Synthesis and characterization of mixed ligand complexes of 8-hydroxy quinoline and Schiff base with some metal ions

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Abstract:

A new mixed ligand complexes have been prepared between 8- hydroxy quinoline and *o*-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on with Mn(II),Fe(II),Co(II),Ni(II) and Cu(II) ions . the prepared complexes were isolated and characterized by (FT-IR)and (UV-Vis) spectroscopy. Elemental analysis (C.H.N) Flame atomic absorption technique . in addition to magnetic susceptibility and conductivity measurement.

Introduction:

Schiff bases are an important class of ligands in coordination chemistry and find extensive application in different fields (1-3), Schiff bases derived from the salicylaldehydes are well known as polydentate ligands (4,5).coordinating in neutral forms . In recent years metal complexes of Schiff bases have attracted considerable attention due to their remarkable antifungal , antibacterial and anti tumor activities (6,7). Schiff bases complexes derived from 4-hydroxy salicylaldehyde and amines have strong anticancer activity (8). In particular Merck company has successfully developed an antibacterial drug Cilastatin using chiral copper(II) Schiff base complexes derived from salicylaldehydes and chiral amine (9). A search through literature (10-15) reveals that no work has been done on the transition metal complexes of the Schiff base *o*-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on and 8-hydroxy quinoline. In this paper we presented here synthesis and characterization of some new Mn(II),Fe(II),Co(II),Ni(II) and Cu(II) complexes with these two ligands as mixed ligand complexes.

These complexes have been studied by (FT-IR) and (U.V-Vis) spectroscopy. Flame atomic absorption techniques, in addition to magnetic susceptibility and conductivity measurements.

Experimental:

Material and instrumentation:

Metal salts($MnCl_2.4H_2O$,$FeCl_2XH_2O$, $CoCl_2.6H_2O$,$NiCl_2.6H_2O$,$CuCl_2.2H_2O$) were obtained from Riedel – Dehaenage in high purity , salicylaldehydes , P-Amino-2,3-dimethyl-1-phenyl-3-pyrazolin-5-on , 8- hydroxy quinoline,ethanol , methanol and dimethyl foramaide from B.D.H.

Physical measurements and analysis:

Melting point were recorded on Gallen Kamp melting point apparatus and were uncorrected.FT-IR spectra were recorded as CsI discs using FT-IR.3800 Shimadzu in the range of(4000-200cm^{-1}). Electronic spectra were obtained using UV-160 shimadzu

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Spectrophotometer at room temperature(10^{-3}M)in DMF.Conductivity was measured by capacitor analyzer and in DMF solution ($1x10^{-3}$M) at room temperature .elemental analysis (C.H.N) were performed by using a Carlo Erba 1106 elemental analyzer. Magnetic susceptibility measurements were obtained at 25C by Balance magnetic susceptibility model MSB-$MK_{1.}$ The metal percent in the complexes were determined by using the Hitachi Atomic Absorption .

Preparation of the Schiff base (L):

The Schiff base ligand was prepared by the condensation of the P-amino-2,3-dimethyl-1-phenyl-3-pyrozoline-5-on (0.5g,2.47mmol) with the (0.26ml,2.47mmol) salicylaldehyde in methanol (15ml) .The resulting mixture was then refluxed for (1h) .The yellow precipitate formed was filtered and recrystalized from absolute ethanol to give yellow needles (14).

General method for preparation of the Complexes:

To the aqeous solution of the metal salts (0.5) gm an ethanolic KOH solution of ligand (L) (0.64-1.06)gm (2.08-3.44)m.mole was added followed by the solution of ligand (Q) (0.30-0.50)gm (2.06-3.44)m.mole .dissolved in ethanolic KOH.the reaction mixture was stirred continuously. the required product was shortly precipitated at room temperature. The precipitates were filtered off .washed with (1.1) (ethanol: water) crystallized from ethanol and dried at (60C). The table (1) shows the physical properties of the prepared Complexes.

Results and Discussion:

The physical properties and data of the Ligands (L) and (Q)with there metal Complexes are given in table (1).
The low values of molar conductivity (Ω) in DMF indicate the non .electrolyte behavior of these Complexes (16-19)

Table(1): Physical Characteristics and analytical data for (L),(Q) and its metal complexes

Compound	Color	Melting Point C	Yield	C,H,N Analyses, found(calcd)(%)				Molar conductivity ohm^{-1}cm^{2}mol^{-1}
				M	C	H	N	
L	Yellow	189 256(d*)	85%	- -	69.8 (70.34)	6 (5.5)	13.1 (13.67)	4.47
Q	White	73		-				12.258
[Mn(L)(Q)]	Green yellow	260(d*)	65%	0.1 (0.108)	64.16 (63.5)	4.38 (4)	5.14 (4.7)	15.2
[Fe(L)(Q)]	Dark green	245(d*)	71%	0.125 (0.11)	64.04 (64.8)	4.37 (5.1)	5.13 (5.5)	16.888
[Co(L)(Q)]	Green yellow	270(d*)	60%	0.13 (0.11)	63.65 (64)	4.34 (4.6)	5.1 (5.8)	19
[Ni(L)(Q)]	Yellow green	265(d*)	80%	0.1 (0.115)	63.68 (62.7)	4.35 (3.9)	5.11 (4.6)	15.2
[Cu(L)(Q)]	Dark green	225(d*)	62%	0.14 (0.123)	63.088 (62.8)	4.3 (4)	5.06 (4.9)	12.063

(d*) = Decomposition Q=8- hydroxy quinoline ,
L= *o*-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on

Magnetic properties:

The magnetic moment (μ_{eff}) for the complexes of Mn^{+2},(d^5) , Fe^{+2}(d^6) , and Co^{+2} (d^7) were found to be (4.656)B.M ,(4.7) B.M and (3.522) B.M respectively .within the expected spin- only values (20). but the higher value of (μ_{eff}) of the Ni^{+2}(d^8)complexes (3.9)B.M may be due to orbital contribution (21,22) .finally the (μ_{eff}) of the Cu^{+2}(d^9) complexes (1.884)B.M within the expected value for one electron .

Table (2): The magnetic properties of the complexes at (25C)

Complexes	No. of electron	No. of unpaired electron	Electron configuration	Term symbol	Term symbol Ground state in Td	Orbital contribution	μ_{eff}		Suggested structure
							Found	calc	
[Mn(L)(Q)]	d^5	5	$e^2t_2^3$	^6S	$^6A_1$	NO	4.656	4.916	Tetrahedral
[Fe(L)(Q)]	d^6	4	$e^3t_2^3$	^5D	^5E	NO	4.7	4.898	Tetrahedral
[Co(L)(Q)]	d^7	3	$e^4t_2^3$	^4F	$^4A_2$	NO	3.522	3.872	Tetrahedral
[Ni(L)(Q)]	d^8	2	$e^4t_2^4$	^3F	$^3T_1$	Yes	3.9	2.828	Tetrahedral
Cu(L)(Q)]	d^9	1	$e^4t_2^5$	^2D	$^2T_2$	Yes	1.884	1.732	Tetrahedral

The Electronic Spectra

(a)Electronic Spectra of free ligands:

The spectrum of free ligand (L) showed a strong band at λmax (256nm) attributed to ($\pi \longrightarrow \pi^*$) and another at λ_{max} (340,362nm) due to ($n \longrightarrow \pi^*$) $^{(4).}$

The 8- hydroxyl quinoline spectrum show absorption bands at λ_{max} (294,307nm) respectively due to ($\pi \longrightarrow \pi^*$) and another bands of λ_{max} (372,391nm) belongs to ($n \longrightarrow \pi^*$) $^{(10)}$.

Electronic Spectra of the complexes :

[Mn(L)(Q)] Complexes:

The Green–yellow complex showed strong bands at λ_{max} (296,345nm),these are attributed to charge transfer (C.T) and the shoulder at λ_{max} (351nm) due to $^6A_1 \longrightarrow {}^4E_{(D)}$ finally the band at λ_{max} (366nm) belong to $^6A_1 \longrightarrow {}^4T_{2(D)}$,these values are accepted for tetrahedral complexes $^{(23-25)}$.

[Fe(L)(Q)] Complexes:

The dark-green complex of iron (II) showed band at λ_{max} (351nm) belong to charge transfer and another bands at λ_{max} (442,455nm) caused by the electronic transition $^5E_{(D)} \longrightarrow {}^5T_{2(D)}$ $^{(23-27).}$

[Co(L)(Q)] Complexes:

The Green-yellow complex of Cobalt (II) showed band at λ_{max} (277nm) attributed due to charge transfer (C.T) .and the other bands at λ_{max} (351,365nm) attributed to $^4A_{2(F)} \xrightarrow{v3} {}^4T_{1(P)}$ .transitions and the electronic transition of $^4A_{2(F)} \xrightarrow{v2} {}^4T_{1(F)}$ was found at λ_{max} (630nm) , finally the absorption band at λ_{max} (715nm) is due to $^4A_{2(F)} \xrightarrow{v1} {}^4T_{2(F)}$ transitions$^{.(23-27)}$.

Accordingly the ligand field splitting energy, (10Dq) was taken as equal to the electronic transition (ν1) (13986.014 Cm^{-1}) and the reach inter electronic repulsion parameter ($B^\backslash$) was found to be (123.907 Cm^{-1}) from the relation

$$(\beta = \frac{B^\backslash}{B^0}) \ \beta$$ was found to be equal to(0.127)

These parameters are accepted for Cobalt (II) Tetrahedral complexes $^{(23-27).}$

[Ni(L)(Q)] Complexes:

The electronic spectrum of yellow-green complex of Ni(II) revealed the folloing electronic transition $^3T_{1(F)} \longrightarrow {}^3A_{2(F)}$ with (C.T) , $^3T_{1(F)} \longrightarrow {}^3T_{1(P)}$ and $^3T_{1(F)} \longrightarrow {}^3T_{2(F)}$ transitions at λ_{max} (381nm) , λ_{max} (514nm) and λ_{max} (820nm) respectively . The (10Dq) value for the complex was equal (15243.902Cm^{-1})and the ($B^\backslash$) value found to be (607.733Cm^{-1}) (β) is equal to (0.590). are characteristic for Tetrahedral complexes of Ni(II) $^{(23-27)}$.

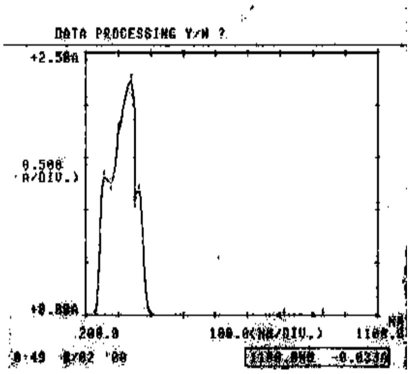
[Cu(L)(Q)] Complexes:

The spectrum of the dark-green complex of Cu(II) showed very strong band at λ_{max}(270nm) which belongs to the charge transfer, the shoulder found in the visible region at λ_{max} (430nm) was attribute to the electronic transition $^2T_{2(D)} \longrightarrow {}^2E_{(D)}$ $^{(23-28).}$

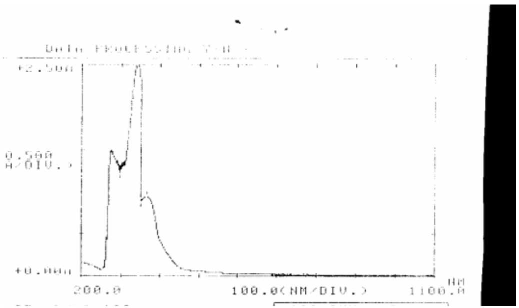
All transitions with their a assignments are summarized in Table (3).

Table (3): U.V-Visible Spectra of free Ligand and their Complexes (10^{-3}) M in DMF

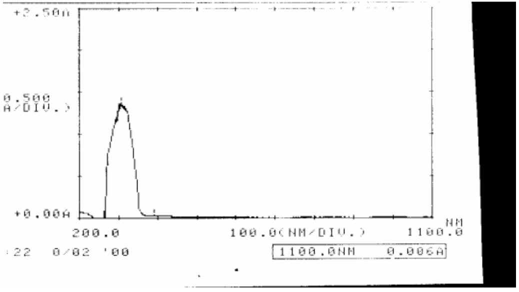
Compounds	λ_{max} nm	ABS	Wave number Cm^{-1}	ε_{max} ($L.mol^1.Cm^1$)	Transitions	Remarks
L	256	2.632	39062.5	2632	$\pi \rightarrow \pi^*$	-
	340	4.458	29411.764	4458	$n \rightarrow \pi^*$	-
	362	2.354	27624.306	2358	$n \rightarrow \pi^*$	-
Q	294	2.500	34013.605	2500	$\pi \rightarrow \pi^*$	
	307	1.370	32573.289	1370	$\pi \rightarrow \pi^*$	
	372	0.027	26881.72	27	$n \rightarrow \pi^*$	
	391	0.037	25575.447	37	$n \rightarrow \pi^*$	
[Mn(L)(Q)]	296	1.235	33783.783	1235	(C.T)	-
	345	2.500	28985.507	2500	(C.T)	-
	351	0.882	28490.028	882	${}^6A_1 \rightarrow {}^4E_{(D)}$	-
	366	0.946	27322.404	946	${}^6A_1 \rightarrow {}^4T_{2(D)}$	-
[Fe(L)(Q)]	351	0.779	28490.028	779	(C.T)	New band
	361	0.814	27700.831	814	$n \rightarrow \pi^*$	Hypo chromic effect
	416	0.148	24038.461	148	-	Red shift
	442	1.796	22624.434	1796	${}^5E_{(D)} \rightarrow {}^5T_{2(D)}$	New band
	455	0.178	21978.022	178		
[Co(L)(Q)]	244	0.143	40983.606	143	-	Blue shift with Hypo chromic effect
	277	1.859	36101.083	1859	(C.T)	New band
	351	0.896	28490.028	896	${}^4A_{2(F)} \xrightarrow{v3} {}^4T_{1(P)}$	Corresponding with (C.T)
	365	0.966	27397.26	966		
	630	0.016	15873.015	16	${}^4A_{2(F)} \xrightarrow{v2} {}^4T_{1(F)}$	New band
	715	0.022	13986.014	22	${}^4A_{2(F)} \xrightarrow{v1} {}^4T_{2(F)}$	New band
[Ni(L)(Q)]	304	0.665	32894.736	665	-	Blue shift
	358	0.763	27932.96	763	(C.T)	New band
	381	1.200	26246.719	1200	${}^3T_{1(F)} \rightarrow {}^3A_{2(F)}$	Corresponding with(C.T)
	514	0.063	19455.252	63	${}^3T_{1(F)} \rightarrow {}^3T_{1(P)}$	New band
	820	0.044	12195.122	44	${}^3T_{1(F)} \rightarrow T_{2(F)}$	New band
[Cu(L)(Q)]	270	1.143	37037.037	1143	(C.T)	New band
	312	0.912	32051.282	912	-	Red shift
	430	0.024	23255.813	24	${}^2T_{2(D)} \rightarrow {}^2E_{(D)}$	New band



Spectra (3)U.V-Visible spectrum of free Ligand (L)



Spectra (2) U.V-Vis of complex [Mn(Q)(L)]



Spectra (3) U.V-Vis of 8-hydreoxy quinoline

Infrared spectral studies :

Infrared spectra of free ligands:

The characteristic vibrations and assignments of ligand (L) and (Q) and their complexes as C_sI disc are described in table (4).

The spectrum of ligand (L) exhibited weak bands at 3465Cm^{-1}and (3060) Cm^{-1}attributed to the stretching vibration of ν(O-H)and ν(C-H) aromatic respectively (9,14,29) ,but the strong band at (1656)Cm^{-1}belong to str.vibr ν(C==O) and the another strong bands belong to the str.vibr ν(C==N) , ν(C==C) and ν(C—O) was found at (1600) Cm^{-1},(1490)Cm^{-1}and (1265) Cm^{-1}respectively (29-32)

In the spectrum of ligand (Q) was noticed broad band at (3213) Cm^{-1}attributed to str. Vib ν (OH) with shoulder at (3074) Cm^{-1} due to str . vibr ν(C-H) aromatic and the another strong bands appeared at (1577) Cm^{-1},(1500) Cm^{-1} and (1284) Cm^{-1} attributed str. Vibr ν (C=N). ν (C=C) and ν (C-O) (10-13,33)

Infrared spectra of Complexes:

The infrared spectra of the prepared Complexes exhibited str .vibr. ν(C=N) in the range (1591-1597)Cm^{-1}show shifting to the lower frequencies by (9-7)Cm^{-1}in Comparison with ligand (L) .but with ligand (Q) the shafting was between (14-16) to the higher frequencies which indicated the coordination of ligands with metal ions through the nitrogen atoms in their structure . and the another bands appeared . in the range (1122-1130) Cm^{-1}belong to the str . vibr ν (C-O) was shifted to the lower frequencies by (143-135) Cm^{-1}and (162-154) Cm^{-1}in Comparison with ligands (L) and(Q) respectively the disappearing of band for ν(OH) vibration in the spectar of Complexes indicating Coordination of phinolic oxygen with metal ions(31,33,34).

The spectra bands of complexes at (1656) Cm^{-1} was characteristic for the carbonyl group which did not Sutter a shift, suggesting that the oxygen atom of the carbonyl group is not Coordinated to the metal ion (31, 33).

M-ligand bonds:

The infrared of prepared complexes showed weak bands in the range (501-509) Cm^{-1} and (445-452) Cm^{-1} was attributed to the str.vibr of ν(M-O) and ν(M-N) respectively .

Table (4) the characteristic bands of Infrared spectra of the ligand s and their complexes

Compounds	ν(O-H)	ν(C-H)ar	ν(C=O)	ν(C=N)	ν(C=C)	ν(C-O)	ν(M-O)	ν(M-N)
Q	3213 b	3074 sho	-	-	1500 v.s	1215 s	-	-
L	3465 w	3060 w	1654 v.s	1600 s	1490 s	1265 s	-	
[Mn(L)(Q)]	-	3062 sho	1651 s	1593 sho	1481 s	1130 m	445 w	501 w
[Fe(L)(Q)]	-	3074 v.w	1655 m	1593 sho	1481 m	1130 m	452 w	502 w
[Co(L)(Q)]	-	3050 v.w	1653 vs	1591 sho	1481 m	1126 w	451 w	505 w
[Ni(L)(Q)]	-	3070 v.w	1654 w	1593 w	1481 w	1122 w	448 vw	509 w
Cu(L)(Q)]	-	3065 w	1655 w	1592 w	1481 w	1130 vw	450 w	501 w

S=strong, m=medium, w=weak, sho=shoulder, v=very, b=broad

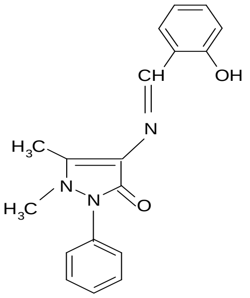
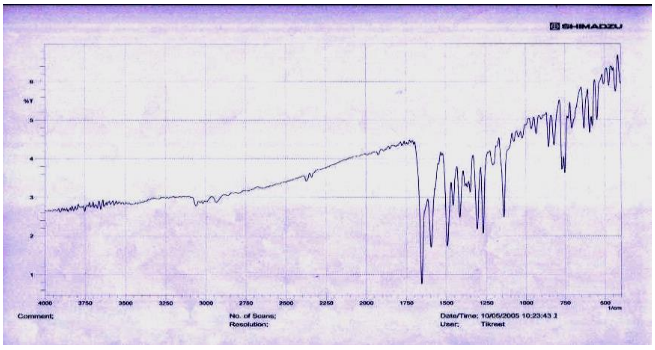


Fig. (1) Structure of the Ligand (L)



Spectra (4) Infrared (FT-IR) of Schiff base (L)

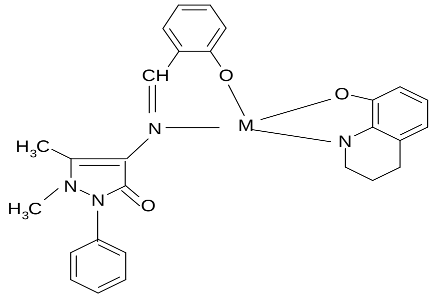
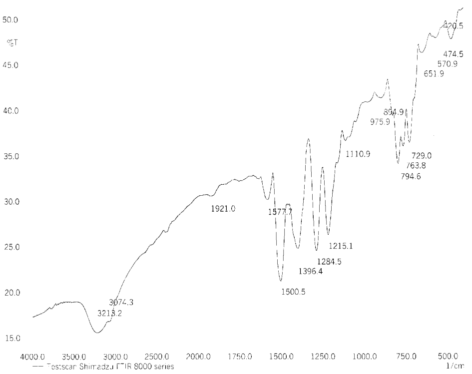
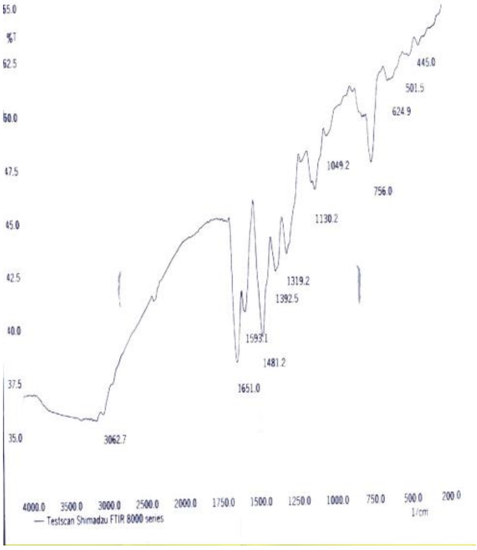


Fig. (2) Suggested Structure of the tetrahedral complexes (M=Mn(II),Fe(II),Co(II),Ni(II)andCu(II))



Spectra (5) Infrared (FT-IR) of 8-hydreoxy quinoline



spectra(6) intrared (FT-IR)of complexes[MN(Q)(L)]

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تحضير وتشخيص معقدات الليكاندات المختلطة من 8 -هايدروكسي كوينولين وقاعدة شف مع بعض الايونات الفلزية

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الخلاصة :

حضرت معقدات جديدة ذات ليكاندات مختلطة من 8-هايدروكسي كوينولين و 0-هيدروكسي بنزلدين -6-فنيل-3,2-ثنائي مثيل -4-أمين-3-بايرازولين-5-اون .مع ايونات عناصرالمنغنيز (II) ،الحديد(II) ،الكوبلت(II)، النيكل(II) والنحاس (II) .

عزلت المركبات المحضرة وتم تشخيصها باستخدام طيف الأشعة تحت الحمراء وطيف الأشعة فوق البنفسجية-المرئية كذلك استخدمت في التشخيص تحليل العناصر(C.H.N) و تقنية الامتصاص الذري أللهبي للعناصر إضافة إلى قياسات الحساسية المغناطيسية والتوصيلية الكهربائية .