. 4.61cal.	18.76exp 18.29cal	…….	…….	228-230	……
2	[Cr(L)Cl_3H_2O]	dark green	……..	…….	……..	9.12	21.9	270-272	3.8
3	[Co(L)Cl_2]	brown	……	……..	……	13.95	6.65	207-210	3.9
4	[Ni(L)Cl_2]H_2O	green yellowish	…….	……	……	11.55	14.12	255-258	0.51
5	[Cu(L)Cl_2]H_2O	green	…….	…….	……	14.92	2.72	181-184	1.9

3.2 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 seven major bands at (3380), (3115), (1585), (1541), (1247),(1165) and (1035) [28]cm^{-1} Which are attributable to(υO-H), (υN-H), (υC=N)imine, (υC=N)Oxa, (υC-O-C)sym, (υ C-O-C)asy and structure movement bands respectively, as shown below in table (2) and figure(10). New bands were formed Attributed to the coordinated (M- N), (M- O) and (M-Cl) bonds and appeared at the region (528-620) cm^{-1}, (347) $cm^{-1}$and (223-266) cm^{-1} respectively. This indicates that the coordinate occurred through the (N), (O) and (Cl) atoms.

Table 2: Infrared spectra of L and its metal complexes (υ cm^{-1})

No.	υ(O-H)	υ(N-H)	υ (C=N) imine	υ (C=N) oxadiazole	υ (C-O-C)	Structure movement	M-N	M-Cl	M-O
L	3380	3115	1585	1541	1247 (asy) 1165 (sy)	1035	……	……	…….
Cr	3356	3132	1580	1515	1253 (asy) 1168 (sy)	1043	624	254	347
Co	3446	3115	1590	1510	1251 (asy) 1166 (sy)	1024	650	266	
Ni	3599	3136	1560	1510	1257 (asy) 1166 (sy)	1011	528	223	
Cu	3410	3115	1570	1519	1253 (asy) 11157 (asy)	1022	615	244	

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3.3 Nuclear Magnetic Resonance

The ^{1}HNMR spectra of the ligand showed signals at (3.87ppm, s, 3H) due to OCH_3 protons, (6.9ppm,dd, 1H) due to Ha proton, (6.95ppm,d, 1H) due to Hb proton, (7-7.73ppm,m,8H) due to protons of aromatic rings, (9.24ppm,d,1H) due to proton of azo methane group (-N=CH-), (10.01ppm,s,1H) due to OH group and (14.04ppm,s,1H) due to N-H proton[28]. As shown in figure (5).

3.4 Mass spectra

The mass spectra of ligand appeared molecular ion peak at 336 m/z which is in conformity with the molecular formula $C_{18}H_{16}N_4O_3$. Other peaks are due to the subsequent fragments like $[C_{18}H_{15}N_4O_2]^{\cdot+}$ =319 m/z, $[C_8H_7N_4O_2]^{\cdot+}$ =191 m/z, $[C8H6N3O2]^{\cdot+}$ =176 m/z, $[C_{10}H_{10}NO]^{\cdot+}$ =160 m/z, $[C_9H_9N]^{\cdot+}$ =131m/z, $[C_4H_4N_3O]^{\cdot+}$ =110 m/z,$[CHN_2]^{\cdot+}$ =41 m/z.

The mass spectral of the Cr(III) complexes showed molecular ion peaks at 512 m/z corresponding to $[Cr(L_1)Cl_3H_2O]^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 458,423,388 m/z due to loss one, two and three chlorine atom respectively.

The mass spectral of the Co(II) complexes showed molecular ion peaks at 466 m/z corresponding to $[Co(L_1)Cl_2]^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 430 m/z,395 m/z due to loss one and two chlorine atom respectively. The mass spectral of the Ni(II) complexes showed molecular ion peaks at 483 m/z corresponding to $[Ni(L_1)Cl_2]H_2O^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 430 m/z,395 m/z due to loss one and two chlorine atom respectively. The mass spectral of the Cu(II) complexes showed molecular ion peaks at 483 m/z corresponding to $[Cu\ (L_1)Cl_2]H_2O^{\cdot+}$ stoichiometry. This complex shows another a fragmentation peaks at 435 m/z,399 m/z due to loss one and two chlorine atom respectively. sa shown in figure (6-9).

3.5 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 Cr(III) complex was 3.8 BM, expected for octahedral geometry. the magnetic momentum value was 3.9 BM for Co(II) complex with six paired electrons, This value confirms that cobalt(II) suggesting tetrahedral geometry. 0.51 BM for Ni(II) and 1.9 for Cu(II) suggesting square planar geometry respectively[29].

3.6 Evaluation of Antioxidant Activity

DPPH scavenger was used for evaluation of antioxidant activity of the ligand 2-(5-(2-((1E,2E)-3-(2-methoxyphenyl)allylidene)hydrazinyl)-1,3,4-oxadiazol-2-yl)phenol. The ethanol solution of each DPPH scavenger and the ligand was prepared in concentration 1 mM and then 0.5 ml of DPPH solution was mixed with (1, 0.5, 0.250)ml of the ligand solution to become the new concentration (200, 100, 50)mM and then absorption measuring for each solution at 517 nm after (1, 2, 3, 4) hour, the ligand gave high antioxidant activity because the phenol group donate the hydrogen atom to the free radical by HAT mechanism to become a stable free radicals. This stability increases with the extent of delocalization [30] and the IC50 (compound concentration required to reduce the

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absorbance of the DPPH control solution by 50%) [31] was determined. The IC50 is decrease with the increasing of a solution concentration and the time. As shown in table (3) and in figure (1)

Activity inhibition = (AC-ACS/AC) *100

Where, AC= the absorbance of control DPPH, ACS= the absorbance of control and sample

Table 3: Antioxidant data

Conc. µM	Time (1h)		Time (2)		Time (3h)		Time (4h)	
	inh	IC50	inh	IC50	inh	IC50	inh	IC50
50	30	95	32	91	34	80	36	76
100	52		54		59		61	
200	79		82		87		88	

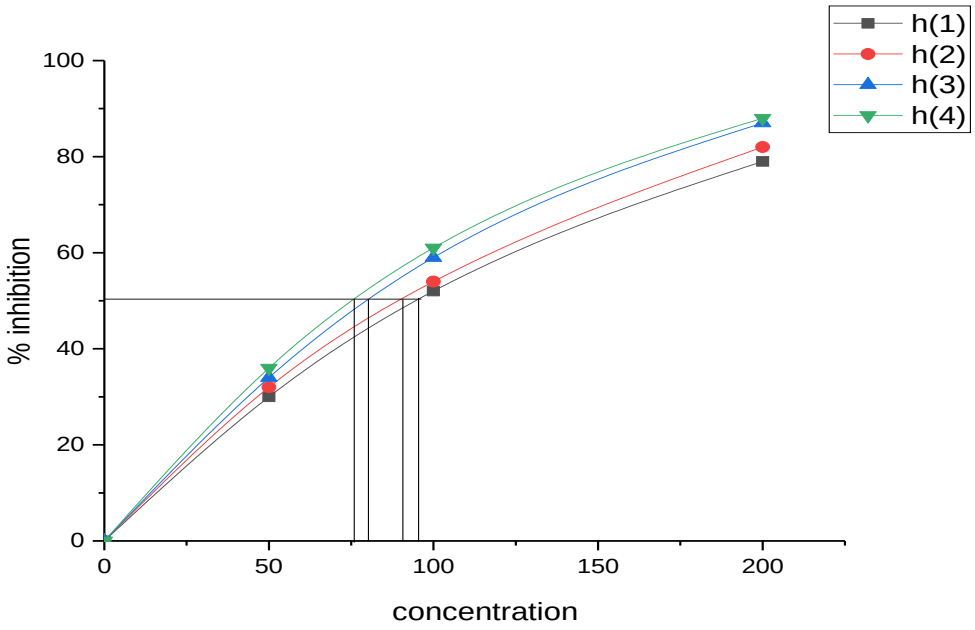

Figure 1: Antioxidant activity for ligand

3.7 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 10 mg/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 ^{0}C [32]. From the observation of the results in table (4) and figure (2)

Affirms that all compound shows good anti-bacterial activity. Out of all the synthesis compounds Nickel(II) complex is more bactericidal than others Even the standard drug.

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Table 4: Anti-bacterial data of ligand and its complexes

No.	compound	Escherichia coli Inhibition zone(mm)	Staphylococcus Aurens Inhibition zone(mm)
	control	25	35
1	$C_{18}H_{16}N_4O_3$ (L)	10	24
2	$[Cr(L)Cl_3H_2O]$	17	20
3	$[Co(L)Cl_2]$	23	21
4	$[Ni(L)Cl_2]H_2O$	25	30
5	$[Cu(L)Cl_2]H_2O$	38	35

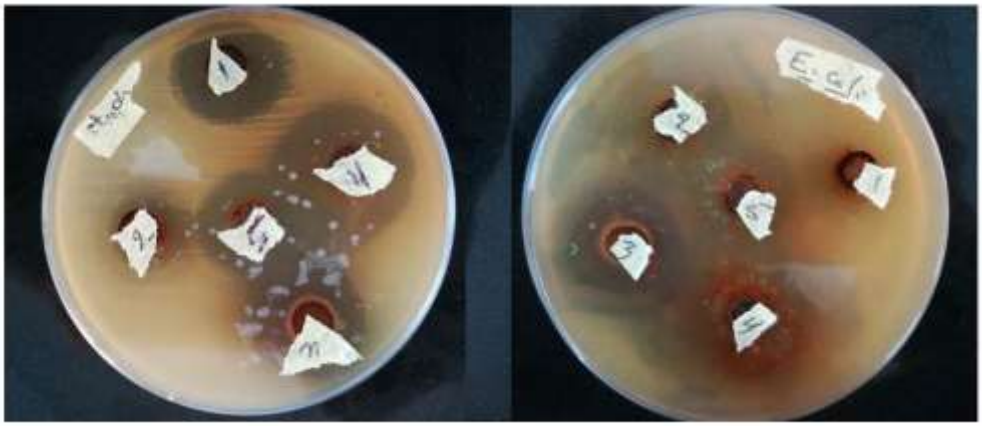


Figure 2: Antibicterial activity

IV. MOLECULAR ELECTROSTATIC POTENTIAL (MEP)

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 [33] as shown in figures [3-4].

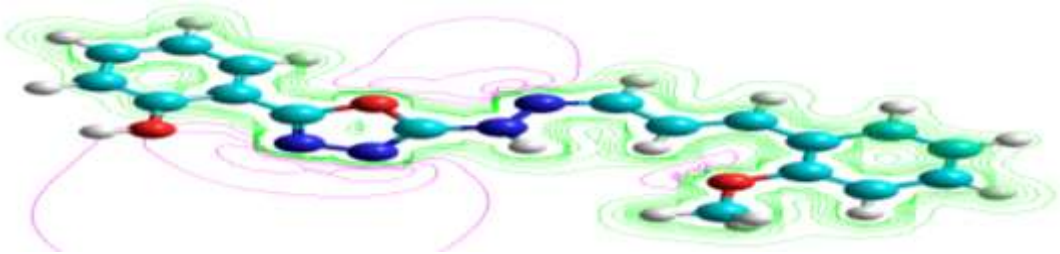
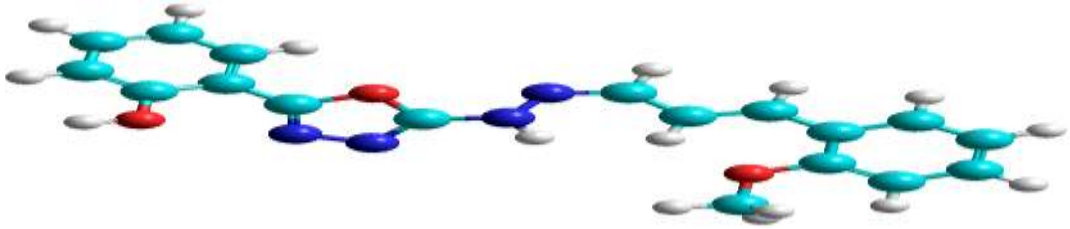


Figure 3: HOMO Electrostatic Potential as Contours for ligand



Ligand

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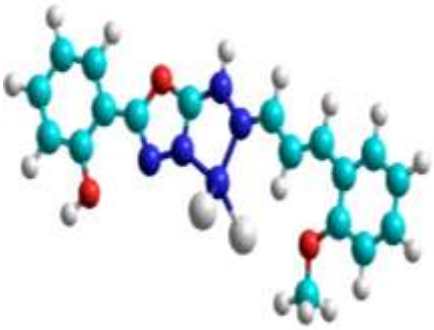
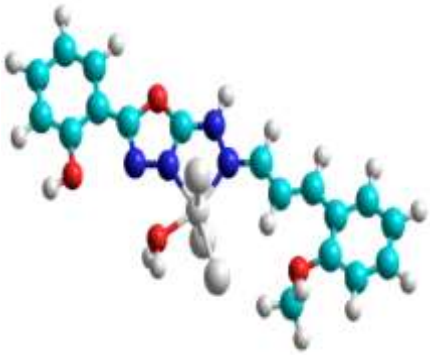
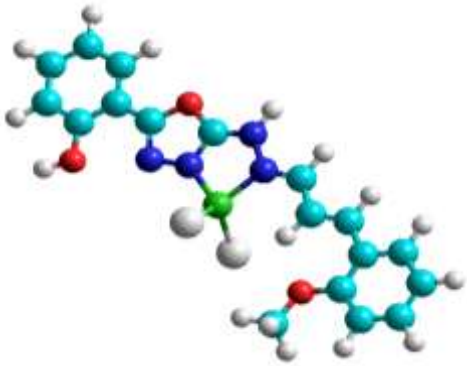


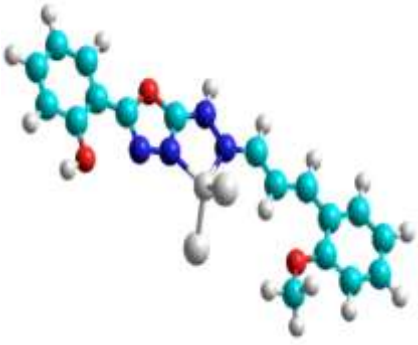
T.h $[Co(L_1)Cl_2]$



O.h $[Cr(L_1)Cl_3H_2O]$



S.p $[Cu(L_1)Cl_2]$



S.p $[Ni(L_1)Cl_2]$

Figure 4: Graphical presentation of stereochemistry of the Ligand and the complexes

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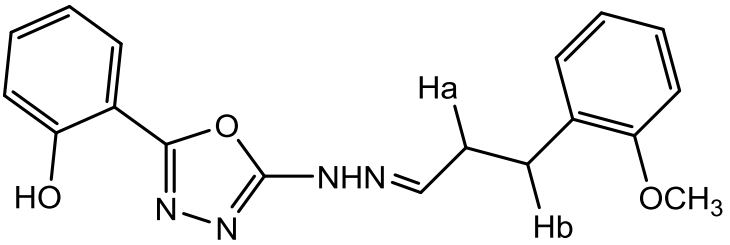

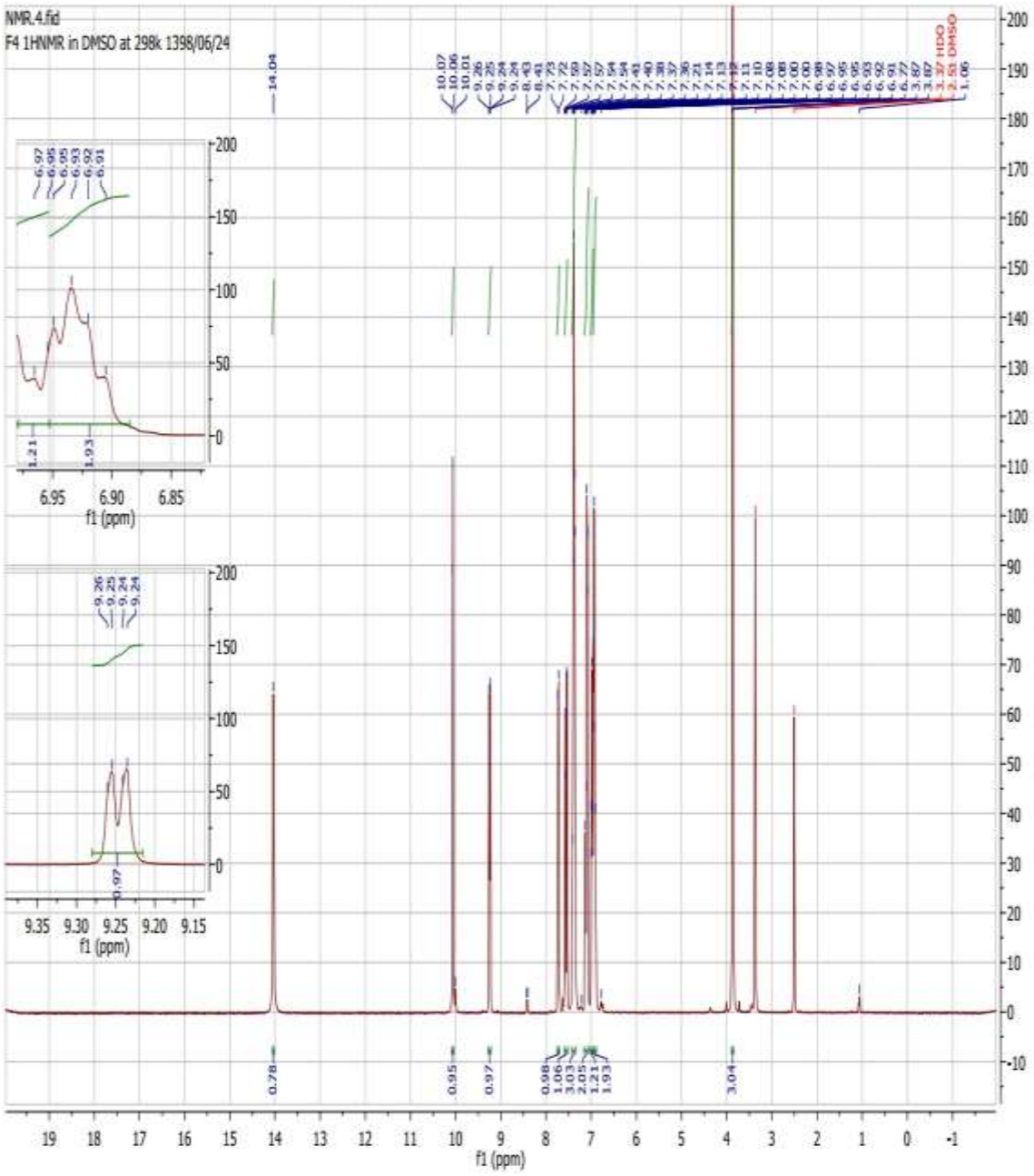

Figure 5: NMR spectra of the ligand

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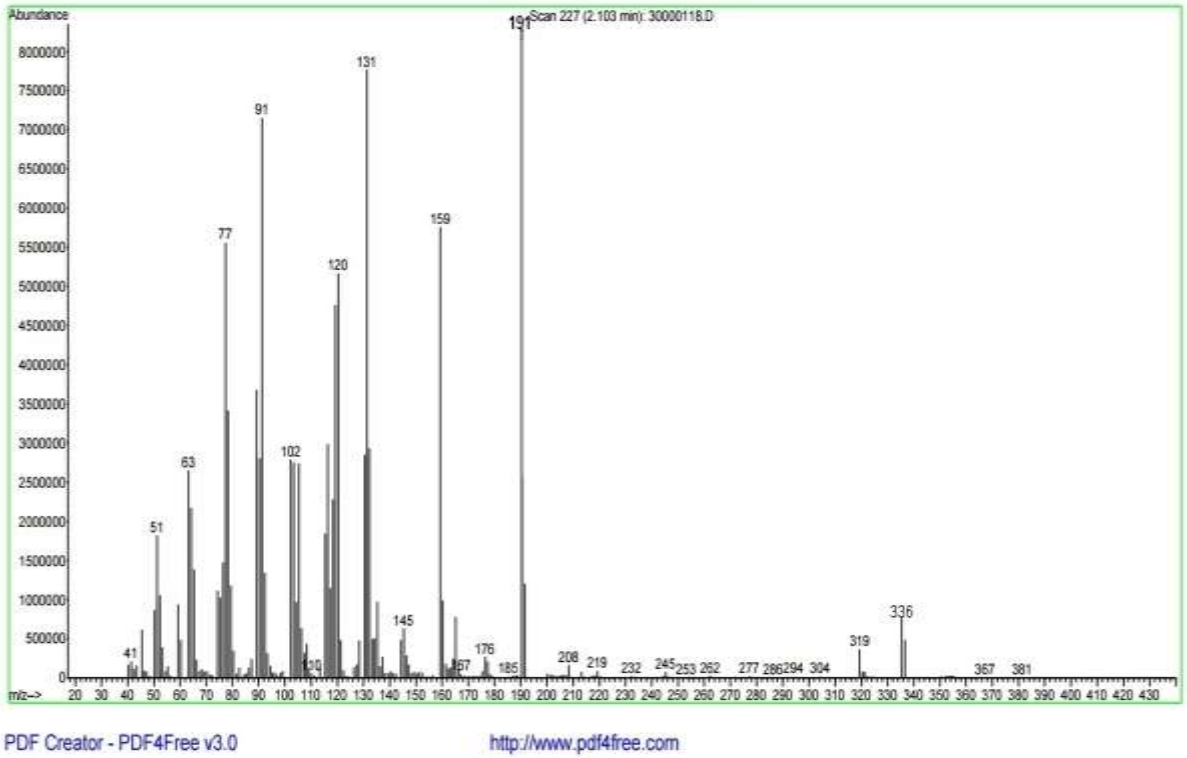

Figure 6: Mass spectra of ligand

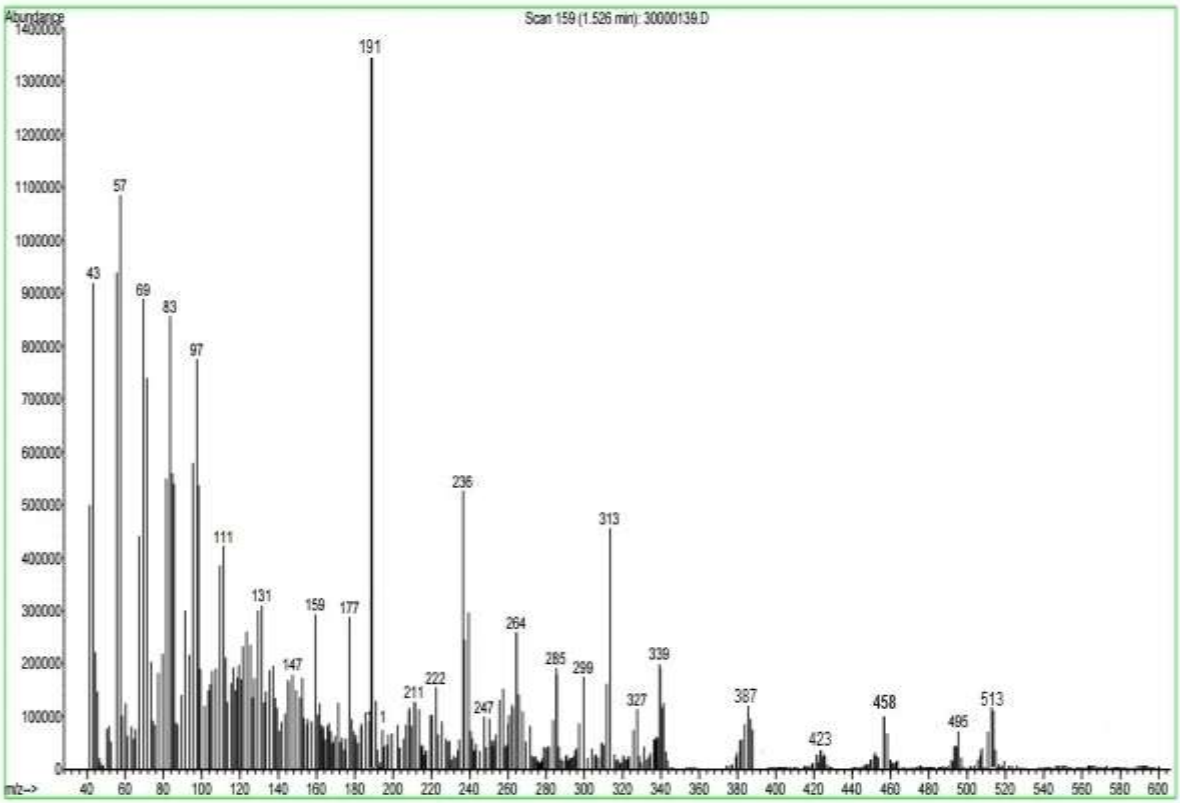

Figure 7: mass spectra of $[Cr(L_1)Cl_3H_2O]$

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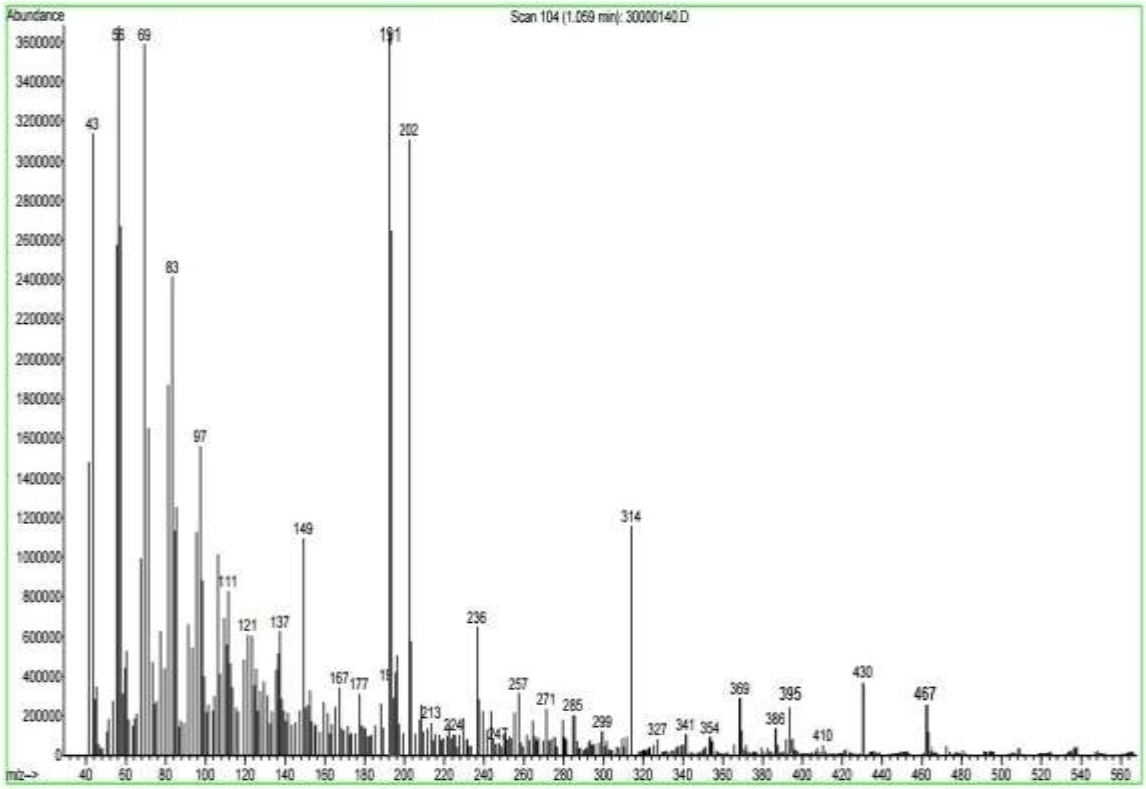

Figure 8: Mass spectra of $[Co(L_1)Cl_2]$

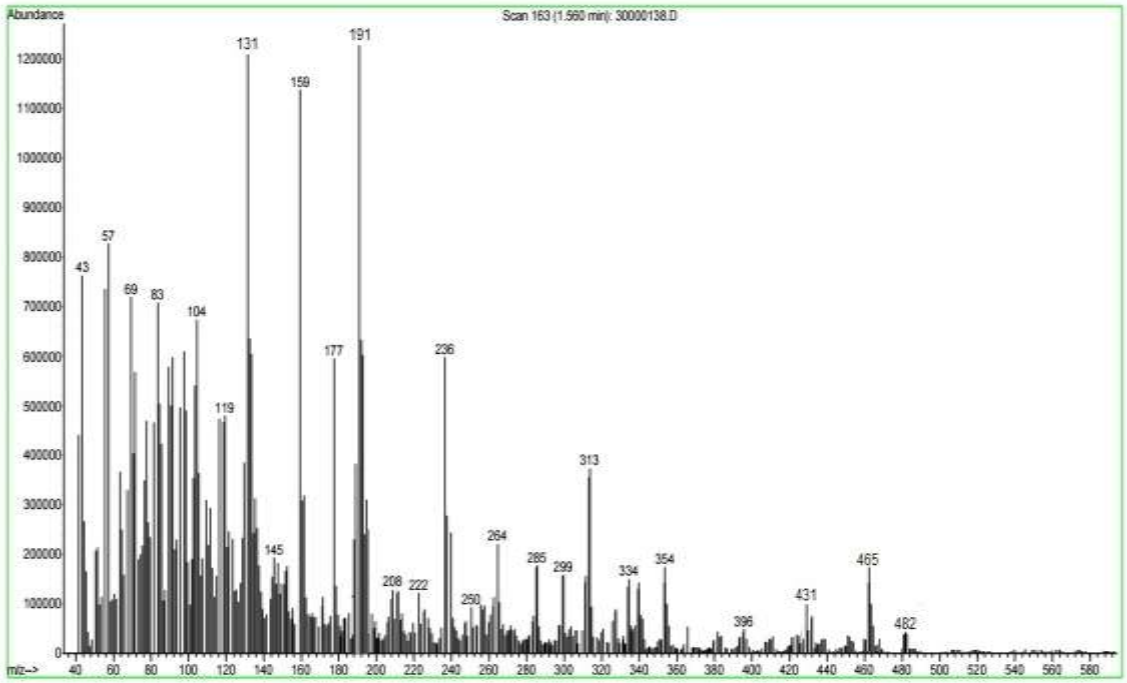

Figure 9: Mass spectra of $[Ni\ (L_1)Cl_2]H_2O$

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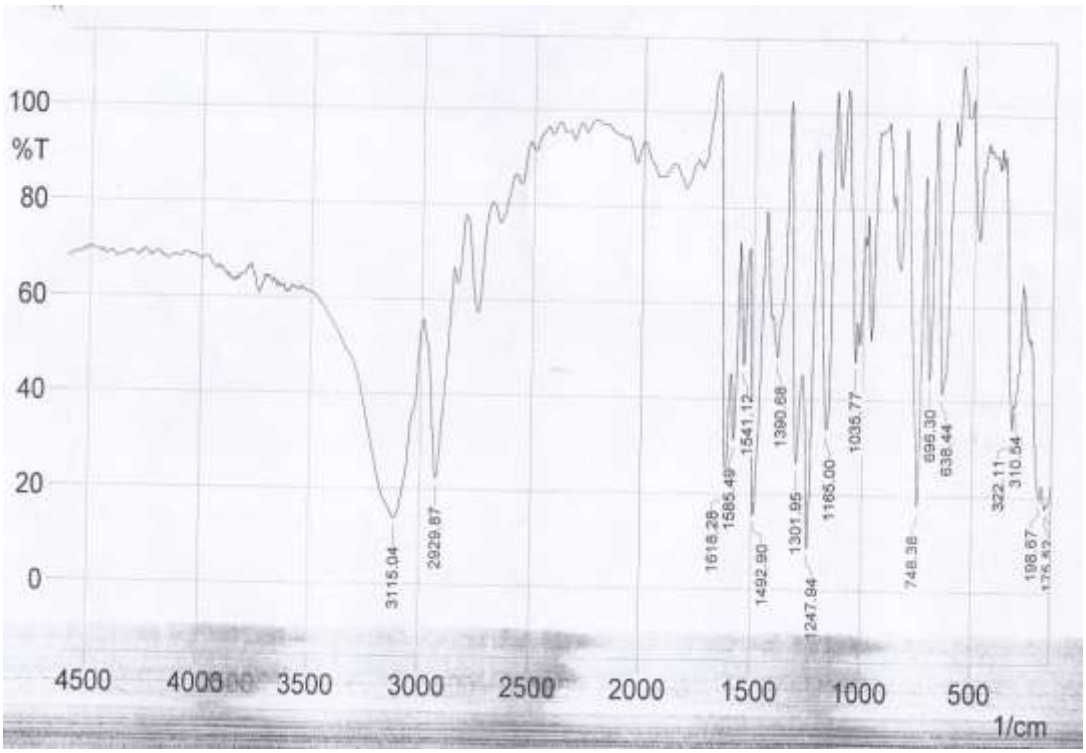

Figure 10: IR spectra of ligand

V. CONCLUSION

The 1,3,4-oxdiazole derivative acts as a bidentate ligand. The spectroscopic data exhibit the involvement of NH and CH=N groups in coordination to the central transition metal ion. various techniques have been used such as (FTIR, 1H.NMR and Mass) spectra as well as Molar conductance and magnetic susceptibility to characterization of transition metal complexes. an octahedral geometry for Cr(III) complex,square plainer geometry for Ni(II) and tetrahedral geometry for Cu(II) complex is proposed. The results of the electrostatic potential study were quite consistent with the practical results of the complexity sites.

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