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Synthesis, Characterization and Bioactivities of V(III),Cr(III),Fe(III),Rh(III)and Al(III) Complexes with Bis(Salicylaldehyde)MalonylDihydrazone and 8-HydroxyQuinoline as Mixed Ligands

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Abstract

A new mix ligand complexes were synthesized using the prepared Schiff base **(BM)** as a primary ligand and 8-hydroxy quinoline(HQ**.)** as a secondary ligand with metal ions [V(III), Cr(III), Fe(III), Rh(III) and Al(III)] .These complexes were characterized by (FT-IR and Uv-Vis) Spectroscopies , (elemental analysis and flame atomic absorption) techniques as well as (magnetic susceptibility and conductivity) measurements. showed that the metal ions was coordinated with **(BM)** by nitrogen atom of azomethene groups and oxygen atom of hydroxyl groups , in addition of coordination of **(**HQ**.)** by oxygen atom of hydroxyl group and nitrogen atom of the quinoline ring. The antibacterial activity for ligands (**BM** and **HQ.)** and mixed ligand complexes were studied against two selected micro-organisms **(Escherichia coil) and (staphylococcus aureus).**The antifungal activity against two micro-organism **(penicillumSpp** and **Aspergillusflavus**) were studied for ligands (**BM** and **HQ.)** and mixed ligand complexes . The results showed great enhancement of activity of the all complexes relative to that of their respective ligands.

Keywords:8-hydroxy quinolone,Schiff base, Antimicrobial activity, Spectroscopic studies.

تحضير وتشخيص ودراسة بايولوجية لمعقدات V (III) وCr(III) وFe(III) وRh(III) وAl(III) مع مزيج ليكاندات بس (سالسالديهايد) مالونايل ثنائي هايدرازون و 8- هيدروكسي كوينولين

رحاب عبد المهدي الحسني وانعام مجيد رشيد وسنان مدحت البياتي

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الخلاصة

تم في هذا البحث تحضير معقدات مختلطة الليكاند باستخدام قاعدة شف المحضرة (BM) كليكاند اولي و٨-هيدروكسي كوينولين(HQ.) كاليكاند ثانويمع الايونات الفلزية [V(III) وCr(III) وFe(III) وRh(III) وAl(III)].تم عزل المعقدات المختلطة الليكاند في الحالة الصلبة وتشخيصها باستعمال طيف الاشعة تحت الحمراء(FT-IR) وطيف الاشعة فوق البنفسجية والمرئية(Uv-Vis) والتحليل الدقيق للعناصر(C.H.N.) والتحليل الكمي للعناصر باستعمال تقنية الامتصاص الذري اللهبي، فضلا عن قياسات الحساسية المغناطيسية والتوصيلية الكهربائية. واستنادا الى نتائج هذه التشخيصات التي بينت ان الايونات الفلزية تتناسق مع (BM) عن طريق ذرة النتروجين لمجموعتي الأزوميثين وذرة الأوكسجين لمجموعتي الهيدروكسيل فضلا عن تناسقها بال (HQ.) عن طريق ذرة الاوكسجين لمجموعة الهيدروكسيل وذرة النتروجين لحلقة الكوينولين. تم تقويم الفعالية المضادة للبكتريا لليكاندات (BM و.HQ) ومعقداتها المحضرة لمختلطة، وأختير نوعان من البكتريا هما (*Escherichia coli*) و(*Staphylococcus aureus*) لهذا الغرض، كما تم إجراء التقويم الحيوي لليكاندات (BM و.HQ) ومعقداتها المحضرة المختلطة الليكاند المضادة للفطريات على نوعين من الفطريات هما (*Panicillum Spp.*) و(*Aspergillusflaveus*)، وقد أظهرت نتائج التقويم زيادة كبيرة في فعالية المعقدات بالمقارنة مع الليكاندات المحضرة منها.

Introduction

During the last fifteen years, synthetic organic chemistry has been enormous growth, not only in terms of development of new methodologies for construction of carbon-carbon and carbon-hetero atom bonds but also in terms of development of new strategies, reagents, catalysts, transformations and technologies(1-3). From the last survey appears that Schiff bases have played a vital role in developing the synthetic organic chemistry(4-7). The presence of nitrogen atom along with other features may impart interesting biological activities to the parent compound(8,9).The essential feature of Schiff base reaction is the condensation of aldehyde or ketone with a primary amine to produce the imine compoundwhich are important synthetic intermediates for various pharmaceuticals and natural products(10). It is well-known from the earlier work that the compounds containing imine moiety as functional group have been found to possess donor properties and exhibit a wide range of biological activities, literature study also reveals that a broad spectrum of biological activity is reported to be associated with a number of heterocyclic compounds(9,11). Metal complexes of Schiff bases have been studied extensively in the recent years due to the selectivity and sensitivity of the ligands towards biological important metal ion(11). The present work aim to synthesis new mixed ligand complexes of Schiff base bis(Salicylaldehyde)malonyldihyd -razone(BM) and 8-hydroxy quinoline(HQ.) to investigate the coordination behavior of the ligands toward some trivalent metal ions[V(III), Cr(III), Fe(III), Rh(III) and Al(III)], then compare the biological activities of ligands with their metal complexes.

Experimental:

All the chemicals were of highest purity and were used as received.

Physical Measurements and Analysis:

Melting points were recorded on Gallen Kamp melting point apparatus and were uncorrected. FTIR spectra were recorded on shimadzu-8300 spectrophotometer in the range of (4000-400)cm^{-1}. Uv-vis. Spectra were measure using UV-1650 PC shimadzu spectrophotometer at room temperature. The measurements were recorded using a concentration of 10^{-3}M of the complex in absolute ethanol as solvent. Elemental C.H.N.S analysis was carried out on an EM-017mth instrument. Atomic absorption measurement of the prepared complexes were obtained using shimadzu atomic absorption 680 flame spectrophotometer. The conductivity measurements were recorded on W.T.W. conductivity meter. These measurements were obtained in DMF as solvent using concentration of 10^{-3} M at 25 ° C. Magnetic susceptibility measurements were applying obtained at 25 ° C on the solid state applying obtained at 25 ° C on the solid state applying Gouy using magnetic susceptibility balance by Sherwood scientific.

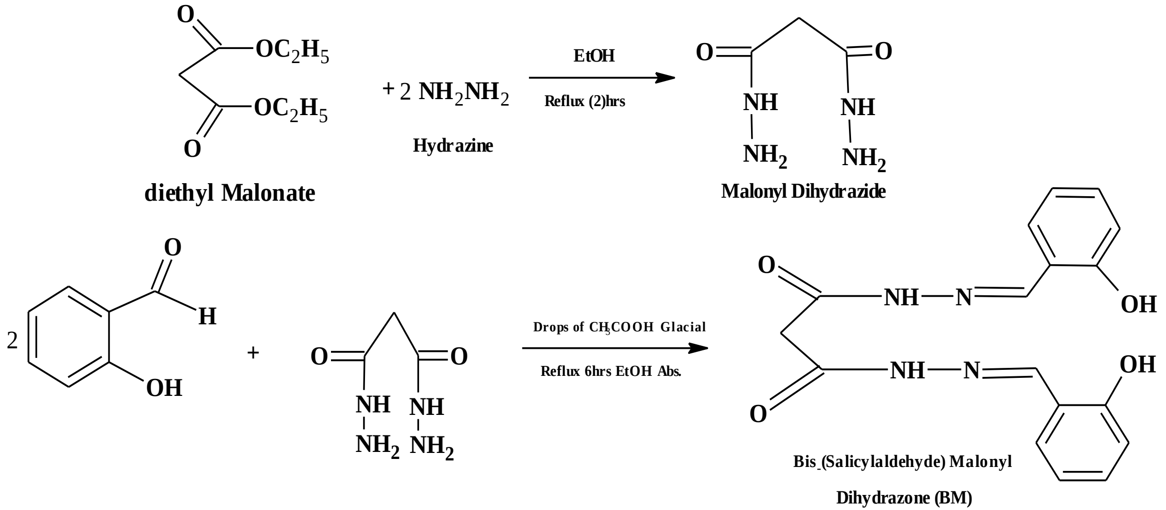
1-Synthesis of ligand:

A-Synthesis of Malonyldihydrazide(12).

Malonyldihydrazide was prepared by reacting the diethyl malonate (0.1mole,1.69g) with hydrazine hydrate (0.02mole,1g) in 20 ml ethanol. The mixture was refluxed for (2hrs). The isolated compound was filtered off as white crystal, washed and recrystallized from absolute ethanol.

B-Synthesis of Bis–(salicyladehyde) Malonyldihydazone(BM)(13).

The ligand (BM) was prepared by reaction a warm dilute ethanol solution of Malonyldihydrazide (0.01mole,1.32g) with salicyladehyde (0.02mole,2.44g). The reaction mixture was refluxed for (6hrs) and cooled to room temperature. The product was collected and crystallized from ethanol. The physical properties of prepared ligand is described in table(1), scheme (1).



Scheme (1)

2-Synthesis of mixed ligand complexes:

An ethanolic KOH solution of ligand (BM) (1mmole,0.34g) and an ethanolic KOH solution of 8-hydroxy quinoline(HQ) (1mmole,0.145g) were added respectively to ethanolic solution of the metal salts (1mmole) [$AlCl_3.6H_2O$,VCl_3,$CrCl_3.6H_2O$,$FeCl_3$

and $RhCl_3.xH_2O$].The mixture was heated under reflux for 2hrs.during this time precipitate was formed. The precipitates were filtered off and washed with hot ethanol.

The physical data of the prepared complexes are shown in table(1).

Results and Discussion

The elemental analysis and atomic absorption show (1:1) metal :Ligand stoichiometry for the prepared complexes. The analytical data together with some physical properties of the complexes are summarized in table(1). The isolated solid complexes are stable at room temperature and soluble in dimethyl formamide (DMF). The molar conductance values of the complexes indicating that all complexes are nonelectrolyte, table(3).

Table(1):Some Physical characteristic and analytical data for (BM),(HQ) and its metalcomplexes.

Comp.	**Color**	**M.P ◦C**	**Mol. Formula**	**Elemental analysis found (Calc.)**			
				C%	**H%**	**N%**	M%
$C_{17}H_{16}N_4O_4$(BM)	**Pale white**	**215**	**340**	**61.02 (60.36)**	**4.45 (4.14)**	**16.83 (16.57)**	---
C_9H_7NO(HQ)	**White**	**73**	**145**	---	---	---	---
[Al(BM)(HQ)]	**Light yellow**	**350d***	**509**	**61.82 (61.54)**	**4.01 (3.93)**	**14.11 (13.81)**	**5.75 (5.33)**
[V(BM)(HQ)]	**Brown**	**250d***	**533**	**58.99 (58.76)**	**3.67 (3.39)**	**13.64 (13.18)**	**9.28 (9.60)**
[Cr(BM)(HQ)] H_2O	**Deep orange**	**230d***	**552**	**57.21 (56.73)**	**3.74 (3.27)**	**13.03 (12.72)**	**9.82 (9.45)**
[Fe(BM)(HQ)]H_2O	**Dark brown**	**300d***	**556**	**56.81 (56.32)**	**3.69 (3.25)**	**13.11 (12.64)**	**10.65 (10.07)**
[Rh(BM)(Q)] H_2O	**green**	**240d***	**603**	**52.12 (51.74)**	**3.13 (2.99)**	**12.22 (11.61)**	**16.59 (17.08)**

BM=Bis-(Salicyladehyde) Malonyldihydrazone.

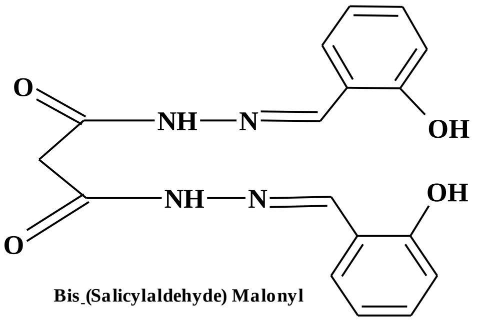
HQ=8-hydroxy quinolone

d^*=decomposition

H^1NMR Spectra of (BM) ligand:

The nuclear magnetic resonance spectra of the synthesized ligand (BM),fig(1) in d^6DMSO solution with tetraethyl silan is regarded as internal reference.

According to the results (14) obtained from the chemical shifts spectra the molecular structure of the ligand[BM] can be illustrated as follows:



Bis_(Salicylaldehyde) Malonyl Dihydrazone (BM)

δH=(11-88)ppm (2H,s,NH)

δH=(10.81)ppm (2H,s,OH)

δH=(8.9)ppm (2H,s,CH)

δ ph=(6.9-7.38)ppm (8H,s,m)

δH=(4.16)ppm (2H,s,CH_2)

Infra red spectra studies:

The chain characteristic vibrations and assignments of ligand (BM) and (HQ) and their complexes are described in table(2).The spectrum of ligand (BM) exhibited bands at (3281 ,3188 and 3070) cm^{-1} were attributed to v(OH) group of phenolic and v(N-H) and v(C-H) aromatic respectively(14). While the strong band at 1678cm^{-1} which belongs to v(C=O) and the other bands belong to the v(C=N),v(C=C) and v(C-O) were found at 1612cm^{-1}, 1487cm^{-1} and 1265cm^{-1} respectively(15,16),fig.(2).

In the spectrum of ligand (HQ) it was noticed that the broad band at 3249cm^{-1} which could be attributed to v(OH) with shoulder at 3066cm^{-1} due to v(C-H) aromatic. The other strong bands are appeared at 1577cm^{-1}, 1506cm^{-1} and 1284cm^{-1} which attributed to v(C=N),v(C=C) and v(C-O) respectively(17,18),fig.(2).

B. Infra-red spectroscopy of complexes:

The infra-red spectra of the prepared complexes exhibited ν(C=N) in the range of (1537-1599) cm^{-1} which shows a shifting to the lower frequencies in compared with ligand (BM): Also appeared shifting to the higher and lower frequencies in compared with ligand (HQ), it is which indicated the coordination of ligands with metal ions through the nitrogen atoms in their structures.

The other bands appeared in the range of (1193-1246) cm^{-1} which belongs to the ν(C-O). It was shifted to the lower frequencies in compared with ligands (BM) and (HQ) respectively. The disappearing of band for ν(OH) vibration in the spectra of complexes is indicating the coordination of phenolic oxygen with metal ions(19,20).

The bands of complexes at 1678cm^{-1} were characterized for carbonyl group which didn`t suffer a shift. It is suggested that the oxygen atom of the carbonyl group is not coordinated to the metal ion(21).

The bands at (3404,3360 and 3358)cm^{-1} can be assigned to the stretching vibration of (OH) of water molecules for Cr(III), Fe(III) and Rh(III) complexes respectively(22). The result led to a suggestion of presence of water molecules in Cr(III), Fe(III) and Rh(III) complexes(23), Figure (3) showed IR spectra of Rh(III) complex, fig.(3).

C-M-Ligand Bond:

The infrared of prepared complexes have shown weak bands in the range of (439-478)cm^{-1} and (505-590)cm^{-1} which was attributed to ν(M-O) and ν(M-N) respectively(23).

Table (2): Characteristic stretching vibrational frequencies (cm^{-1}) located in the FT-IR mixed ligands and its metal complexes.

Comp.	ν(O-H)phenolic	ν(O-H)water	ν(C=N)	ν(C-O)	ν(M-N)	ν(M-O)
$C_{17}H_{16}N_4O_4$(BM)	3281	---	1612	1265	---	---
C_9H_7NO(HQ)	3249	---	1577	1284	---	---
[Al(BM)(HQ)]	---	---	1599	1209	565	452
[V(BM)(HQ)]	---	---	1579	1207	528	439
[Cr(BM)(HQ)] H_2O	---	3404	1537	1193	530	478
[Fe(BM)(HQ)]H_2O	---	3360	1597	1205	505	447
[Rh(BM)(Q)] H_2O	---	3358	1573	1246	590	444

Electronic spectra, Magnetic moment and conductance studies:

The UV spectrum of free ligand (BM) mostly showed two intense maxima bands at 279nm and 321nm which belong to п→п* and n →п* respectively (24), while the Uv spectrum of 8-hydroxy quinoline (HQ) showed intense bands at 268nm and at 302nm which belong to п→п* and n→п* respectively, Table (3), fig.(4).

According to the electronic spectra of Al(III) complex no d-d transition is located as it belongs to d◦. The prepared complex is off white in color with the diamagnetic being expected, therefore the Uv-vis spectra of this complex show a relative change in the bands position compared to that of free ligands (BM and HQ) as listed in table(3) due to charge transfer between Al(III) ion and ligands. The conductivity measurement indicate nonionic conducting behavior of the complex, table(3). Thus , from the data above and those obtained from FTIR spectra and flame atomic absorption, an octahedral geometry around Al (III) ion(24).

The prepared brown V(III) complex showed two band at(13888,23255)cm^{-1} due to the transition ${}^3T_1g(F) \overset{v_1}{\rightarrow} {}^3T_2g$), ${}^3T_1g(F) \overset{v_2}{\rightarrow} {}^3T_1g(P)$ these assignment when compared to those published for octahedral geometry(25). In addition the measured magnetic for this complex was found to be (2.72BM) and the conductivity measurement in (DMF) showed a non-conductive behavior of the complex table(3).

The deep orange chromium complex gave three absorption band appeared at (17543,25575,34364)cm^{-1} following octahedral filed with(d^3) configuration(26). The effective magnetic moment at room temperature was found to be (3.77)B.M revealing inner-orbital high-spin octahedral stereo chemistry of the ligand around Cr(III) metal ion (27). Conductivity in (DMF) showed that complex was to be nonionic, table(3).

The observance of d→d bands in the Mull transition spectrum is consistent with low-spin Iron(III) center since high – spin iron(III) complexes have only very weak d→d transition(28).The band 22883cm^{-1} is assignable to ${}^{6}A_{1}g \overset{v_1}{\rightarrow} {}^{4}T_{2}g$ transition and 13157cm^{-1} is assignable to${}^{6}A_{1}g \overset{v_2}{\rightarrow} {}^{4}T_{2}g$ transition, the magnetic moment value was found to be (1.72)B.M which is also agree well with the known value, for Fe(III) complex in octahedral geometry(29), the conductivity measurement in (DMF) showed a non-conductive behavior of the complex as shown in table(3) .

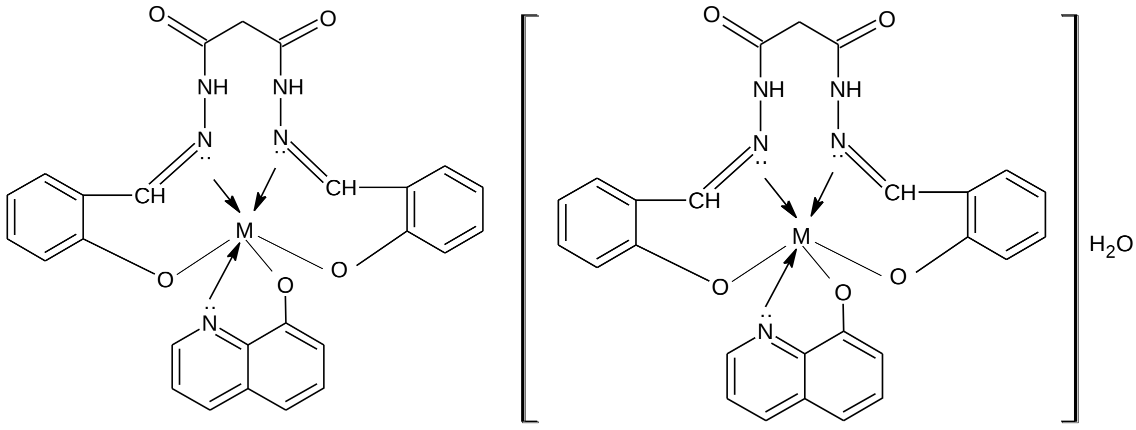
The solution spectrum of the Rh(III) green complex give weak band of 18518cm^{-1}and a shoulder at 23809cm^{-1}, these two bands correspond to those of octahedral Rh(III) complex(30) and were assigned to $({}^{1}A_{1}g(F) \overset{v_1}{\rightarrow} {}^{3}T_{1}g)$ and ${}^{1}A_{1}g \overset{v_2}{\rightarrow} {}^{1}T_{2}g$ transitions respectively the magnetic moment of the solid complex (2.07)B.M found to be paramagnetic. Conductivity measurement in DMF showed a non-conductive behavior of the complex table (3),Fig.(5) showed electronic spectra of Rh complex.

Table (3): Uv-Visible spectra of free ligand and their complexes in 10^{-3} M in DMF:

Comp.	Comp. No.	λ_{max}	Bands cm^{-1}	Assignment	Molar Cond. µs.cm^{-1}	μ_{eff} (B.M)	Structure
$C_{17}H_{16}N_4O_4$(BM)	L	279 321	35842 31152	$\pi\rightarrow\pi^*$ $n\rightarrow\pi^*$	-	-	-
C_9H_7NO(HQ)	C_1	268 302	37313 33112	${}^{4}A_{2}g\overset{v_1}{\rightarrow}{}^{4}T_{1}g$ ${}^{4}A_{2}g\overset{v_2}{\rightarrow}{}^{4}T_{2}g$	75.32	3.78	o.h
[Al(BM)(HQ)]	C_2	330 338	30303 29586	${}^{6}A_{1}g\overset{v_1}{\rightarrow}{}^{4}T_{2}g$ ${}^{6}A_{1}g\overset{v_1}{\rightarrow}{}^{4}E_{1}g$	14.59	5.12	o.h
[V(BM)(HQ)]	C_3	720 430	13888 23255	${}^{4}T_{1}g(F)\overset{v_1}{\rightarrow}{}^{4}T_{1}g(F)$ ${}^{4}T_{1}g(F)\overset{v_1}{\rightarrow}{}^{4}A_{2}g(F)$	169.7	4.81	o.h
[Cr(BM)(HQ)] H_2O	C_4	570 391 291	17543 25575 34364	${}^{3}A_{2}g\overset{v_1}{\rightarrow}{}^{3}T_{1}g(P)$ ${}^{3}A_{2}g\overset{v_1}{\rightarrow}{}^{3}T_{1}g$ ${}^{3}A_{2}g\overset{v_1}{\rightarrow}{}^{4}T_{2}g$	13.62	2.8	o.h
[Fe(BM)(HQ)]H_2O	C_5	760 437	13157 22883	${}^{2}Eg\overset{v_1}{\rightarrow}{}^{2}T_{2}g$	16.35	1.81	o.h
[Rh(BM)(Q)] H_2O	C_6	540 420	18518 23809	L→ M(C.T)	20.3	Zero	T.h

Suggested stereo chemistry structure for Schiff base (BM) and 8-hydroxy quinolone (HQ) and their mixed ligands metal complexes:

According to the results obtained from elemental and spectral analyses as well as magnetic moment and conductivity measurements, the suggested structure of the above mentioned compounds can be illustrated as follow, fig.(1):



M= Al(III) and V(III)

M=Cr(III) , Fe(III) and Rh(III)

Fig.(1): Suggested Stereo Chemical structure for Mixed ligands metal complexes

Biological activities:-

The in vitro biological screening effects of the investigated compounds were tested against selected types of bacteria which include ***(Escherichia coil)*** and ***(Staphylococcus aureus)***and the fungus, ***(Penicillum Spp.)*** and ***(Aspergillusniger)*** by the Well Diffusion Method(31) using agar nutrient as the medium. Stock solutions (10^{-3}M) were prepared by dissolving the compounds in DMSO solution. In a typical procedure, a well was made on the agar medium inoculated with microorganisms. The well was filled with the test solution using a micropipette and the plat was incubated at 35°C for 72 hrs. During this period, the test solution diffused and the growth of the inoculated microorganisms was affected(32,33).

Table(4) Antibacterial and antifungal activities for Schiff base and 8-hydroxy quinolineas mixed ligands and their metal complexes.

Comp. No.	***E. coli***	***Staph. Aureus***	***Asp. Flavus***	***Penci. SPP.***
Control DMSO	-	-	-	-
[BM]	**6**	**6**	**33**	**36**
[HQ.]	**0**	**6**	**29**	**38**
[Al(BM)(HQ.)]	**8**	**6**	**24**	**28**
[V(BM)(HQ.)]	**6**	**10**	**17**	**19**
[Cr(BM)(HQ.)]H_2O	**8**	**12**	**18**	**20**
[Fe(BM)(HQ.)]H_2O	**10**	**16**	**20**	**18**
[Rh(BM)(HQ.)]H_2O	**8**	**10**	**22**	**20**
	Where:- **(+) 6-8** **(++)8-10** **(+++)10>**		**Where:-** **(+++) 30-40** **(++++)20-30** **(+++++)10-20**	

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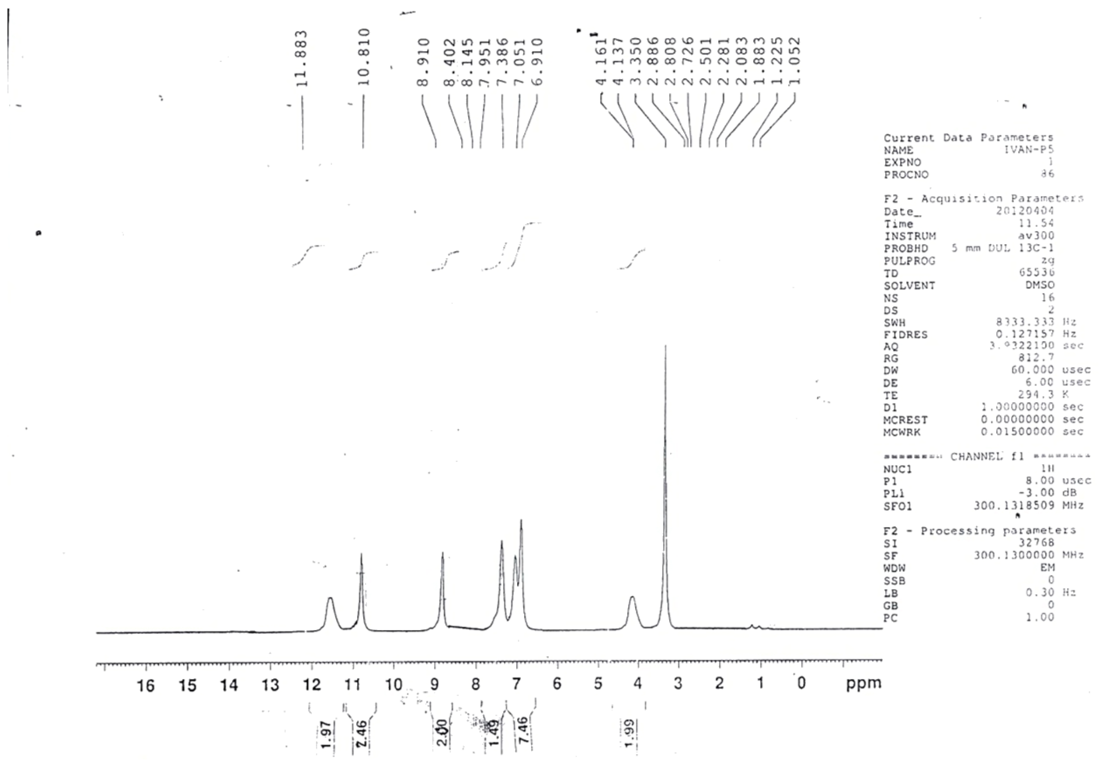

Figure (1): H^1NMR Spectrum of The ligand (BM)

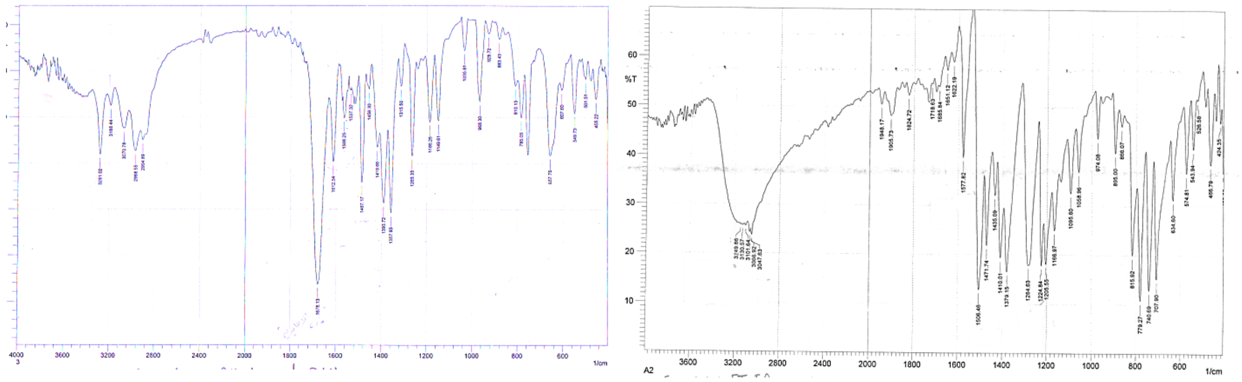

Figure (2): FTIR Spectrum of the Ligand (BM) and(HQ)

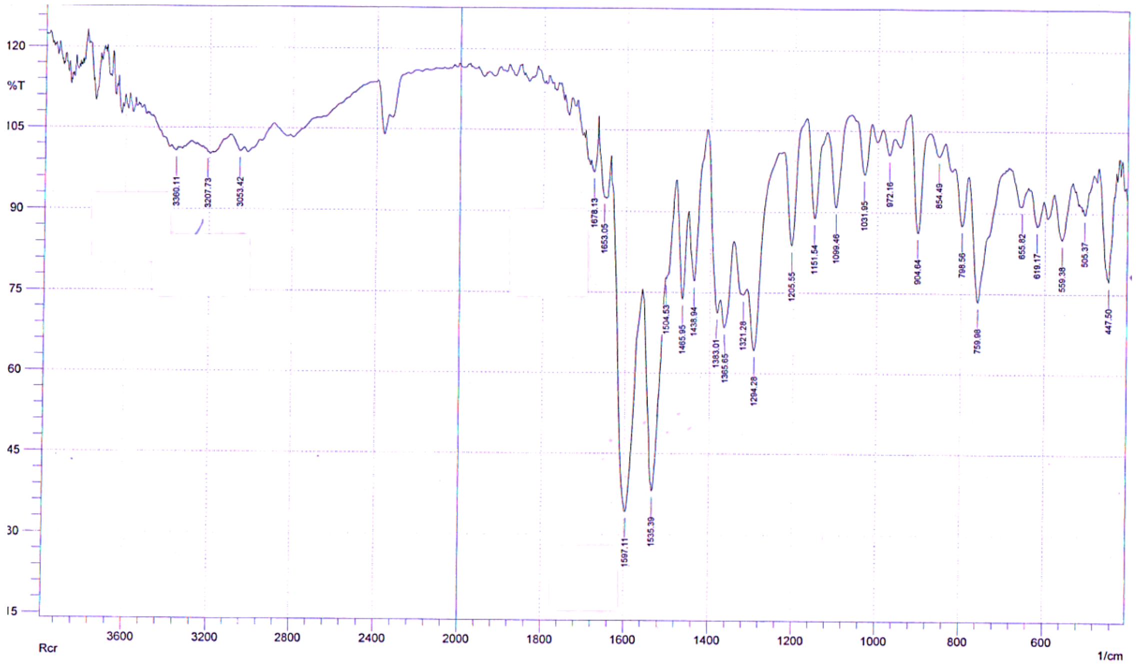

Figure (3): FTIR Spectrum of Rh (III) complex

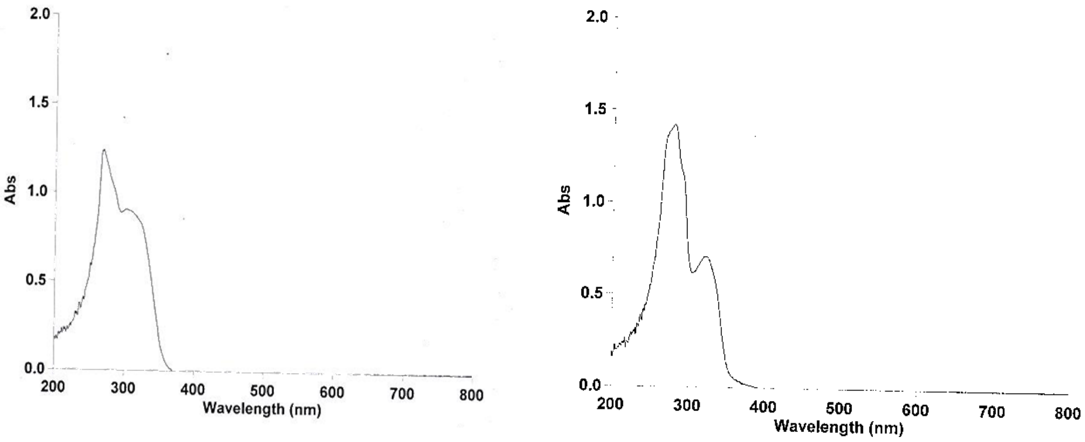

Figure (4): Uv-visible Spectrum of the Ligand (BM) and (HQ)

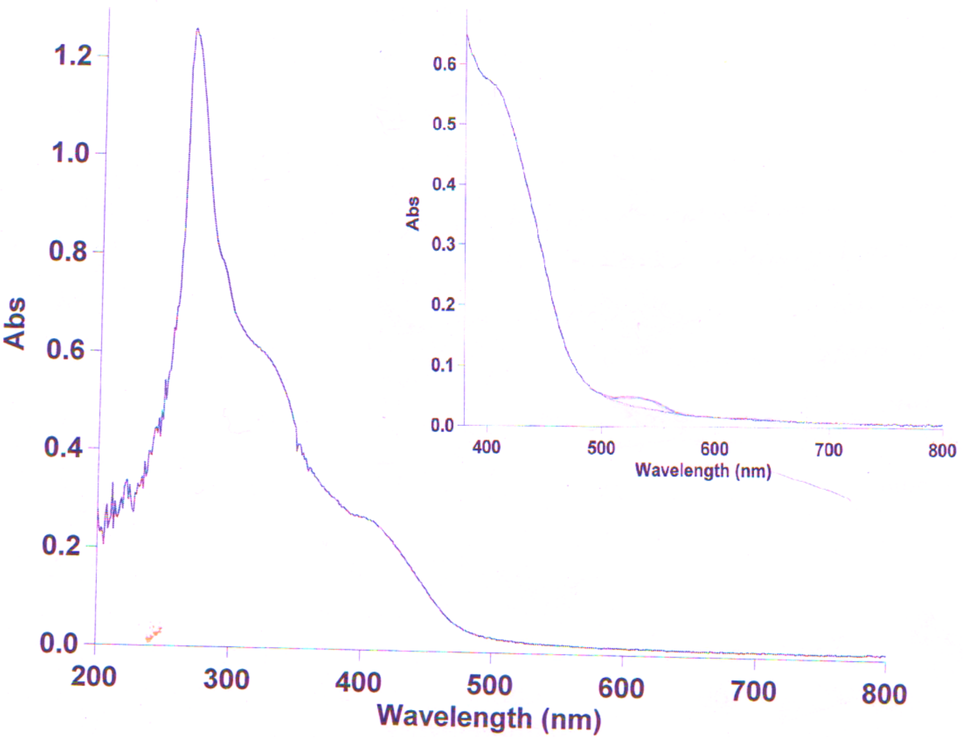

Figure (5): Uv-visible Spectrum of Rh (III) complex