



## Preparation and characterization of new mixed complexes of some transition elements with tyrosine and 8-hydroxyquinoline.

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### ABSTRACT

A new group of metal compounds with the formula  $[\text{Co}(\text{tyr} \& \text{8-H})_2(\text{H}_2\text{O})_2]\text{Cl}_2$  was prepared by reacting 8-Hydroxyquinoline & Tyrosine and selected manganese (II), cobalt (II), and nickel (II) ions (II), and Cu(II). As for the compounds that were diagnosed through the UV-visible spectrophotometer, TGA, and the infrared device. These diagnoses refer to the bonding of the bi-dental metal ion via nitrogen atoms ((amide group)).

**Keyword:** 8-Hydroxyquinoline, Biological activity, Tyrosine

### INTRODUCTION

The organic compound hydroxyquinoline has the atomic formula  $\text{C}_9\text{H}_7\text{NO}$ . This compound is formed from heterocyclic quinolines by substituting an OH group on carbon number 8. The compound is light yellow in color and is widely used in the field of commerce and under various names. It is made mostly from quinoline-8-sulfuric acid and through the synthesis of a scrap of 2-aminophenol. 8-hydroxyquinoline is a di-protein chelating agent. Related links are Schiff bases derived from salicylic aldehydes - such as salicylic abdomen and salt. In a mild solution, the hydroxyl radical is bonded to a proton ( $\text{Pka}:0.89$ ) and nitrogen is not bound to the proton ( $\text{Pka}:5.13$ ). But there is a state of excitation in the form of zwitterion, where (+H) shifts from oxygen to give the oxygen anion to nitrogen and gives the nitrogen proton cation. Ni (ii) and Cu (ii).

### PRACTICAL PART

#### Equipment and materials used

By looking at various references, we have been able to apply it with chemical and analytical equipment, with very little purification used with that

equipment. USA, Merck, India and Sigma-Aldrich supplied high purity chemicals and solvents. Fourier transform infrared spectroscopy analysis was performed using (Shimadzu-Japan) and a KBr disk with in the range ( $400\text{--}4000\text{ cm}^{-1}$ ). A thermoelectric Mp apparatus (SMP-10 Stuart) was used and the melting point of the compounds was determined.

The prepared compounds were prepared using a glass test tube and using a (UV-Vis spectrophotometer (Shimadzu UV-1800)) at a concentration (3–10) in a solvent (dimethyl sulfoxide) using a quartz cell (1.0 cm) and a sampling interval (2 nm). A ((Bruker)) 300 MHz NMR was also used to take electron spectra of the produced compounds. spectrometer) and he changes in the chemical composition of (NMR- and -1H) spectra were recorded in (Dimethyl sulfoxide -  $\text{d}_6$  & Trivinylmethylsilane). Using the (Shimadzu-AA 680) device, the percentage of the in the complexes was found. STA (PT-1000 Linseis) was carried out at a temperature between ( $0\text{--}1000$ ) °C of argon gas. Thermogravimetric analysis was used to analyze the prepared compounds

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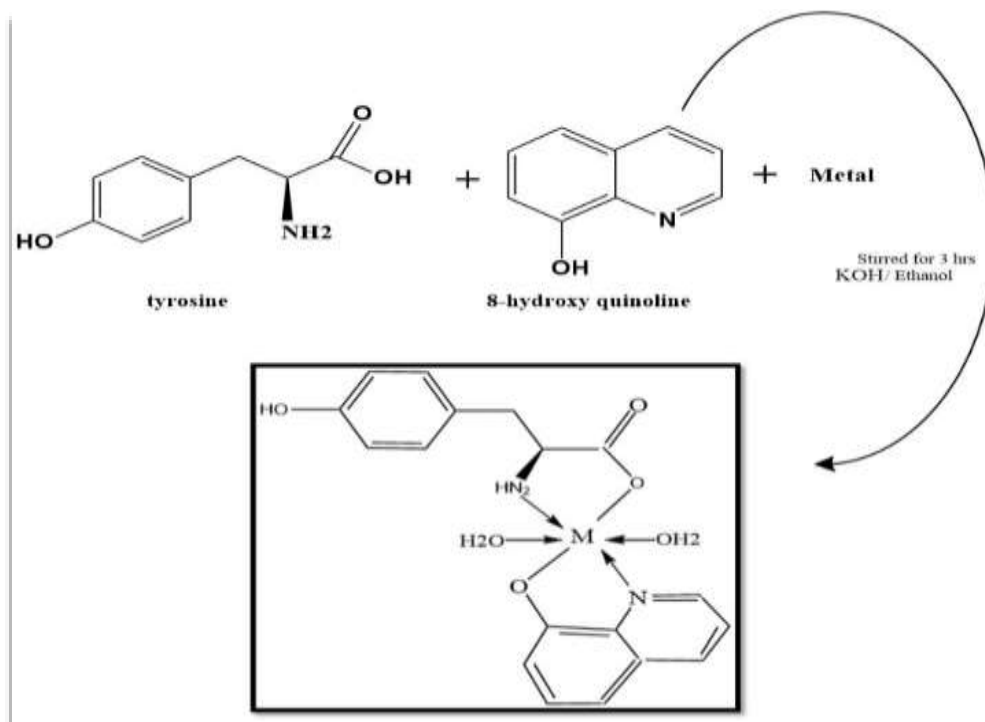
### The Organic Compound

#### Preparation of metal complexes with mixed ligands (Tyr & 8-H).

The structures were made in a molar ratio (1: 1: 1) (L: M: L) by dissolving grains of a base such as NaOH or KOH in distilled water, and metal chloride (0.034) was dissolved in 5 ml of ethanol, then tyrosine was added (0.1) in 5 ml of ethanol and add drops of the base solution prepared in No. (1) until the pH becomes ... Equivalent to 9. Then dissolve 8-hydroxyquinoline (0.08) in 5 ml of

ethanol and add drops of the base solution prepared in No. (1) until the pH becomes. Equivalent to 9.

Then a solution of the metal salt is added to that is, the answer to the first ligand salt tyrosine that rousing is done for about (10)minutes, then a solution is added to the second ligand salt and the filtered mixture is left for (3)hours for each complex, then a precipitate appears, then filtration and recrystallization with ethanol solvent, left to dry.



Scheme (1): Preparation course for the complexes

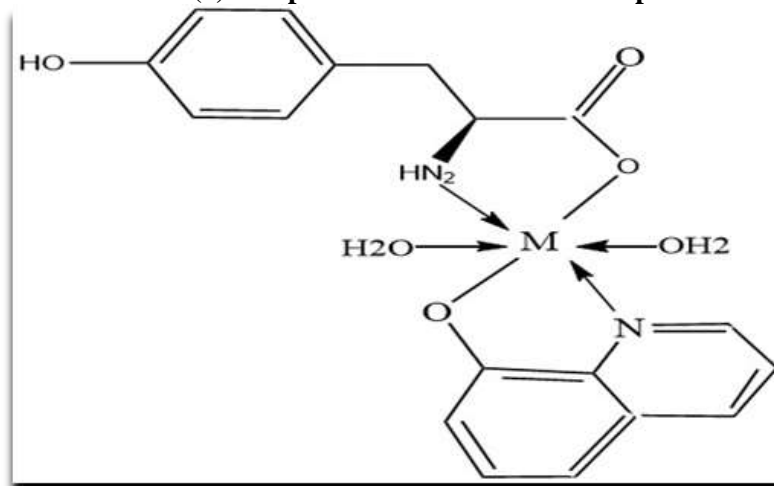


FIGURE 1: The proposed structure of the complexes

**TABLE 1.** Group of physical properties of the prepared complexes

Compound	Empirical Formula Formula _ wt	m.p C	Color	Conducts Ohm <sup>-1</sup> cm <sup>2</sup> mol <sup>-1</sup> in solvent (DMSO)
[Co(tyr &8-H)2(H2O)2]Cl	C <sub>32</sub> H <sub>32</sub> Cl <sub>6</sub> Mn N <sub>4</sub> O <sub>6</sub> 836.30	189-192	Green	73.3
[Mn(tyr &8-H)2(H2O)2]Cl	C <sub>32</sub> H <sub>32</sub> Cl <sub>6</sub> CoN <sub>4</sub> O <sub>6</sub> 840.32	199-202	Off white	76.5
[Cu(tyr &8-H)2(H2O)2]Cl	C <sub>32</sub> H <sub>32</sub> Cl <sub>6</sub> Ni N <sub>4</sub> O <sub>6</sub> 840.08	118-120	Brown	73.1
[Ni(tyr &8-H)2(H2O)2]Cl	C <sub>32</sub> H <sub>32</sub> Cl <sub>6</sub> CuN <sub>4</sub> O <sub>6</sub> 844.77	168-170	Green	71.4

### CONCLUSIONS AND DISCUSSION

Through the work in this research, the results showed the distinctive characteristics of the prepared metallic compounds in terms of thermal stability and the chemical and physical nature in terms of colors and properties in the selected solvents, namely (Dimethyl sulfoxide and Dimethylformamide). All of the ready pools were estimated based on the theoretical and practical findings of group (a) measurements. Like a tab (1)

#### FT-IR Spectra

##### Ligand (8-Hydroxyquinoline)

In the (3297 cm<sup>-1</sup>) region of the (8-H) spectrum of  $\nu$  NH While another absorption spectrum appears at the (1651 cm<sup>-1</sup>) region to be seen as meaning anything unique to (C = O amide). In addition to the (C-H aliphatic) group's (2924 cm<sup>-1</sup>) and (C-H aromatic) group's (3053 cm<sup>-1</sup>) and (C-Cl) region's (2838 cm<sup>-1</sup>) absorption areas, there is also an additional absorption region at (16) (Picture 2).

##### Complexes of ligand (8-Hydroxyquinoline&Tyrosine)

These spectra showed a remarkable inter-band variation As the NH (amide group) is stretched, a stretching vibration occurs in the (3429-3408 cm<sup>-1</sup>) migrated upwards by (130-105 cm<sup>-1</sup>) suggesting that the nitrogen atom of the amide group may be involved in the coordination of (8-Hydroxyquinoline-tyrosine) (17-18). Since there is no chance of a shift in frequency for this family (1655-1650 cm<sup>-1</sup>) in the ligand complex spectrum, we know that the (C = O amide) in the a ligand that isn't attached to primary ion (19,20).Also, new frequencies appeared  $\nu$  (M-O) for the aqueous group) and  $\nu$  (M-N)-(for the amide group) within the region (824-819 cm<sup>-1</sup>) and (489-496) cm<sup>-1</sup> respectively, which indicates compounding of metals. (22) In Tab(2) FT-IR data were reviewed. In Fig(2) the spectra of (8-Hydroxyquinoline - Tyrosine) and their complexes are shown in Fig (3).

**TABLE 2:** F T-IR data-- of (8-Hydroxyquinoline&Tyrosine) (cm<sup>-1</sup>) and their complexes.

Compo.	$\nu$ (C-H) From.	$\nu$ (C-H) Aliph.	$\nu$ (N-H)	$\nu$ (C=O) amide	$\nu$ (C-Cl)	$\nu$ (M-N)	$\delta$ (M-O) of water
Ligand (8-Hydroxyquinoline)	3053	2921	3299	1652	721	-	-
Co(tyr &8-H)2(H2O)2]Cl	3033	2925	3421	1650	717	823	487
[Mn(tyr &8-H)2(H2O)2]Cl	3051	2904	3402	1650	717	821	491
[Cu(tyr &8-H)2(H2O)2]Cl	3053	2920	3423	1649	717	821	491
[Ni(tyr &8-H)2(H2O)2]Cl	3035	2991	3413	1654	715	819	489

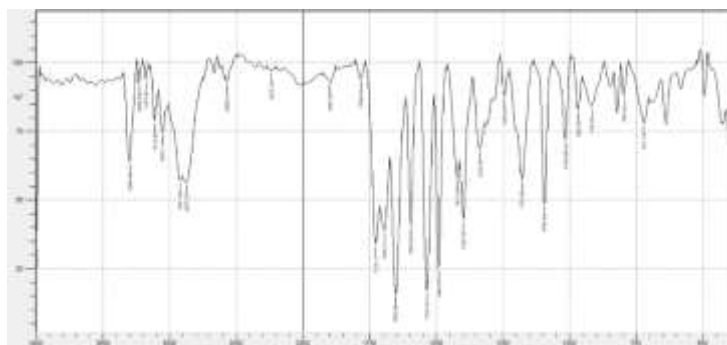


FIGURE 2. FTIR spectrum for the spectra of the ligand (8-Hydroxyquinoline)

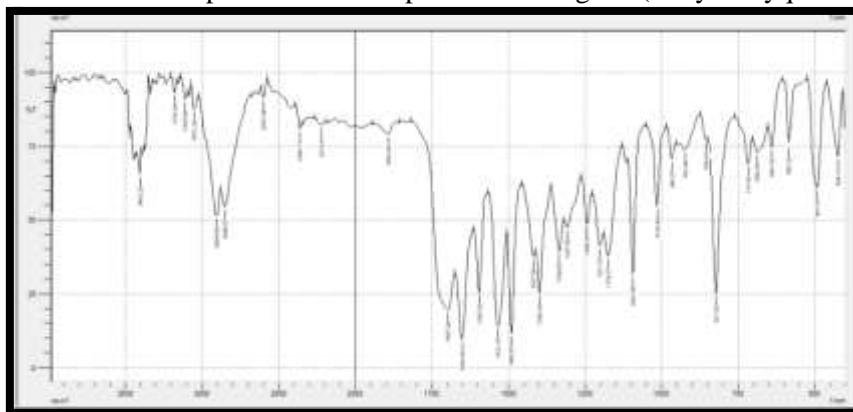


FIGURE .3 FTIR spectrum of the (8-Hydroxyquinoline&Tyrosine) and its complexes.

### Electronic spectra

#### Ligand (8-Hydroxyquinoline)

The electronic absorption spectrum of 8-Hydroxyquinoline in dimethyl sulfoxide solution is shown in Fig (4) with the highest absorption spectrum width (2nm) at (254 nm) It equals ( $39370 \text{ cm}^{-1}$ )  $\epsilon_{\text{max}} = (3720 \text{ molar}^{-1} \text{ cm}^{-1})$ , ((292 nm) It equals ( $34246 \text{ cm}^{-1}$ )  $\epsilon_{\text{max}} = (3853 \text{ molar}^{-1} \text{ cm}^{-1})$ ) which are set for ( $n \rightarrow \pi^*$ ), ( $\pi \rightarrow \pi^*$ ) in order in Tab (3).

#### 3.4.2. Special complexes of Ligand (8-hydroxyquinoline and tyrosine).

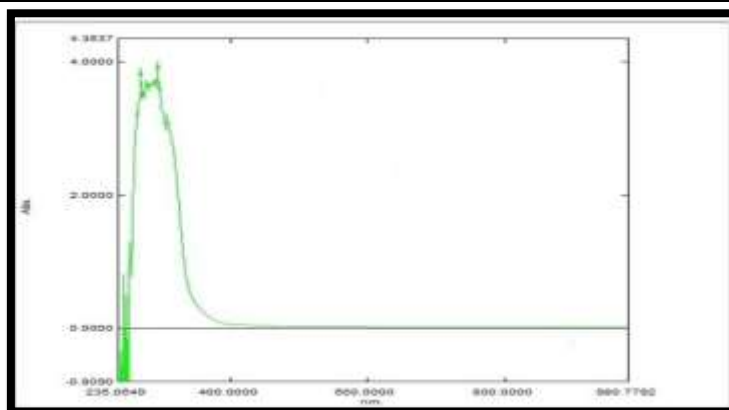
Complex width peaks may be seen in the (d-d) region of the electronic absorption spectra of (Mn-ii) j. at (888nm) that cause the  ${}^6A^1g \rightarrow {}^4T^2g$  (G) transition and (911 nm) assigned to  ${}^6A^1g \rightarrow {}^4T^1g$  (G). Other peaks are at ((275 nm) and (314 nm)) These peaks assigned to the inner bond indicate a manganese ion

As for the (Co-ii) complexes, tops out at ((272 nm) and (310 nm)) appear because the into

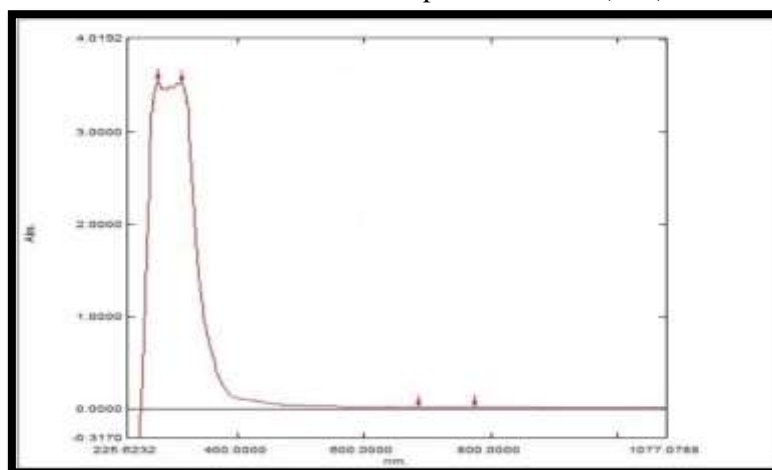
asymmetry. The emergence of other peaks in a region (d-d) (605nm) due to the transition of  ${}^4T^1g$  (F)  $\rightarrow$   ${}^4A^2g$  (P) and (682nm) due to  ${}^4T^1g$  (F)  $\rightarrow$   ${}^4T^2g$  (F). These electronic transitions that appeared in the cobalt complexes indicate geometry based on eight vertices, like in the complex named above.. electronic absorption radiation Nickel-iron complex spectrum (d-d) shows peaks at (686nm) designated for the ( ${}^3A^2g \rightarrow {}^3T^1g$ ) (P) transition and (774 nm) due to ( ${}^3A^2g \rightarrow {}^3T^2g$ ) (F). That appeared in specific areas refer to the octahedral geometric shape Fig (5). electronic absorption peaks (d-d) area peaking at (944 nm) in the spectra of the (Cu-ii) complex, which are ascribed to ( $2Eg$   $2T2g$ ). Other peaks at (264 nm) and (310 nm) due to internal symmetry, all of these peaks confirm the distorted octahedral geometry of the Cu ion. In Tab(3), the UV data for the compounds were shown

**TABLE 3:** The most important electronic transitions in absorption spectra of the prepares complex

Comp.	$\lambda_{\max}$ (nm)	$\nu$ ( $\text{Cm}^{-1}$ )	$\epsilon_{\max}$ ( $\text{moler}^{-1}.\text{cm}^{-1}$ )	Transition	Shape Geometry
Ligand 8-Hydroxyquinoline	254 292	39370 34246	3720 3853	$n \rightarrow \pi^*$ $\pi \rightarrow \pi^*$	-
(Mn)-8-Hydroxyquinoline&Tyrosine	215 314 887 910	36363 31847 11273 10959	3768 3653 15.8 16.2	Intra-ligand Intra-ligand ${}^6A_{1g} \rightarrow {}^4T_{2g(G)}$ ${}^6A_{1g} \rightarrow {}^4T_{1g(G)}$	Octahedral
Co-8-Hydroxyquinoline&Tyrosine	272 310 600 680	36764 32258 16667 14705	7048 6920 212.9 428.9	Intra-ligand Intra-ligand ${}^4T_{1g(f)} \rightarrow {}^4A_{2g}$ ${}^4T_{1g(f)} \rightarrow {}^4T_{2g(f)}$	Octahedral
Ni-8-Hydroxyquinoline&Tyrosine	272 312 686 774	36764 32051 14577 12919	7048 3597 17.2 16.8	Intra-ligand Intra-ligand ${}^3A_{2g} \rightarrow {}^3T_{1g(f)}$ ${}^3A_{2g} \rightarrow {}^3T_{2g(f)}$	Octahedral
Cu-8-Hydroxyquinoline&Tyrosine	264 310 944	37878 32258 10593	7222 6920 264.1	Intra-ligand Intra-ligand $2E_g \rightarrow 2T_{2g}$	Octahedral



**FIGURE 4:** Electronic spectrum of the (8-H).



**FIGURE 5:** Electronic spectrum of [Ni] complex.

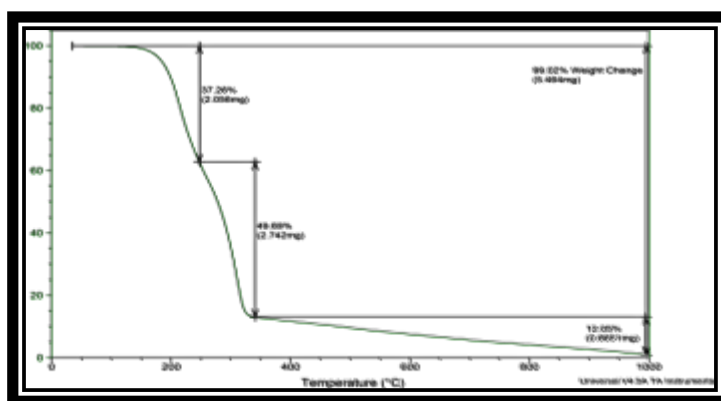
### Thermal analysis

[Co-8-Hydroxyquinoline & Tyrosine] were prepared and measured for A thermal analyzer made by a German firm (the STAPT-1000 by Linseis company) is used. This measurement was performed in a vacuum Specifically,

between zero and one thousand degrees Celsius, with a heating rate of 10 °C/min in a (atm) composed of (Ar)(g) (31-32min). Where the reading was done and recorded, all the results shown are extracted and derived from the TG curves in Table. Fig (4).

**TABLE 4.** Analysis results at different temperatures plus weight loss for each temperature

Com.	Stage	TAG			Fragmentation
		Thermo Gravimetry range (°C)	Found (calculate)		
			loss of Mass (mg)	Total mass lose 9(mg)	
Com. Co	1	190-250	2.056 (2.046)	5.464 (5.441)	C <sub>32</sub> H <sub>32</sub> C <sub>16</sub> MnN <sub>4</sub> O <sub>6</sub> 836.27 -11C + 1/2 Cl <sub>2</sub> -C <sub>3</sub> H <sub>4</sub>
	2	251-340	2.742(2.741)		
	3	341-1000	0.665(0.654)		



**FIGURE 6:** Thermal study of [Com. Co]

### CONCLUSION

This investigation revealed the Mn(ii), Co(ii), Ni(ii), and Cu(ii) compounds: synthesis and characterisation bound to the ligand (8-Hydroxyquinoline&Tyrosine). Based on the data (TGA and Fourier-transform infrared), for which octahedral geometry was suggested the prepared compounds. In its bidentate role, 8-Hydroxyquinoline&Tyrosine coordinates through the NH-C=O group's N atoms.

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