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Mn(II),Fe(III),Co(II)and Rh(III) complexes with azo ligand: Synthesis, characterization, thermal analysis and bioactivity

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Abstract

New series of metal ions complexes have been prepared from the new ligand [4-Amino-N-(5-methyl-isaxazol-3-yl)-benzenesulfonamide] derived from Sulfamethoxazole and 3-aminophenol. Accordingly, mono-nuclear Mn(II), Fe(III), Co (II), and Rh(III) complexes were prepared by the reaction of previous ligand with $MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $FeCl_3.6H_2O$ and $RhCl_3H_2O$, respectively. The compounds have been characterized by Fourier-transform infrared (FTIR), ultraviolet–visible (UV–vis), mass, ^{1}H-, and ^{13}C-nuclear magnetic resonance (NMR) spectra and thermo gravimetric analysis (TGA& DSC) curve, Bohr magnetic (B.M.), elemental microanalyses, metal ions, chloride containing, and molar conductance.These reviews uncovered octahedral geometries for complexes. The investigation of complexes development by means at molar proportion andoccupation strategy in DMF solution has been researched, and results were reliable to those found in the solid complexes with a proportion of (M:L) as (1:2).

Keywords: Azo complexes, Bioactivity, Mass spectroscopy, Spectroscopic studies, Thermal analysis.

Introduction:

The coordination compounds resulting from azo compounds are of increasing importance in the various fields of industry, agriculture, medicine and medicine, and as auxiliary factors, and what helped them in this is the effective groups present in the compound originally, which gave them additional stability and effectiveness at the same time[1-4]. Antibiotics are substances which, even at low concentrations, inhibit the growth and reproduction of bacteria. Infectious disease treatment would have been inconceivable today without antibiotics [5]. In this work, we have tried in using diazotization coupling reaction between sulfamethaxazole (sulfur–containing organic compound) and 3-aminophenol(m- aminophenol)forming (E)-3-((4-amino-2-hydroxyphenyl)diazenyl)-N-(4-methylisoxazol-3-yl)benzenesulfonamide can be classified as azo compound that in turn coordinate with each of Mn(II), Fe(III),Co(II)and Rh(III) metal ions in 2:1 ratio. In such reactions, the use of acidic media to prepare the intermediate compound is the basis for the preparation of important azo compounds with widespread uses in various fields[6-9]. The aim of this work is to synthesize a novel azo ligand from an aromatic amine with spectroscopic analysis of (NMR, Mass, IR, UV-Vis) and studying of thermal decomposition and thermal stability by using TGA and DSC. its composition as well as to synthesize Mn(II),Fe(III), Co(II) and Rh(III)complexes with spectroscopic analysis and studying of thermal decomposition and thermal stability by using TGA and DSC.

Material and methods:

Materials and instrumentation

The Sulfamethoxazole, Hydrochloric acid, Sodium Nitrite $NaNO_2$, 3-aminophenol, Sodium Hydroxide NaOH, ethanol, methanol, DMSO, DMF, $MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $FeCl_3.6H_2O$ and $RhCl_3H_2O$ Equipped with (Sigma-Aldrich, Merck, and others). The complexes' molar conductances were measured by using a Conductometer WTW at 25°C at a concentration of 1×10^{-3}M.DMSO was used to dissolve all of the complexes (DMSO). On a mass spectrometry (MS) QP50A: DI Analysis

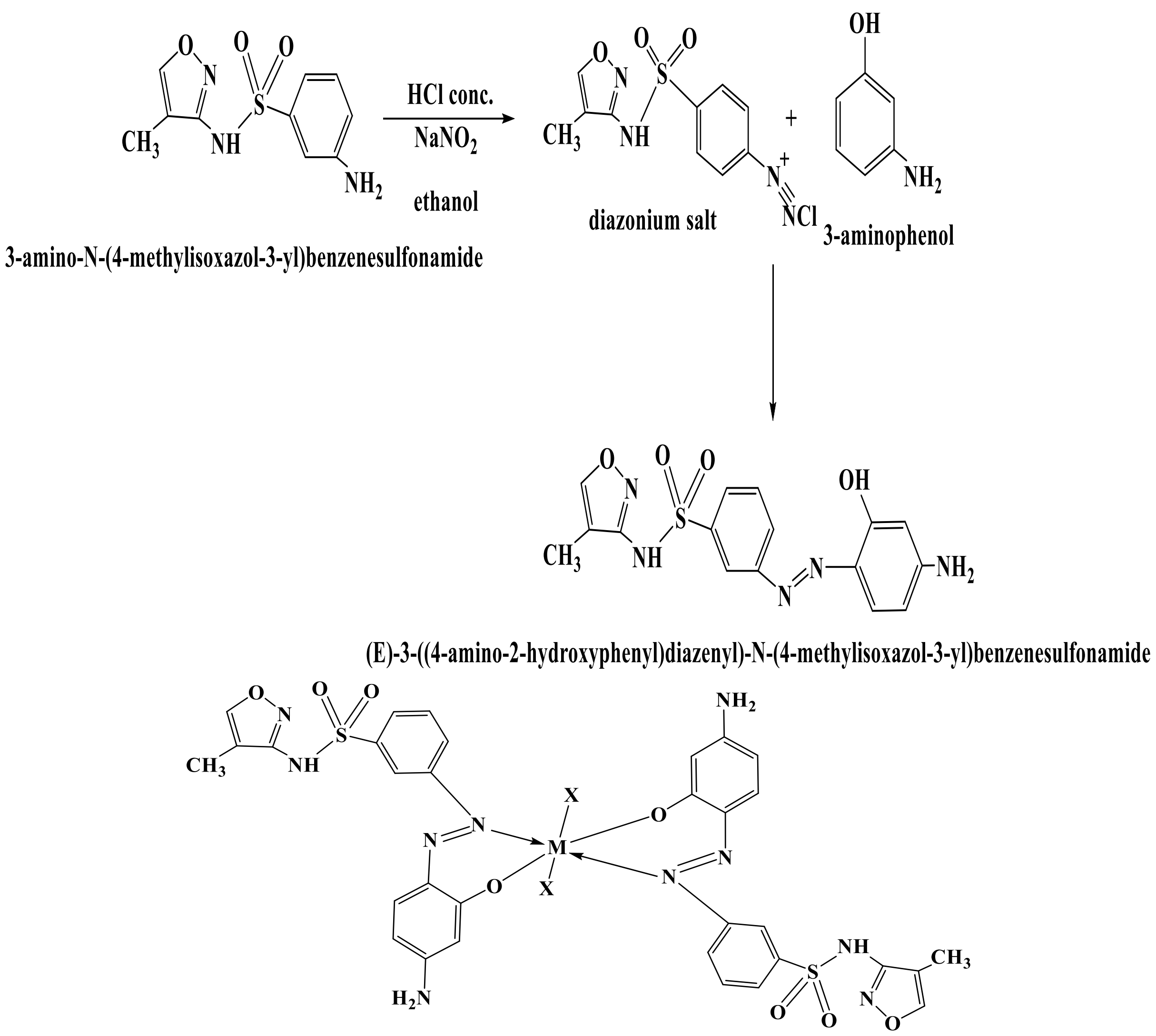
ShimadzuQP-2010-Plus (E170Ev) spectrometer, the UV–Vis spectrophotometer UV-1800 Shimadzu was used to analyze the spectra in the ultraviolet–visible (UV–Vis) range, the IR Prestige-21 was used to investigate the (FTIR) spectra, and the Perkin-Elmer Pyris Diamond TGA&DSC was used to conduct thermogravimetric studies. A Brucker500 MHz was used to record the proton nuclear magnetic resonance (^{1}H&^{13}C-NMR) spectra for ligand in DMSO-d^6. The Euro vector model EA/3000, single-V.3.O-single, was used to conduct elemental analyses (C, H, N, S and O). Metal ions were estimated as metal oxides using a gravimetric method, mass spectra for substances were recorded..

Synthesis of azo dye ligand

The ligand in Scheme 1, was synthesized according to the suggested method[10]. Dissolving (1g, 3.948 mol) of Sulfamethoxazole in a mixture composed of 37% (2ml) concentrated Hydrochloric acid HCl, (15ml) distilled water and 15 ml of ethanol. The solution is cooled from 0°C up to 5°C then a gradual addition of (10%, 1g, 14.49mmol) hydrated Sodium Nitrite $NaNO_2$ to the solution occurs with continuous stirring and avoid any increasing in temperature up to 5°C. Then leaving the solution for about 45 min. to perform the diazotezation process resulting in diazonium salt, salt solution is adding gradually with continuous stirring onto (0.43g, 3.948mmol) of 3-aminophenol dissolved in 20ml of ethanol. Change in solution's color into dark color is observed keeping on stirring about 30 min. to perform the reaction. The solution isolates until being stable then adding few drops of Sodium Hydroxide NaOH solution in order to equivalence the middle of reaction till reaching to 6.8pH at which the perfect precipitating of ligand occurs. Finally, the former is filtered and making recrystallization by methanol and dried resulting in 1.34g, 90.70%, 192-194ºC m. p and orange precipitate., The yield is (1.34g), 90.97%, mp.192-194°C:^{1}H-NMR (DMSO-d$_6$, ppm) Ar-OH (11.14 , 1H , singlet) , N-H (9.51,1H , singlet)Ar-H(7.78-7.80, 4H, multiplet) C-H(aromatic) besides OHC-H(aromatic) besides OHC-H (aromatic) besides NH_2(6.87, 2H, douplet) Ar-NH_2 (5.94, 2H, singlet) C-H (aromatic) besides CH_3 (5.49, 1H, singlet) CH_3 (2.51, 3H, singlet) DMSO (solvent) (2.55 – 2.64) : ^{13}C-NMR (DMSO , ppm ,): 15.16(C-1), 174.60(C-2), 107.50(C-3), 167.70(C-4), 162.15(C-5), 131.97(C-6 C-7), 132.02(C-8), 146.94(C-9), 148.76(C-10), 157.27(C-11), 134.31(C-12), 115.39(C-13), 121.95(C-14), 151.10(C-15), 153.24(C-16) [11-13]. Fig.1, show the ^{13}C-NMR spectrum of ligand.

General method for the preparation of metallic ions complexes

The metallic ions complexes were made with metal chlorides for Mn(II), Fe(III), Co(II) and Rh(III). An amount of (0.373g, 1m.mol) from azo ligand, dissolved in 10 mL absolute ethanol, was gradually added in drops wise with stirring to a (0.5m.mol) amount of [1:2] M:L for Mn(II), Fe(III), Co(II) and Rh(III), $MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $FeCl_3.6H_2O$ and $RhCl_3H_2O$. The mixture was heated for 2 hours at (50-70)°C, then chilled in an ice bath until precipitated, then left overnight. To remove any unreacted components, the solid complexes were separated and washed with distilled water and a little amount of hot ethanol. Finally, vacuum desiccators were used to dry the complexes. The analytical and physical properties of the ligand and its metal complexes are summarized in Table 1.



Scheme 1. Synthesis of ligand and its complexes

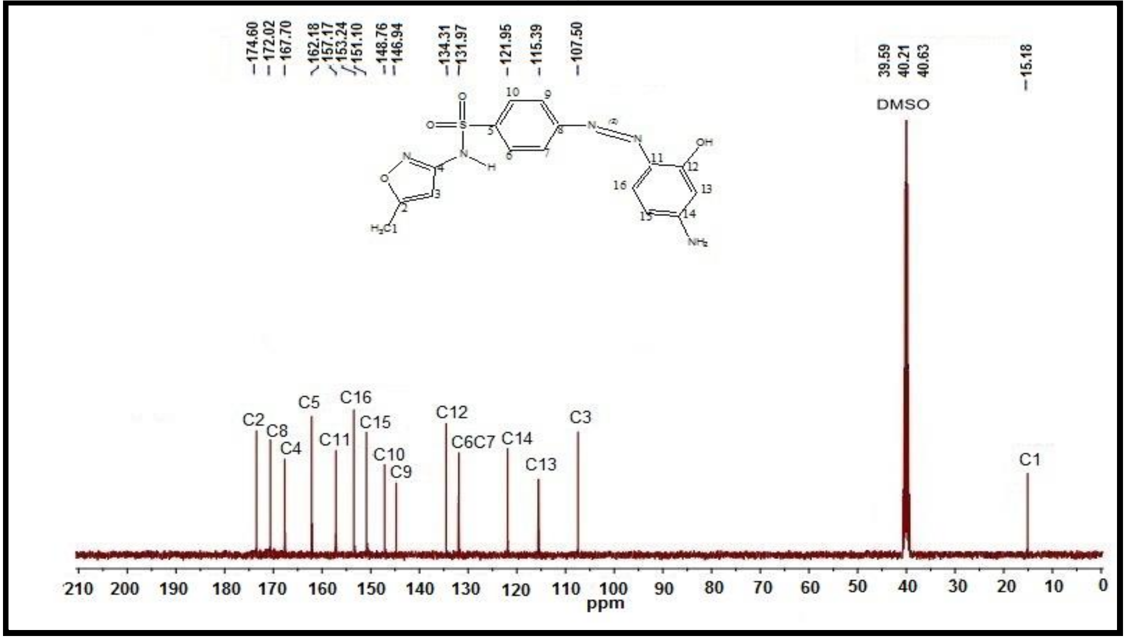


Figure 1. ^{13}C-NMR spectrumof ligand

Result and Discussion:

Physical and chemical properties of azo dye ligand

Microscopic analysis of the elements, the infrared spectrum, the proton NMR spectrum, carbon NMR spectrum, the malar conductivity, TGA & DSC and Mass, and the azo complexes was used to identify the prepared ligands and their complexes. Table 1 shows this with some physical properties

Table 1. Pysical properties & analytical data of ligand & their complexes.

Comp.	**Chemical Formula M.wt**	**Color**	**m.p °C**	**Elemental microanalysis%**						
				C Found C Calc	H Found H Calc.	N Found N Calc.	O Found O Calc.	S Found S Calc.	M Found M Calc.	Cl Found Cl Calc.
LH	$C_{16}H_{15}N_5O_4S$ 373.39	Orange	192-	52.44	4.99	20.01	16.10	7.66	-	-
			194	51.47	4.05	18.76	17.14	8.59	-	-
$[Mn(L)_2(H_2O)_2]$	$C_{32}H_{32}N_{10}CoO_{10}S_2$	Dark brown	266 d	44.89	4.60	17.09	20.20	7.12	5.96	-
	835.73			45.99	3.86	16.76	19.14	7.39	6.57	-
$[Fe(L)_2(H_2O)Cl]$	$C_{32}H_{30}N_{10}FeO_9S_2Cl$	Brown	256-	45.04	4.23	17.21	16.31	7.79	6.33	4.88
	854.07		257	45.00	3.54	16.40	16.86	7.51	6.54	4.15
$[Co(L)_2(H_2O)_2]$	$C_{32}H_{32}N_{10}CoO_{10}S_2$	Brown	213-	46.24	4.15	17.07	18.81	7.89	6.75	-
	839.72		215	45.77	3.84	16.68	19.05	7.64	7.02	-
$[Rh(L)_2(H_2O)Cl]$	$C_{32}H_{30}N_{10}RhO_9S_2Cl$	Red brown	301 d	43.05	2.98	14.89	14.91	8.08		8.08
	901.13			42.65	3.36	15.54	15.98	7.12		7.12

D = decompose

Electronic spectra measurements

The UV–Vis spectra of the ligand LH and its complexes. With absorption maxima at (253 nm, 38759.6 cm^{-1}) ascribed to the π⟶π* transition and two peaks at (435 nm, 22988.5 cm^{-1}) attributed to the n⟶π* transition a peak with a high intensity band formed with absorption maxima. There were six absorption peaks in the electronic spectra of the $[Mn(L)_2 (H_2O)_2]$ complex. The peaks at 275nm and 360nm is ascribed to the ligand, while the (π⟶π*) complex, the peak at 402 nm is ascribed to the ligand, while the n⟶π* complex and three peaks in the (602nm) (713nm) and (809nm) while the one peak at are attributed to the (d-d) electronic transitions types $^6A_{1g}\longrightarrow{}^4A_{1g}$, ^4E_g(G), $^6A_{6g}\longrightarrow{}^4T_{2g}$(G)and $^6A_{6g}\longrightarrow{}^4T_{1g}$(G)respectively, Furthermore, the magnetic moment of the Mn(II) (d^5) complexes is found to be (3.71 B.M)[14-16]. All the above mentioned data correspond to an octahedral geometry. The electronic spectrum of the paramagnetic (3.66B.M)Fe(III) complex was ascribed to the peak at 280nm and 360nm is ascribed to the ligand, while the(π⟶π*) and (n⟶π*), and the three peaks at 684nm, 850nm and 996nm was assigned to the $^6A_{1g}\longrightarrow{}^4A_{1g}$, ^4E_g (G), $^6A_{6g}\longrightarrow{}^4T_{2g}$(G)and $^6A_{6g}\longrightarrow{}^4T_{1g}$(G)transitions, respectively, indicating an Octahedral geometry Fig.2. Whilethe electronic spectra of the Co(II) complex revealed to the peak at 267nm and 451nm is ascribed to the ligand, while the(π⟶π*) and [n⟶π* & C.TML] and three peaks at 730nm, 922nm and 1027nm corresponding to$^4T_{1g}\longrightarrow{}^4T_{1g}$, $^4T_{1g}\longrightarrow{}^4A_{2g}$and $^4T_{1g}\longrightarrow{}^4T_{2g}$,respectively indicating an octahedral geometry and the Co(II) complex was paramagnetic (3.88 B.M.)[17-19], The electronic spectrum of the diamagnetic Rh(III) complex was ascribed to the peak at 268nm and 446nm is ascribed to the ligand, while the (π⟶π*) and [n⟶π* & C.TML], and the three peaks at 752nm 900nm and 998nm was assigned to the $^1A_{1g}\longrightarrow{}^1T_{1g}$, $^1A_{1g}\longrightarrow{}^1T_{2g}$ and $^1A_{1g}\longrightarrow{}^1T_{2g}$ transitions, respectively Fig.3, indicating an Octahedral geometry diamagnetic[20] Table 2 show the data of complexes electronic spectra and molar conductivity.

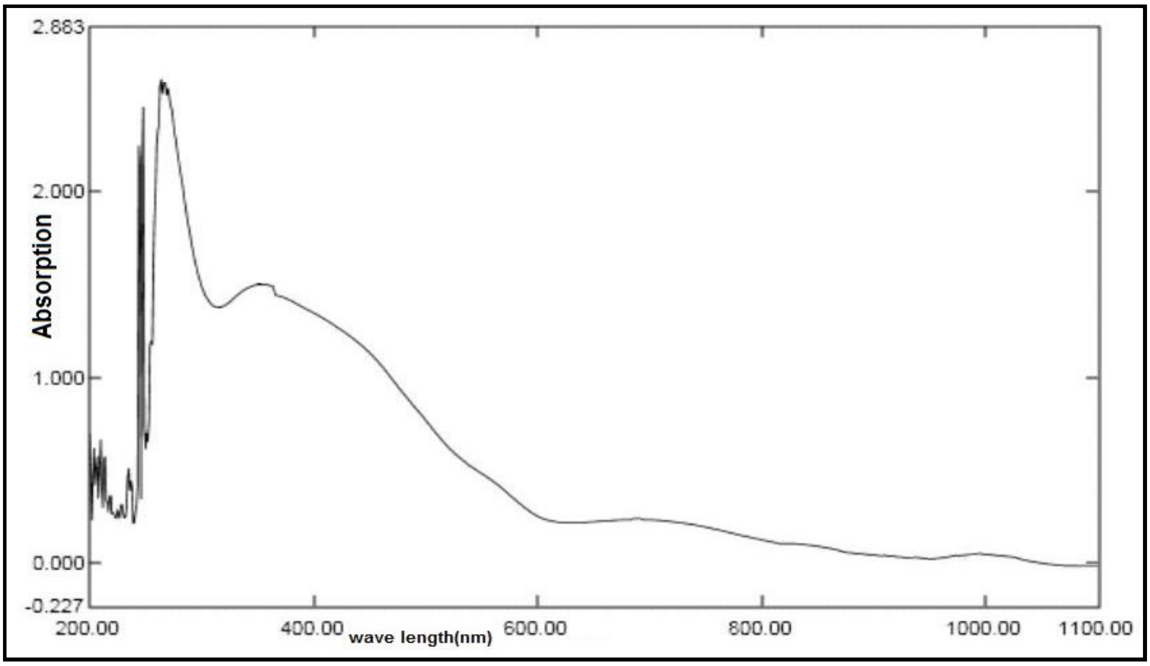


Figure 2. Electronic spectra of Fe-complex

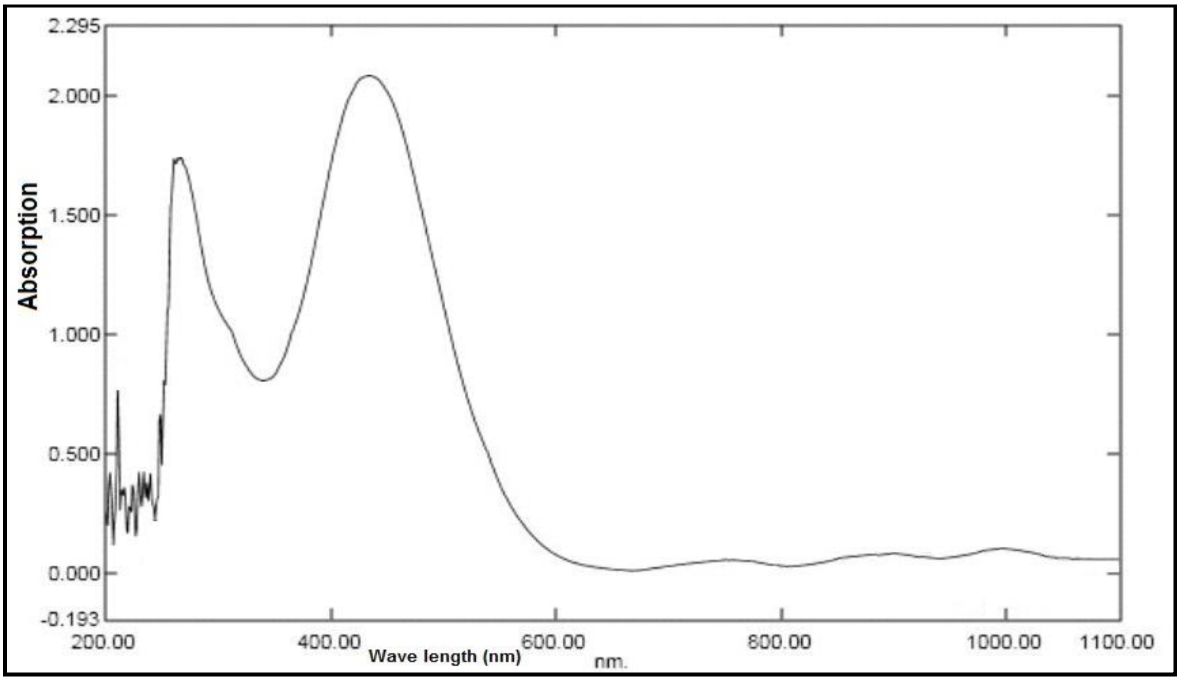


Figure 3. Electronic spectra of Rh(III)complex

Table 2. Electronic spectral data of the metal complexes with LH ligand and molar conductivity in DMSO (1 × 10^{-3} M)

Complexes Geometry	λ_{max} (nm)	υcm^{-1}	ABS	ε_{max} L mol^{-1}cm^{-1}	Assignment	Λ_m cm^2 Ω^{-1}mol^{-1}
LH	258	38759.6	1.182	1182	$\pi^* \rightarrow \pi$	-
	435	22988.5	2.571	2571	$\pi^* \rightarrow n$	
[Mn(L)$_2$ (H$_2$O)$_2$]	275	36363.6	2.061	2061	$\pi^* \rightarrow \pi$	9
	360	27777.7	3.200	3200	$\pi^* \rightarrow \pi$	
Octahedral	402	24875.6	3.298	3298	$\pi^* \rightarrow n$	
	602	16611.2	0.197	197	$^6A_{1g} \rightarrow {}^4T_{2g(G)}$	
	713	14025.2	0.225	225	$^6A_{1g} \rightarrow {}^4T_{1(G)}$	
	809	12360.9	0.180	180	$^6A_{1g} \rightarrow {}^4E_{g(D)}$	
[Fe(L)$_2$ (H$_2$O)Cl]	280	35714.2	2.661	2661	$\pi^* \rightarrow \pi$	16
	360	27777.7	1.500	1500	$\pi^* \rightarrow n$	
Octahedral	684	14619.8	0.350	350	$^6A_{1g} \rightarrow {}^4T_{2g(G)}$	
	850	11764.7	0.180	180	$^6A_{1g} \rightarrow {}^4T_{1(G)}$	
	996	10040.	0.151	151	$^6A_{1g} \rightarrow {}^4E_{g(D)}$	
[Co(L)$_2$ (H$_2$O)$_2$]	267	37453.1	0.480	480	$\pi^* \rightarrow \pi$	12
	451	22172.9	0.998	998	π^*+C.TML$\rightarrow n$	
Octahedral	730	13698.6	0.110	110	$^4T_{1g(p)} \rightarrow {}^4T_{1g}$	
	922	10845.9	0.108	108	$^4A_{2g(f)} \rightarrow {}^4T_{1g}$	
	1027	9737	0.130	130	$^4T_{2g(f)} \rightarrow {}^4T_{2g(f)}$	
[Rh(L)$_2$ (H$_2$O)Cl]	268	37313.4	1.753	1753	$\pi^* \rightarrow \pi$	14
	446	22421.5	2.100	2100	π^*+C.TML$\rightarrow n$	
Octahedral	752	13297.8	0.098	98	$^1T_{1g} \rightarrow {}^1A_{1g}$	
	900	11111.1	0.113	113	$^1T_{2g} \rightarrow {}^1A_{1g}$	
	998	10020	0.196	196	$^3T_{2g} \rightarrow {}^1A_{1g}$	

Liquid chromatography–mass spectrometry (LC–Mass) measurements

The electron impact of fragmentation was used to acquire mass spectra of the ligand and metal complexes. High-resolution MS of the free azo ligand and its complexes, as well as large fragments linked to breakdown products, was obtained in general. The electron impact mass spectrum of ligand LH. This ligand molecular weight is calculated to be 373.39g/mol. The spectra showed a peak at 373 m/z, which was attributed to [M]$^+$ and corresponded to a novel azo moiety $C_{16}H_{15}N_5O_4S$.Their brightness indicates the pieces' stability Fig.4 depicts the mass spectrum of the Mn(II) complex. The complexmoiety $C_{32}H_{32}S_2N_{10}MnO_{10}$ had a peak at 836 m/z, which corresponded to the complexmoiety $C_{32}H_{32}S_2N_{10}MnO_{10}$ in the spectrum. Fig.5 depicts the mass spectrum of the Fe(III) complex. The compound moiety $C_{32}H_{30}N_{10}O_9FeClS_2$ was identified by a peak at 854 m/z in the spectra. The electron impact mass spectrum of the Co(II) complex. The complexmoiety$C_{32}H_{32}N_{10}O_{10}CoS_2$ was identified by a peak at 839 m/z in the spectra. The electron impact mass spectrum of Rh(III). This complex molecular weight is calculated to be 901.13/mol. The spectra showed a peak at 901 m/z, which was attributed to [M]$^+$ and corresponded to a novel azo moiety $C_{32}H_{30}N_{10}O_9S_2RhCl$.[21-23]. In Schemes 2–6, suggested fragmentation routes and structural assignments of pieces are given.

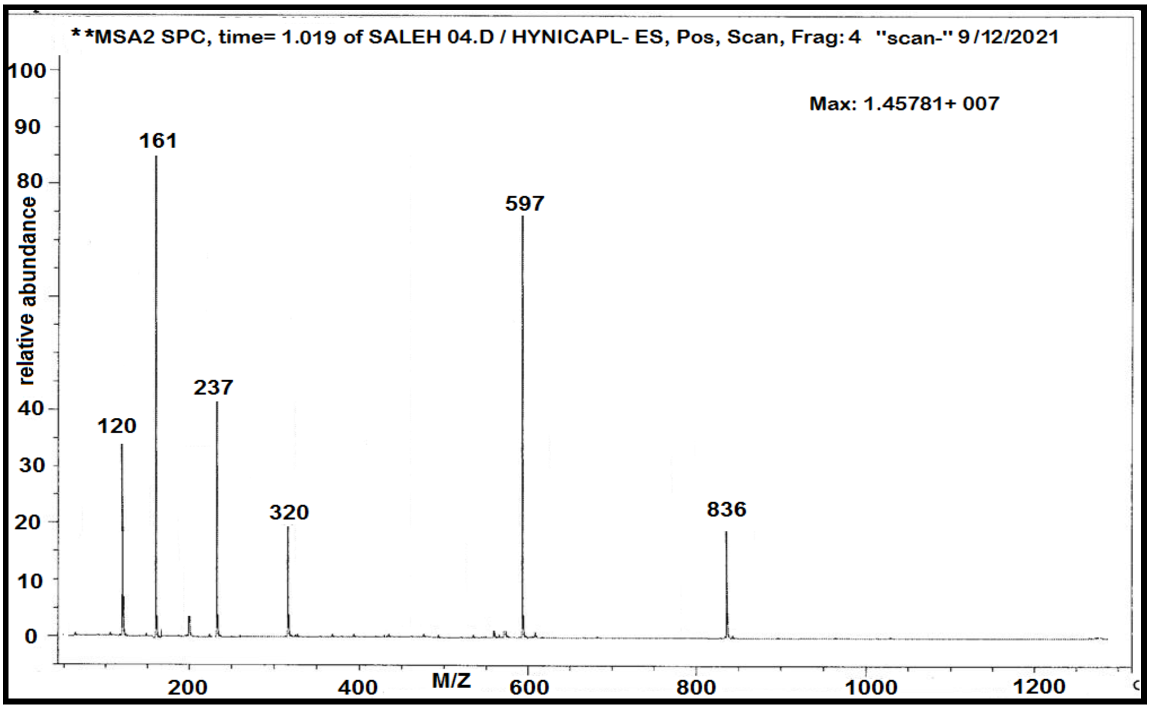


Figure 4. (LC–Mass) spectrum of Mn-complex

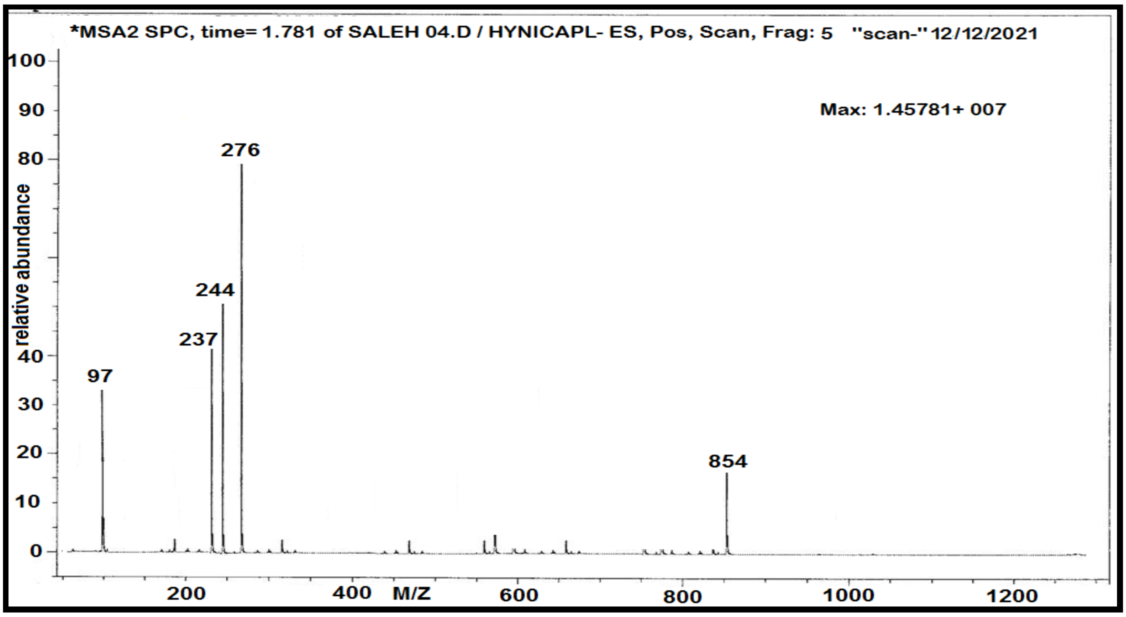
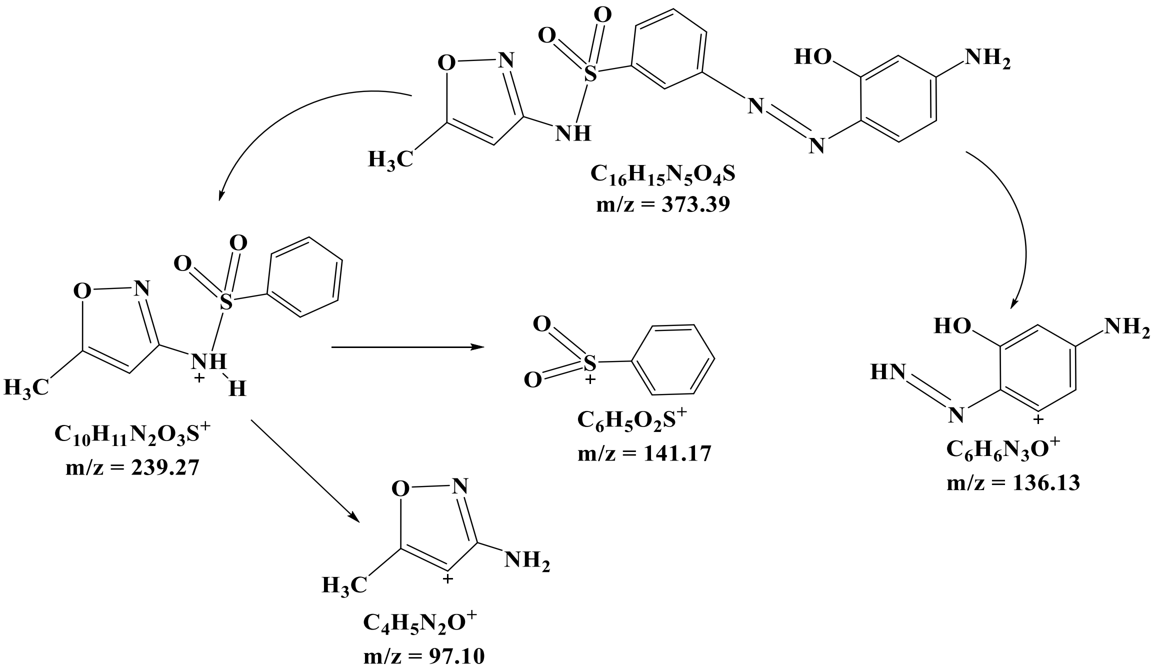
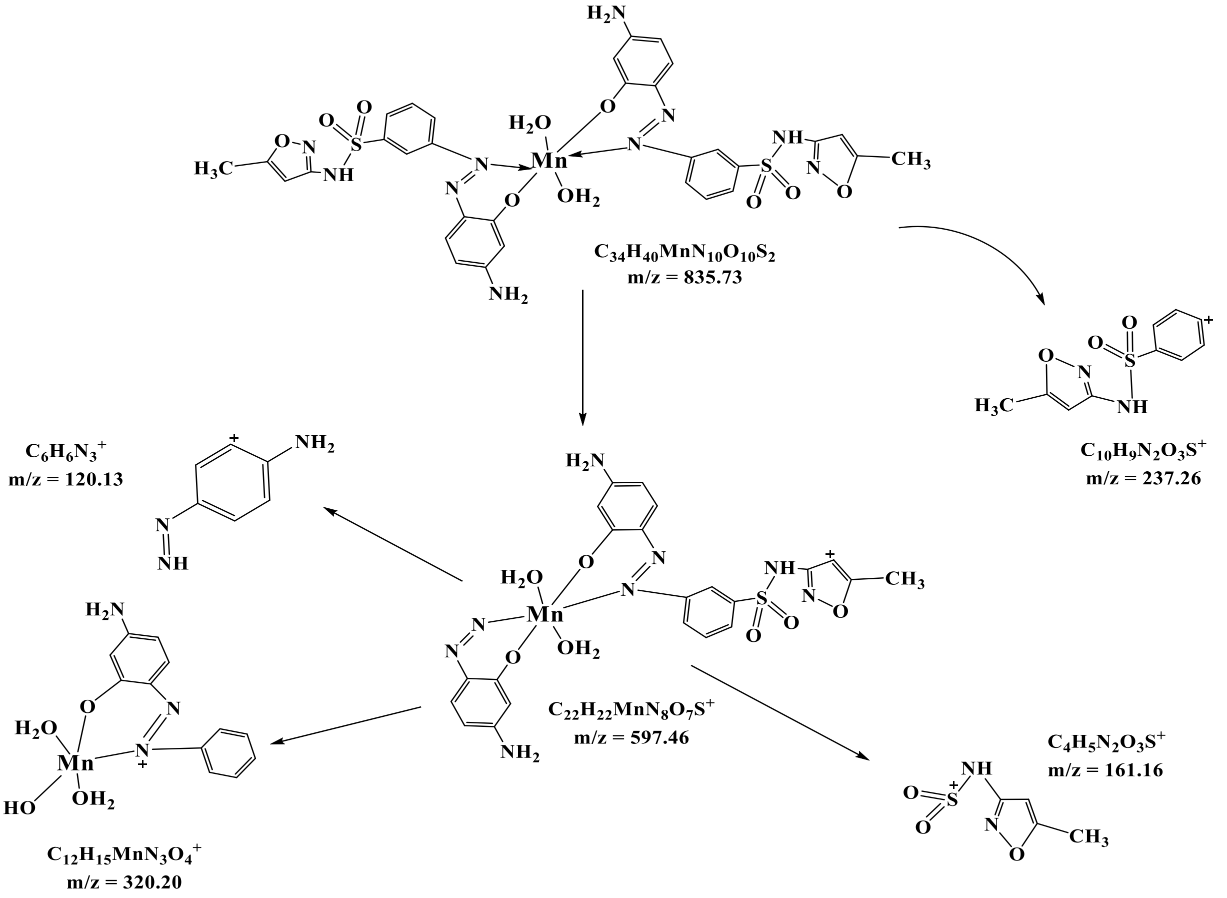


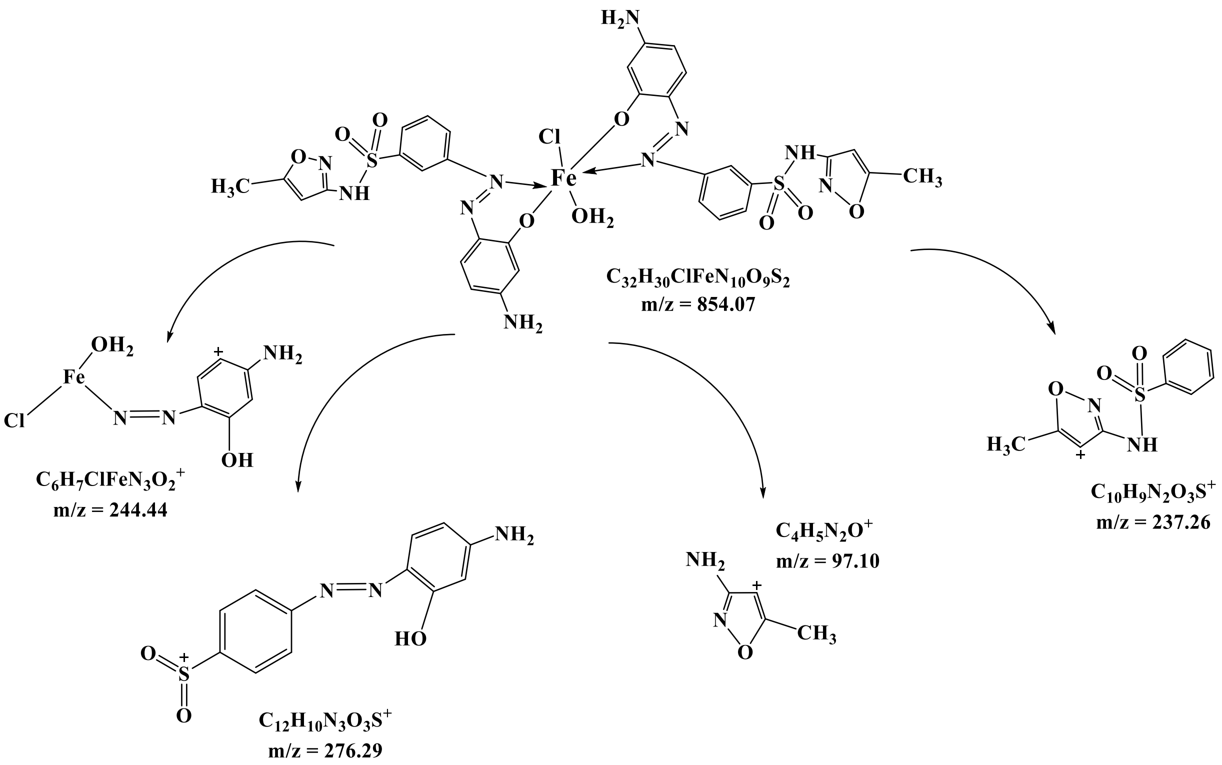
Figure 5. (LC–Mass) spectrum of Fe complex



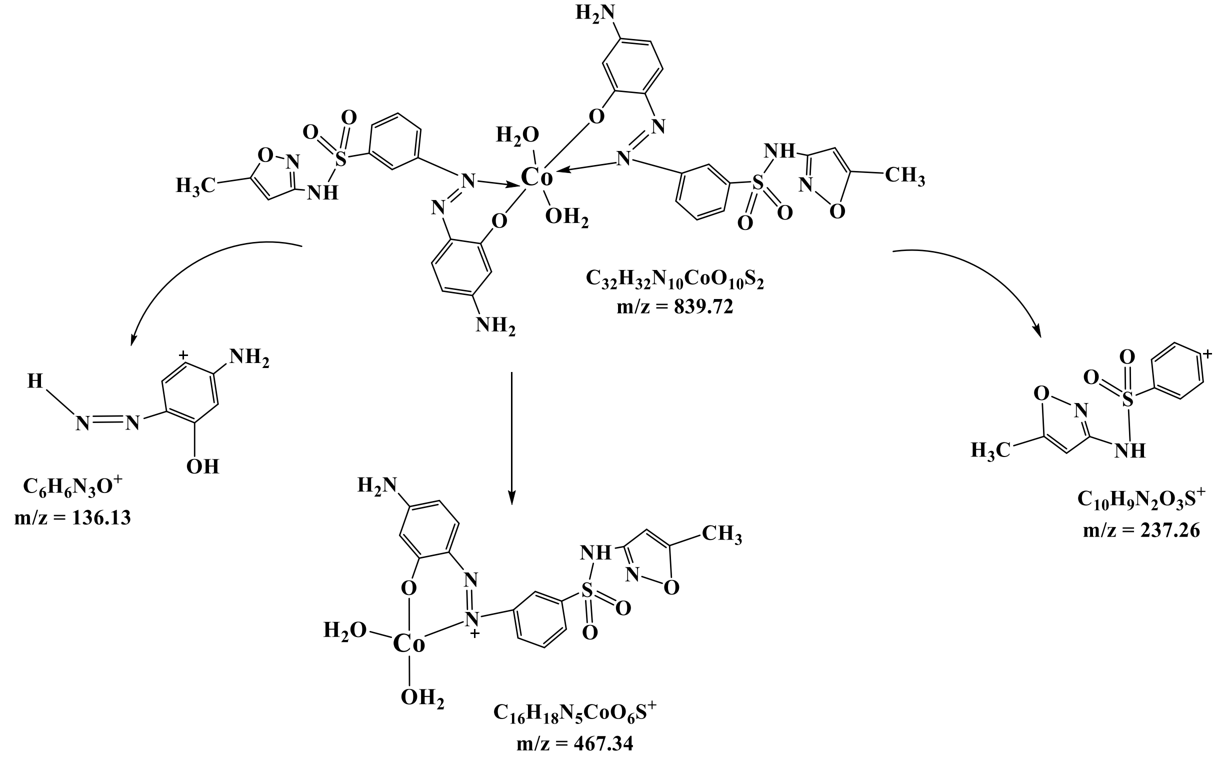
Scheme 2. Fragmentation pattern of ligand



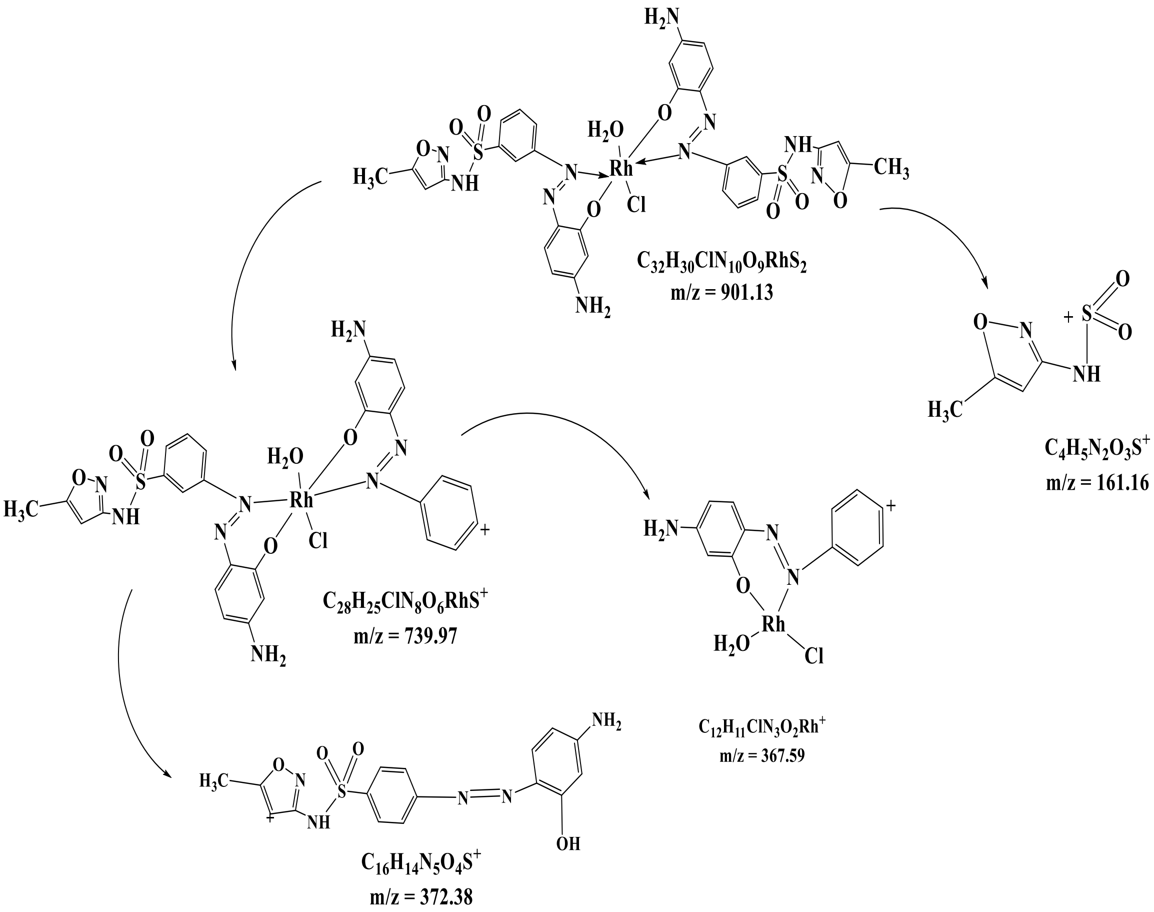
Scheme 3. Fragmentation pattern of Mn complex



Scheme 4. Fragmentation pattern of Fe complex Scheme



Scheme 5. Fragmentation pattern of Co complex



Scheme 6. Fragmentation pattern of Rh complex

Infrared spectra measurements

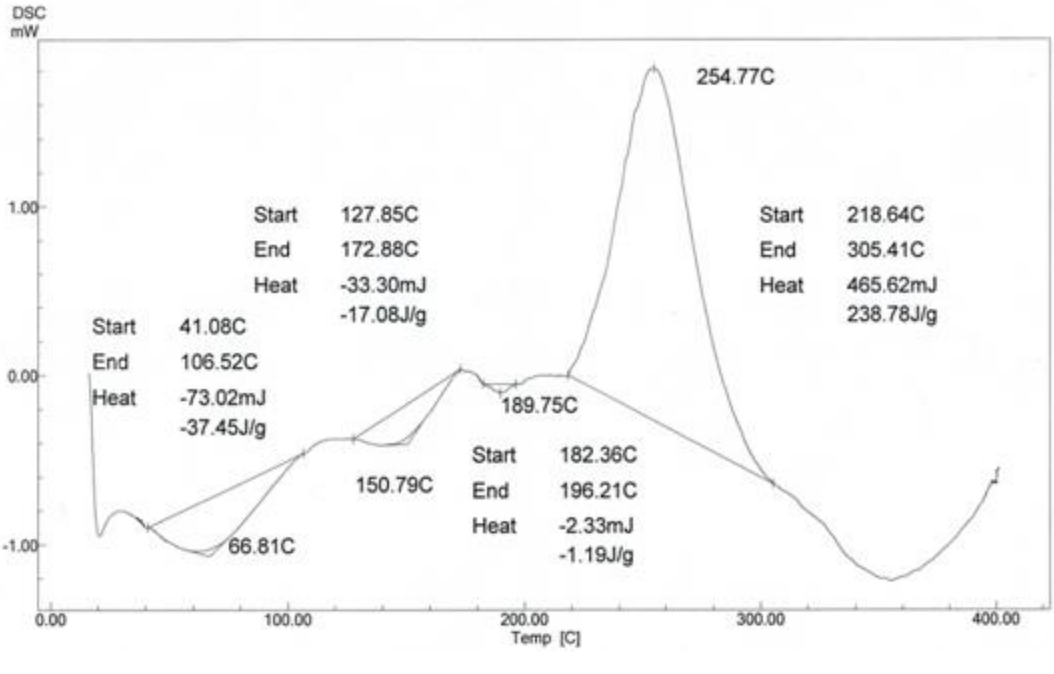
The azo ligand spectra and their metal chelates complexes with Mn(II), Fe(III), Co(II) and Rh(III) have been compiled, and the data has been organized in Table.3. The ligand displayed bands at 3377, 3318and 1618 cm^{-1}that were ascribed to the stretching vibration asυ(NH_2), sυ(NH_2), and out of plane of δ(NH_2), but these bands were reduced to a non lower frequency in the spectra of all generated compounds, indicating not coordination with a metal ion[24]. The (N=N) stretching vibration was attributed to the band seen at 1467, 1406 cm^{-1} [24-26]in the unbound azo ligand (LH). This band was discovered in the compounds' spectra around (1464-1442),(1444-1411)cm^{-1}.The engagement of the azo group in chelation was verified by a change in the azo group of the azo ligand [25-27].In addition, prior study has shown that in the presence of transition metals, the azo-dye nitrogen is always more likely to favor complication [26]. Because of the presence of coordinated water molecules in the complexes, it was difficult to confirm that this group was involved in chelate formation. The existence of coordinated water molecules in the coordination sphere was ascribed to the appearance of OH bands in the IR spectra of the Ni(II) complex in the (3733-3655)cm^{-1}. In addition, stretching vibrations in the range of (1544-1579)and (737-768) cm^{-1} were shown to correlate to ν(M-OH$_2$) in (500-569)cm^{-1} This is a strong evidence that water molecules are involved in the coordination process [27]. For the unbound ligand, the IR spectra revealed a wide stretching vibration band at 3492 cm^{-1}, which might correlate to the OH of the phenolic group[25]. The band at 1284 cm^{-1} was assigned to the ν(C-O) stretching vibration of the phenolic group of the free azo-dye ligand due to the coordination with the metal ions[27], and the band at 1284 cm^{-1}was attributed to the ν(C-O) stretching vibration of the phenolic group of the free azo-dye ligand. After this, the IR spectra of all produced compounds revealed that the azo-dye ligand connected to metal ions through two sites: the azo group's nitrogen site, and oxygen site via deprotonation of the phenolic group [19,22.25,28].As a result, in the all complexes, the ligand behaved as an N, O bidentate ligand.

Table 3. The IR spectra bands (cm^{-1}) of the ligand azo and its complexes

Compounds	(H_2O) aquaν	(NH_2)ν	(NH)ν	ν(C-H) aromatic aliphatic	(N=N)ν	(SO_2)ν	(M-O)ν (M-N)ν
LH	-	3377 3318 1618	3142	3042 2927	1467 1406	1035 1091	-
[Mn(L)$_2$ (H_2O)$_2$]	3733 1579 762	3443 3332 1625	3285	3079 2929	1457 1411	1041 1096	508 569
[Fe(L)$_2$ (H_2O)Cl]	3742 1566 768	3311 3287 1642	3191	3053 2917	1464 1444	1021 1119	500 686
[Co(L)$_2$ (H_2O)$_2$]	3655 1565 737	3330 3267 1615	3174	3098 2922	1442	1078 1159	569 639
[Rh(L)$_2$ (H_2O)Cl]	3739 1544 768	3322 3287 1644	3122	3055 2917	1464 1444	1014 1123	514 669

Study of Thermo Gravimetric Analysis for compounds by TGA & DSC Curve

The results of DSC and TGA of ligand and Fe and Co complexes are given in Table 4 and Fig.6-8. The thermograms have been carried out in the range of 25–700C at a heating rate of 10°C/min in nitrogen atmosphere the ligand showed little thermal stability at 41°C, similar to the little stability complexes in the range of 48.623°C for Fe and 74.433°C for Co complexes, indicating the presence of water molecules in the Co and Fe complexes, whether water hydrate or aqua and as well as DCS the determination of the exothermic and endothermic given in table. 4. They show an agreement in weight loss between their results obtained from the thermal decomposition and the calculated values, which supports the results of elemental analysis and confirms the suggested formulae[28–34] shown in Scheme 6.



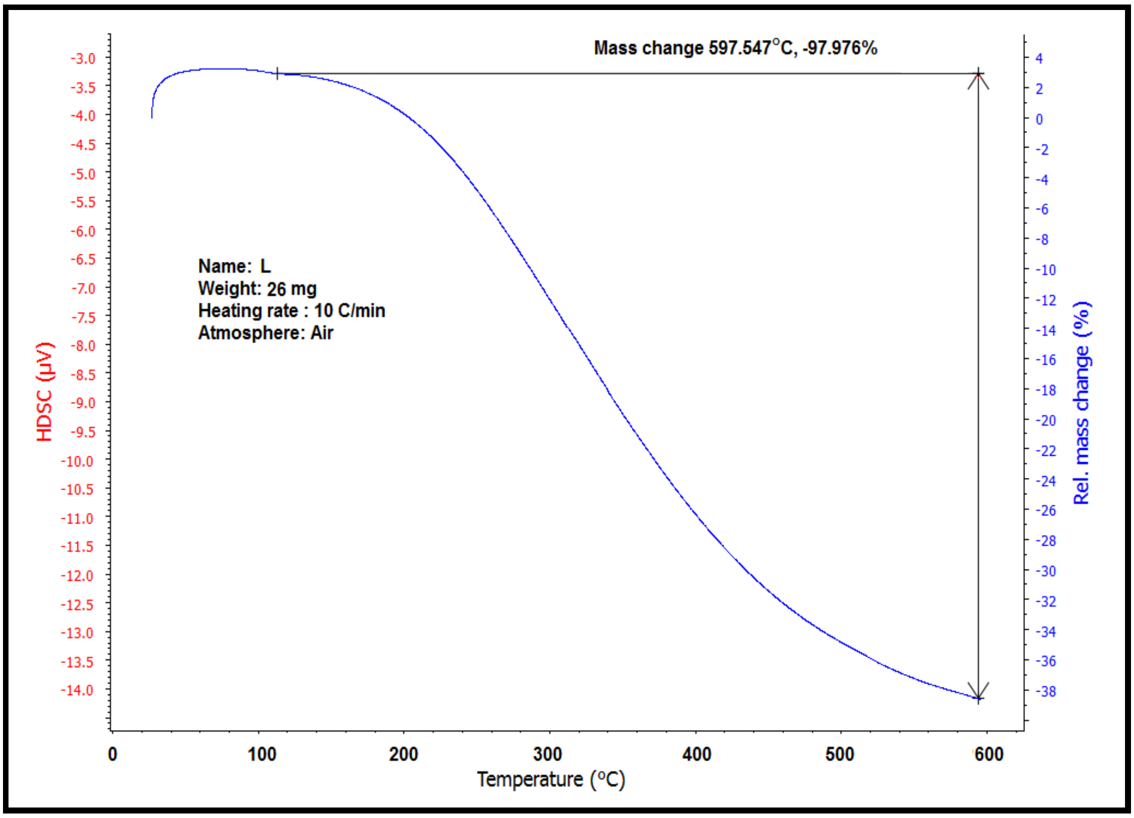
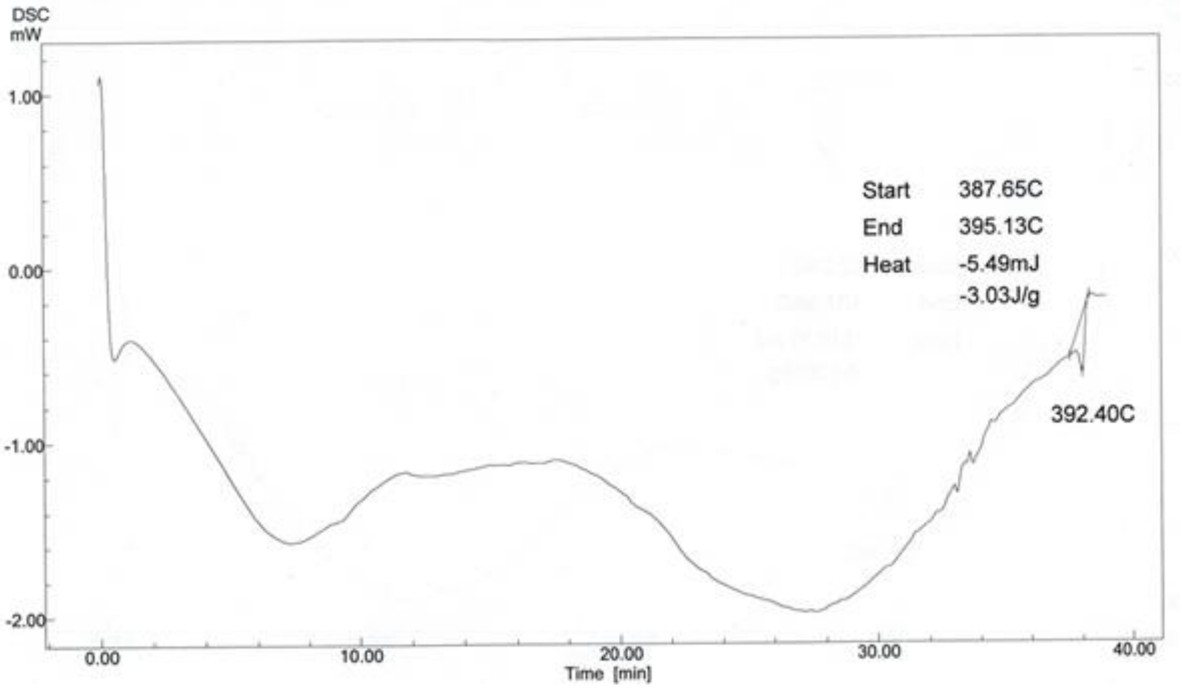


Figure 6. DSC & TGA curve of Ligand



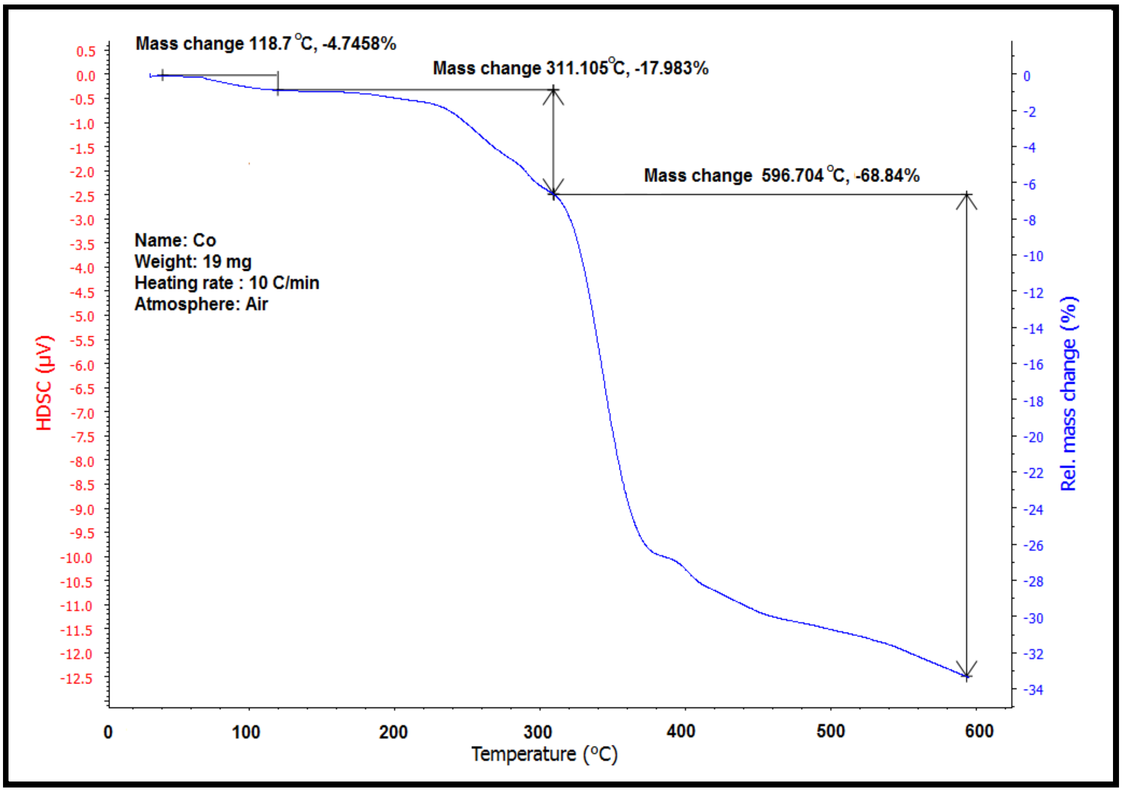
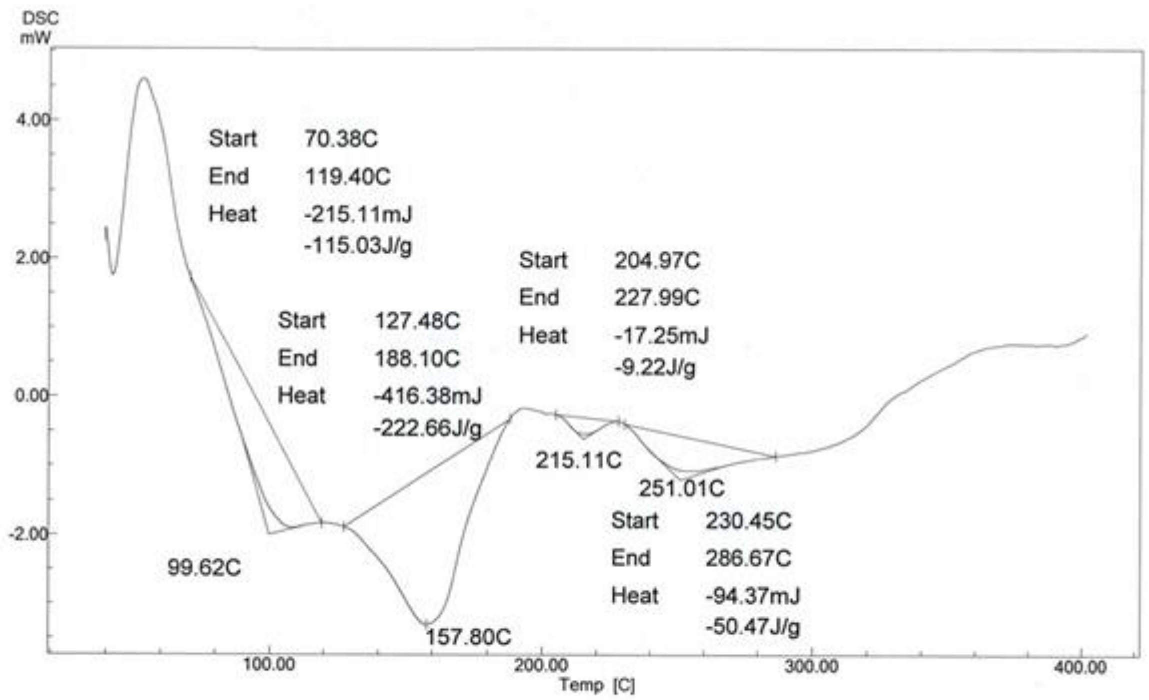


Figure 7. DSC & TGA curve of Co-complex



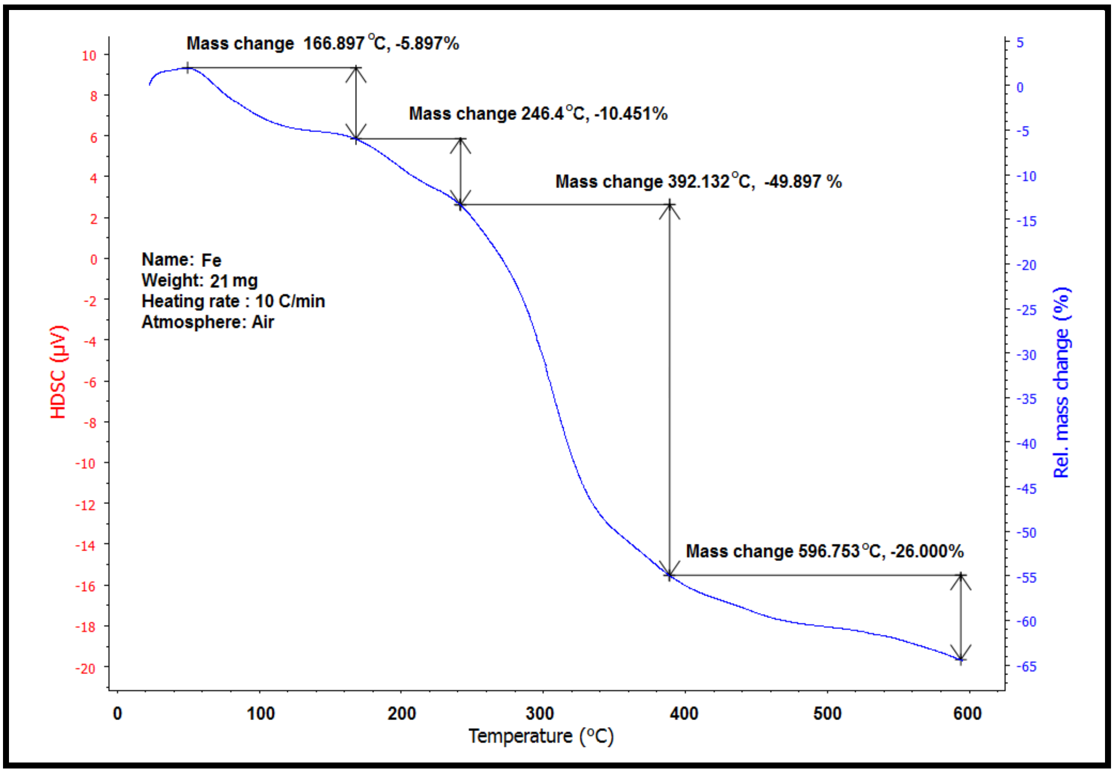
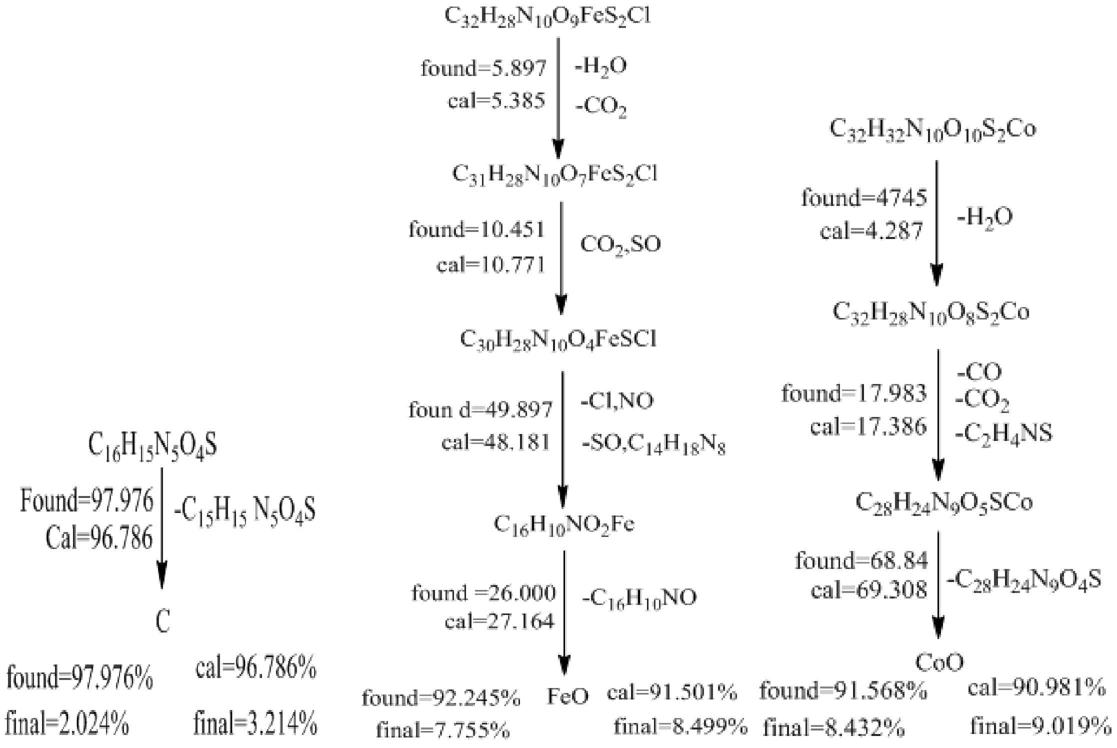


Figure 8. DSC & TGA curve of Fe-complex



Scheme 7. Tentative decomposition reaction of complexes

Table 4. DSC data TGA records for the ligand and its complexes

TGA records							**DSC data**	
Compound	**T_i / °C**	**T_f/ °C**	**T_{DTG} Max**	**% (calculated) Mass loss**	**Estimated Total mass loss**	**Assignment**	ΔH J/g	Maximum temperature point°C and Type
(HL)	119.634	597.547	306.763	97.976 (96.786) 2.024 (3.214)	97.976 (96.786)	- $C_{15}H_{15}N_5O_4S$ C	-37.45 -17.08 -1.19 238.78	66.8-endothermic 150.79-endothermic 189.75-endothermic 254.77-exothermic
[Fe(L)$_2$(H$_2$O)Cl]	48.623	166.897	106.113	5.987(5.386)	92.245 (91.501)	$-H_2O$ -CO	-115.03 -222.66	99.62-endothermic
	166.897	246.4	202.206	10.451(10.771)		$-CO_2$ SO	-9.22 -50.47	157.80-endothermic
	246.4	392.132	298.663	49.897(48.181)		-SO, NO, Cl $C_{14}H_{18}N_8$		215.11-endothermic 251.01-endothermic
	392.132	596.753	483.625	26.000(27.164)		$C_{16}H_{10}N_5$ FeO		
[Co(L)$_2$(H$_2$O)$_2$]	74.433	118.700	93.642	4.745(4.287)	91.568 (90.981)	$-H_2O$	-1.59 -2.10	75.44-endothermic
	118.700	311.700	256.661	17.983(17.386)		-CO, CO_2, C_2H_4N	-3.03	276.34-endothermic
	311.105	596.704	392.293	68.840(69.308)		$-C_{28}H_{24}N_9O_4S$ CoO		392.40-endothermic

Vital diagnosis

The findings showed that the produced ligand and its constituents were biologically efficient, since the experiment was carried out in aerobic circumstances at a temperature of 37°C. Drilling was used to exposing every pathogenic active compound pathogenic bacteria to two different types of pathogenic bacteria *S aureus*, *P aeruginosa*and Fungi*Pexpansum*, *F graminearum*, *M phasealina*, and *C albicans* which were used the DMF solvent with 1×10^{-3}M concentrations and showed different efficacy to the negative and positive stain-bacteria of the compounds. The data are shown in the table 5 below in (mm)[35-38].

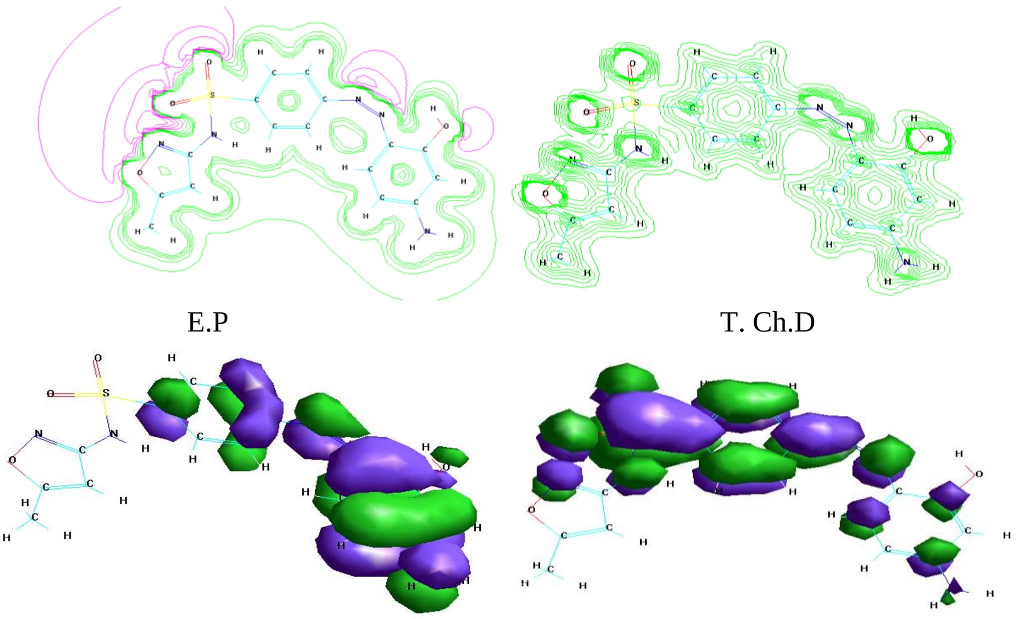
Table 5. vital returns

Compounds	**Positive and negative bacteria**				**Fungi**							
type	*S.T*		*P.A*		*Pe.*		*F.M*		*M.P*		*Ca.a*	
Conc.	A	B	A	B	A	B	A	B	A	B	A	B
MnCl$_2$.4H$_2$O	15	12	16	15	38	28	38	33	—	—	—	—
LH	12	—	12	—	—	—	—	—	—	—	—	—
[Mn(L)$_2$(H$_2$O)$_2$]	16	—	15	—	20	18	28	22	24	16	—	—
CoCl$_2$.6H$_2$O	40	25	23	18	30	18	26	15	0	0	20	10
[Co(L)$_2$(H$_2$O)$_2$]	20	0	15	0	13	0	0	0	25	16	0	0
Control	0	0	0	0	0	0	0	0	0	0	0	0

,S.T =Staphylococcus aureus, P.A= Pseudomonas aeruginosa, Pe.=Penicilliumexpansum, M.P= Macrophominaphaseolina and Candida albicans =Ca.a, A= conc $1*10^{-3}$. B= dilute $1*10^{-6}$.

Study complexes in gas stat (theoretical studies)

The Theoretical study for the formed entity (LH) was accomplished at its gaseous state in order to detect the stretching vibrations and Fourier transform spectra and make a competition for them with the practical returns and detect the mistake percentage, and so forth for the complexes, show as electrostatic potential (HOMO and LUMO) as 2D& 3D Fig.9.



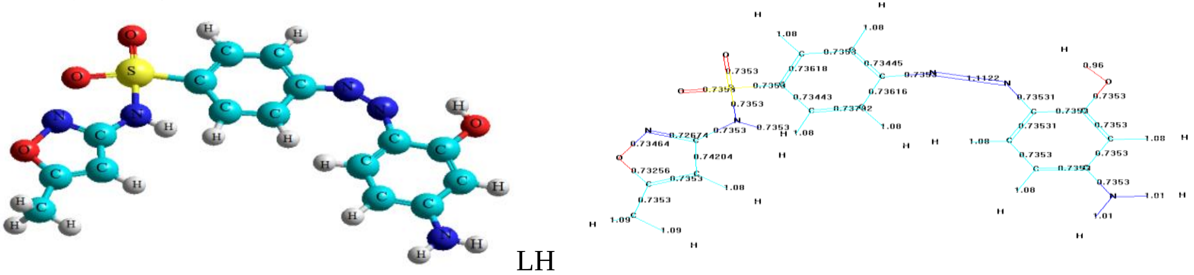
E.P T. Ch.D

HOMO = -8.64499 Ev LUMO = -2.079227 eV

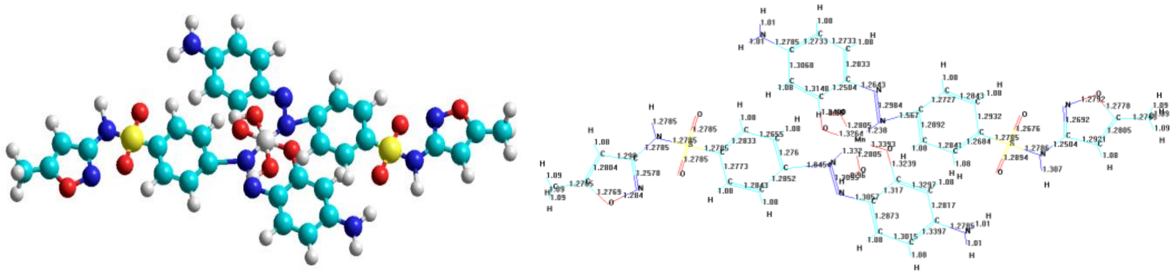
Figure 9. Electrostatic potential (HOMO and LUMO) as 2D& 3D contours for (LH)

Optimized geometries of LH and their complexes

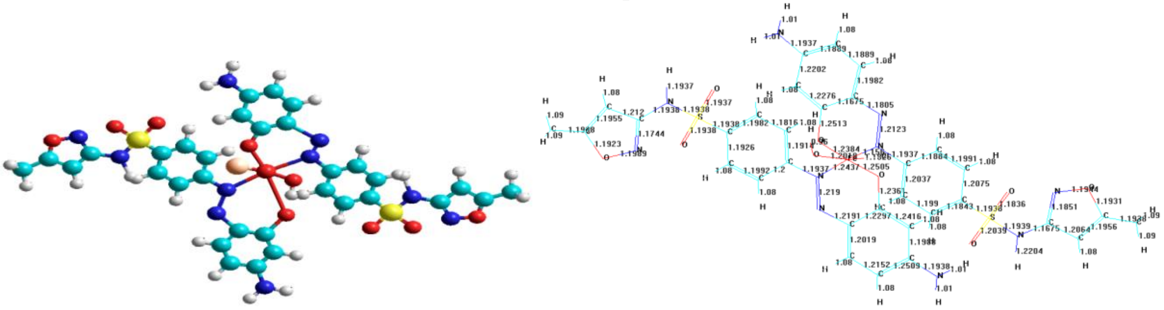
The expected geometric shapes of the prepared compounds were also drawn according to the results of the analyzes Fig.10



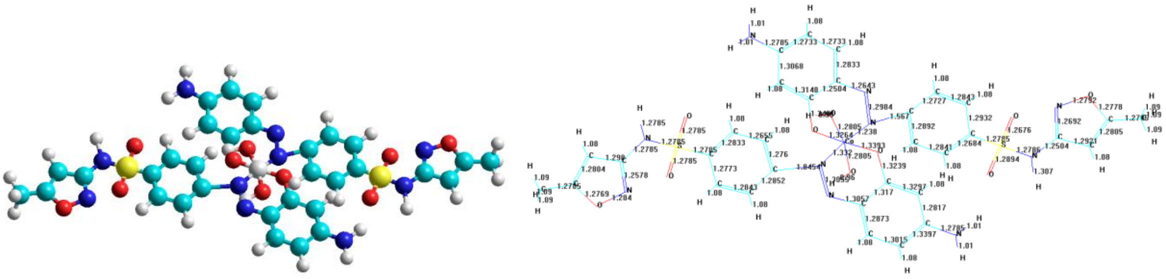
LH



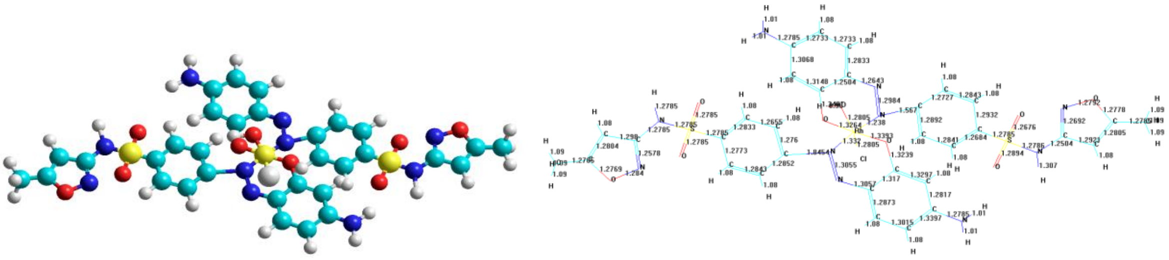
Mn-Complex



Fe-Complex



Co-Complex



Rh-Complex

Figure 10. Conformational structure of (LH) and their metal complexes

Conclusion

The ligand chemical is a brand-new azo dye that has never been made before. ^{1}H &^{13}C-NMR, IR, UV-Vis, TGA, DSC and Mass spectrum techniques were used to identify the ligand and its complexes. The estimated values and the results of the elemental micro analysis were found to be in good agreement. The ligand bidentate character was suggested by IR measurements. Complexation happens through –NO moiety, according to multinuclear NMR data. Complex molecules are more stable, therefore the process requires less equipment to advance. The biological activity of some prepared compounds against two types of bacteria and four type's fungi was also studied, and it gave varying inhibition values.

Author's declaration:

- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Besides, the Figures and images, which are not mine, have been given the permission for re-publication attached with the manuscript
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Authors Contribution:

Al-Hamdani AAS presented the idea, analysis, discussion of the results and writing of the manuscript. Shaimaa Mohammed Reda contributed to the design and implementation of the esearch, laboratory work, verified the analytical methods and discussed the results and contributed to the final manuscript

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معقدات المنغنيز الحديد الكوبلت الروديوم مع ليكاندآزو : تحضير، تشخيص ،تحليل حراري وفعالية حيوية

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الخلاصة:

حضرت سلسلة لمعقدات ايونات المعادن من الليكاند الجديد 4- امينو (5- مثيل-ايكسازول-3- ايل)-بنزينسلفون اميد المشتقة من سلفاميثوكسازول و3- امينو فينول ، المعقدات احادية النوى لكل من المنغنيز الثنائي والحديد الثلاثي والكوبلت الثنائي والروديوم الثلاثيمن تفاعل الليكاند مع املاح ايونات المعادن. المركبات شخصت بأطياف تحت الحمراء وفوق البنفسجية –المرئية و الكتلة و الرنين النووي المغناطيسي للبروتون والكاربون ومنحنى التحلل الحراري الوزني والمسعر الحراري التفاضلي والحساسية المغناطيسية والتحليل الدقيق للعناصر ونسبة الايون ومحتوى الكلور والتوصيلية المولارية وفقا للدراسات التحليلية اعطت المعقدات شكل ثماني السطوح(1:2) (ليكاند : فلز) وبينت الدراسات الحرارية ان المعقدات تحوي جزيئات متناسقة.

الكلمات المفتاحية: معقدات الازو, فعالية حيوية, مطيافية الكتلة, دراسات طيفية , التحليل الوزني الحراري.