Synthesis and characterization of VO(II), Cr(III),Mn(II), Fe(II),Zn(II),Cd(II),Hg(II) and UO₂(VI) complexes with ligands containing (ONO) and (ON) donor set atoms

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Abstract:

A new Schiff base o-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on (HL) ,have been prepared and characterization.(HL) has been used as a chelating ligand to prepare a number of metal complexes VO(II) ,Cr(III) ,Mn(II),Fe(II),Hg(II) and UO₂(II) .and mixed ligands complexes have been prepared between o-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on and 8- hydroxy quinoline with VO(II),Zn(II),Cd(II), Hg(II) and UO₂(II) the prepared complexes were isolated and characterized by (FT-IR)and (UV-Vis) spectroscopy. Elemental analysis (C.H.N) Chloride contents, Flame atomic absorption technique. in addition to magnetic susceptibility and conductivity measurement. Molar ratio measurement in solution gave comparable results with those obtained from solid state study.

Introduction

Metal complexes of Schiff bases have played a central role in the development of coordination chemistry (1) Schiff base ligands from salicylaldehyde and chiral amines have been widely applied in enantioselective cyclopropanation of styrene's (2-5) a symmetric a ziridination of olefins ⁽⁶⁾, enantioselective epoxidation ⁽⁷⁻ enantioselective ring opening epoxides Schiff base salicylaldehyde and its complexes have a variety of applications including antibacterial (12) anti-fungal ,toxicological studies (13) anti tumor activities (14) and strong anti cancer activity (15).transition metal complexes of Co(II),Ni(II),Cu(II) and Zn(II) with Schiff base ligand derived from the condensation of salicylaldehyde with P-Amino-2,3-dimethyl-1-phenyl-3pyrazolin-5-on synthesized chlorinated species of 8hydroxy quinoline has been proved as anti-bacterial and fungal agents (17) .derivatives of Cu

with 8- hydroxy quinoline are anti-fouling agents (18) .Mixed ligand transition metal complexes of Mn(II),Fe(II),Co(II),Ni(II) and Cu(II) ions with Schiff base ligand the derived from condensation salicylaldehyde with P-Amino-2,3dimethyl-1-phenyl-3-pyrazolin-5-on 8- hydroxy quinoline (19). In this paper we presented here synthesis and characterization of some new complexes with two ligands Schiff bases (HL) and 8hydroxy quinoline (Q) of the general formula: $[M(L)_2]$ $(M=VO^{+2}.Cr^{+3})$ Mn^{+2} , Fe^{+2} , Hg^{+2} and UO_2^{+2}) and [M(L)(Q)] $(M = VO^{+2}, Zn^{+2}, Cd^{+2}, Hg^{+2})$ and UO_2^{+2} . These complexes have been studied by and (U.V-Vis) spectroscopy. Elemental (C, H and N) analysis, Chloride Flame atomic absorption contents techniques, in addition magnetic to susceptibility and conductivity measurements.

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Experimental:

Material and instrumentation

Metal salts (VOSO₄. H₂O,CrCl₃ 6H₂O MnCl₂.4H₂O ,FeCl₂ ,CdCl₂.2¹/₂H₂O ,Zn(CH₃COO)₂ 2H₂O ,HgCl₂ UO₂(CH₃COO)₂.2H₂O) were obtained from Riedel – Dehaenage in high purity ,salicylaldehyde , 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

Elemental (C, H and N) analysis were carried out on a Perkin-Elmer automatic equipment model 240.B of the department chemistry, faculty of science, Azerbaijan University of Tabriz, Iran. The Chloride contents of complexes were determined by potentiometric titration method using (686-Titro processor-665. Dosimat Metrohn Swiss). Melting point was 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 using UV-160 obtained shimadzu spectrophotometer at room temperature $(10^{-3}M)$ in **DMF.Conductivity** measured by capacitor analyzer and in **DMF** solution $(1x10^{-3}M)$ at room Magnetic susceptibility temperature. measurements were obtained at 25C⁰ by Balance magnetic susceptibility model MSB-MK_{1.} The metals percent in the complexes were determined by using the Hitachi Atomic Absorption.

Preparation of the Schiff base (HL):

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 ⁽¹⁶⁾.

General method for preparation of the Complexes [1-6]:

To the methanol and KOH, solution of (2m mole) of the ligand (HL) (0.612g) an 15ml methanol solution of (1mmole) of salt [VOSO₄. H_2O the metal (0.181)g,CrCl₃ 6H₂O(0.266)g, MnCl₂.4H₂O (0.197)g, FeCl₂ (0.126)g, (0.271)g. HgCl₂ and UO₂(CH₃COO)₂.2H₂O (0.424)g] was added drop wise with continuous stirring. The mixture was stirred further for (1-1.5)h at 50C⁰. The precipitated solid was then filtered off.

General method for preparation of the Complexes [7-11]:

To the methanol solution of (1m mole) of the meta salts [VOSO₄. H₂O (0.181)g, Zn(CH₃COO)₂2H₂O (0.219)g, $CdCl_2 2^{1}/_2H_2O$ (0.228)g, $HgCl_2$ (0.271) and UO₂(CH₃CO)₂.2H₂O (0.424)g] an methanol and KOH solution of ligand (HL) (0.306g, 1m.mole)was added followed by the solution of ligand (Q) (0.145g,1m.mole) ,dissolved in methanol KOH .The reaction mixture was stirred further for (1-2)h.. The required product precipitated was shortly temperature. The precipitates were filtered off .washed with (1.1) (ethanol: water) crystallized from ethanol and dried at $(60C^{0})$. The table (1) shows the physical properties of the prepared Complexes.

Results and Discussion:

The Schiff base ligand (HL) ,on interaction with metal ion salts yields complexes corresponding to the general formula $[M(L)_2]$ (with M=VO,Mn,Fe,Hg and UO₂) and $[Cr(L)_2]$ Cl .The mix ligand (HL) and 8- hydroxy quinoline on reaction with to the general formula [MLQ] (with M= VO(II),Zn(II),Cd(II), Hg(II) and UO₂(II)).The ligand (HL) claiting with metal ion VO(II),Zn(II),Cd(II), Hg(II) and

U(VI)) to the bidentat but with metal ion Cr(III),Mn(II) and Fe(II),to the tridentate. The complexes were prepared by the general reaction show in below:

M	VO(II)	Mn(II)	Fe(II)	Hg(II)	UO ₂ (II)
X	SO_4	CL	CL	CL	CH ₃ COO
n	1	2	2	2	2
m	1	4	-	-	2

And
$$2HL+CrCL_3.6H_2O$$
 [$Cr(L)_2$] CL

 $HL+Q+MXn.mH_2O \longrightarrow [MLQ]$ With

M	VO(II)	Zn(II)	Cd(II)	Hg(II)	UO ₂ (II)
X	SO_4	CL	CL	CL	CH ₃ COO
n	1	2	2	2	2
m	1	2	$2^{1}/_{2}$	-	2

I.R Spectra:

The (IR) spectrum for Schiff base (HL) ligand (Fig 2a) ,displayed band at $(1600)\text{cm}^{-1}$ due to the $\nu(C==N)$ stretching frequency for the azomethine group $^{(16,20-23)}$.the strong band at $(1654)\text{cm}^{-1}$ belong to $\nu(C==O)$.The weak band at $(3465)\text{cm}^{-1}$ is attributed to the $\nu(O-H)$

In the (IR) spectrum of 8-hydroxy quinoline (Q) ligand (Fig 2b) displayed bands at (3213)cm⁻¹ and (1500)cm⁻¹ attributed to the ν (O-H) and ν (C-N) (26-28). Table 1 presents the most important IR spectral bands of the ligand (HL) and (Q) with all the metal complexes

The IR spectra bands of $[VO(L)_2]$ $[UO_2(L)_2]$ [VO(L)(O)] $[Hg(L)_2]$ [Zn(L)(Q)][Cd(L)(Q)][Hg(L)(Q)],and $[UO_2(L)(Q)]$ complexes at range (1590-1587)cm⁻¹ is characteristic for the v(C=N),show shifting to the lower frequencies by (13-10)cm⁻¹ in comparison with ligand Schiff base (HL).but with ligand 8hydroxy quinoline (Q) the shafting was between (21-42)cm⁻¹ to the higher frequencies which indicated the coordination of ligands with metal ions through the nitrogen atoms in their

quinoline azomethine and (16,19,21,22,25,28-30) and the another bands appeared. in the range (1110-1250) Cm⁻ belong to the v(C-O) was shifted to the lower frequencies by (15-155) Cm⁻¹and (-2-138) Cm⁻¹in Comparison with ligands (HL) and (O) respectively and did not appeared bond belong to the v(O-H) in Comparison with ligands (HL) disappearing of band for v(OH) vibration in the specter of Complexes indicating Coordination of phinolic oxygen with metal ions^(16,19,25,30-33)

The IR spectra bands of complexes at (1654)cm⁻¹ for the carbonyl group did not shift, suggesting that this oxygen atom of the carbonyl group is not coordination to the metal ion (21,22,25,29,30)

The IR spectrum bands of $[VO(L)_2]$ an [VO(L)(Q)] complexes at (956) Cm⁻¹and (964) Cm⁻¹respectivly,due to (V=O) and the complexes $[UO_2(L)_2]$ and $[UO_2(L)(Q)]$ show a strong absorption band in the 895 Cm⁻¹and 900 Cm⁻¹respectivly,due to $(O=U=O)^{(31,32,34,35)}$.

It is suggested they are bidentate complexes from that the oxygen atom of this phenolic group and the nitrogen atom of the azomethine group are coordination to the metal ion (Fig 1 b)

The IR spectra bands $[Cr(L)_2]CL,[Mn(L)_2]$ and $[Fe(L)_2]$ complexes at ((1589)cm⁻¹,(1588)cm⁻¹and (1590)cm⁻¹ attributed to the v(C=N), show shifting to the lower frequencies in comparison with Schiff base ligand (HL) to the higher frequencies which indicated the coordination of ligand with metal ions through the nitrogen atoms in their azomethine structure (16,19,21,22,29) and the another bands appeared. in the (1198)Cm⁻ ¹,(1200) Cm⁻¹ and(1203) Cm⁻¹attributed to the v(C-O) was shifted to the lower frequencies in Comparison with ligand (HL) respectively and did not appeared bond belong to the v(O-H) in Comparison with ligands (HL) disappearing of band for v(OH) vibration in the specter Complexes indicating Coordination of phinolic oxygen with metal ions $^{(16,19,25)}$ and the another bands appeared. in the $(1589)\text{Cm}^{-1}$, $(158)\text{Cm}^{-1}$ and (1590) Cm belong to the v(C=O) was shifted to the lower frequencies in Comparison with ligands (HL) respectively the specter of Complexes indicating Coordination of oxygen atom in their carbonyl structure with metal ions $^{(16,25,31-33)}$

It is suggested they are tridentate complexes from that the oxygen atom of

this phenolic group and carbonyl group the nitrogen atom of the azomethine group are coordination to the metal ion (Fig1 c)

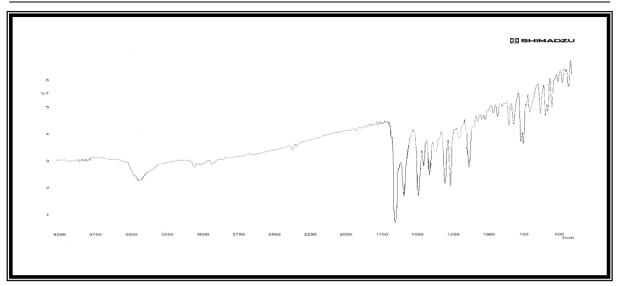
The IR spectrum of prepared complexes showed weak bands in the range (385-456) Cm^{-1} and (475-541) Cm^{-1} was attributed to the v(M-O) and v(M-N) respectively v(M-O) and v(M-N) respectively

(Fig 1)The suggests Structure (a) Ligand(HL) Schiff base ,(b) [M(L)(Q)]complexes with (M=Zn(II),Cd(II) and Hg(II)),(c) $[M(L)_2]$ complexes (M=Mn(II) and Fe(II))

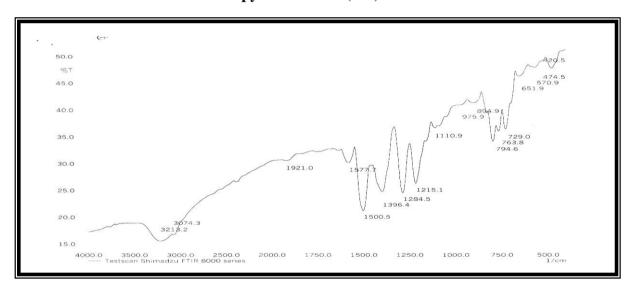
Table (1) the characteristic bands of Infrared spectra of the ligand and their complexes

Comp	v(O-H)	ν(C-H)ar	ν(C-H)al	ν(C=O)	ν(C=N)	ν(C=C)	v(C-N)	v(C-O)	ν(M-O)	ν(M-N)
	3213	3074		.(0 0)	,(6 1,)	1500	1500	1248	,(1.1.0)	,(11)
Q			-	-					-	-
	b	sho	-			v.s	s	s		
	3465	3055	2950	1660	1605	1490		1265		
L	w	w	w	v.s	s	s		s	-	
		3062	2931	1652	1593	1488		1141	425	475
$[VO(L)_2]$	-	sho		s	sho	s		m	w	w
		3050	2939	1642	1589	1496		1198	420	510
[Cr(L) ₂]Cl	-	w	w	v.s	sho	s		m	w	w
		3055	2939	1645	1588	1492		1200	408	503
$[Mn(L)_2]$	-	w	w	v.s	sho	s		m	w	w
		3055	2939	1648	1590	1496		1203	400	480
$[Fe(L)_2]$	-	v.w	w	vs	sho	m		w	w	w
		3043	2943	1653	1590	1495		1250	412	512
$[Hg(L)_2]$	-	v.w	w	v.s	sho	m		w	w	w
		3062	2939	1654	1592	1495		1203	385	532
$[UO_2(L)_2]$	-	v.w	w	v.s	sho	m		w	w	w
	-	3055	2940	1652	1594	1496	1465	1110	402	501
[VO(L)(Q)]		v.w	w	v.s	sho	m	sho	w	w	w
[7, (L)(O)]	_	3061	2919	1655	1590	1487	1479	1215	412	523
[Zn(L)(Q)]		v.w	w	v.s	sho	m	sho	w	w	w
[Cd(L)(Q)]	-	3043	2945	1652	1587	1485	1476	1200	430	541
		v.w	w	v.s	sho	m	sho	w	w	w
III (I VO)?	-	3076	2934	1651	1594	1499	1477	1231	387	516
[Hg(L)(Q)]		v.w	w	v.s	sho	m	sho	w	w	w
		3062	2939	1650	1596	1535	1458	1203	385	532
$[UO_2(L)(Q)]$	-	w	w	w	sho	w	sho	w	vw	w

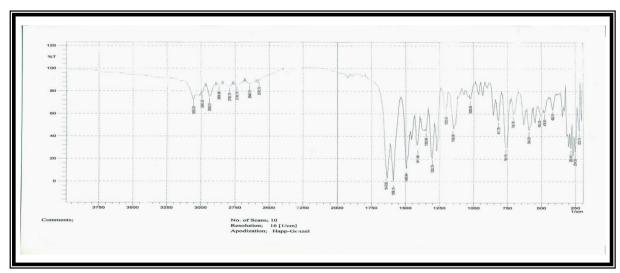
S=strong, m=medium, w=weak, sho=shoulder, v=very, b=broad



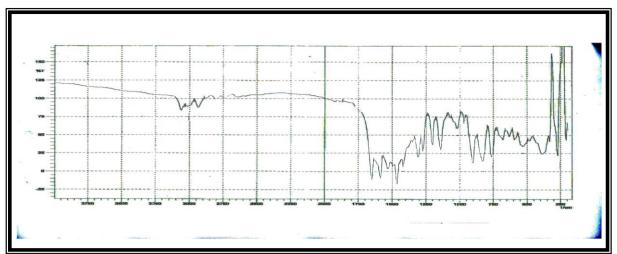
(Fig 2a) IR spectrum o-hydroxybenzylidene-1-phenyl-2,3-dimethyl-4-amino-3-pyrazolin-5-on (HL)



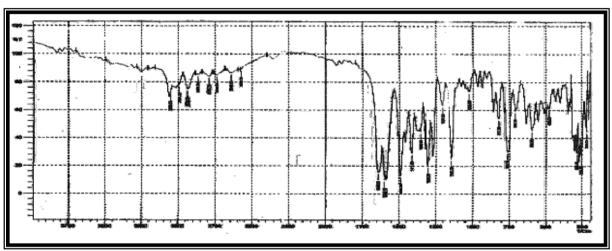
(Fig 2b) IR spectrum 8-hydroxy quinoline (Q)



 $(Fig\ 2c)\ \ IR\ spectrum\ [Cr(L)_2]Cl\ complex$



(Fig 2d) IR spectrum [VO(L)₂] complex



(Fig 2e) IR spectrum [UO₂(L)₂] complex

Electronic Spectra

The (UV-Vis) spectrum for the free ligand schiff base (HL) show a strong bands at (256)nm was attributed to $(\pi \longrightarrow \pi^*)$ and another at (340)nm and (362)nm due to $(n \longrightarrow \pi^*)^{(29)}$. The UV-Vis) spectrum 8- hydroxyl quinoline show absorption bands at (294)nm and (307)nm respectively due to $(\pi \longrightarrow \pi^*)$ and another bands of (372)nm belongs to $(n \longrightarrow \pi^*)^{(27)}$

The electronic spectra data of the complexes are summarized in (Table 2). The (UV-Vis) spectra of the complexes displayed absorptions at range (272-290)nm and (344-350)nm attributed due to the ligand field (16,19,27,36,37) and anther bands are observed at range (350-376)nm

assigned to the charge transfer (C.T) (16,36-38)

In[VO(L)₂] and [VO(L)(Q)] complexes the peaks (642)nm and (573)nm are attributed to (d-d) electronic transition type (2B_2 2A_1) and (2B_2 2E_g) respectively .suggesting an square pyramid of geometry $^{(31,32,36-40)}$

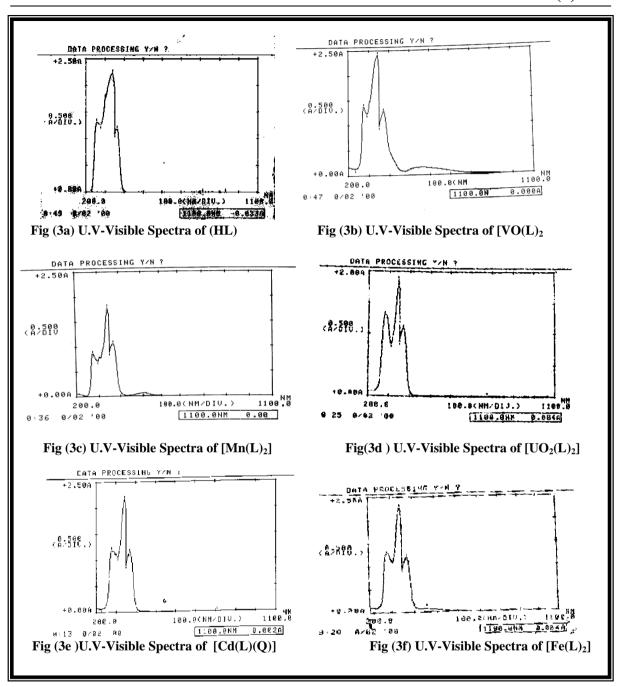
The (UV-Vis) spectra of the $[Cr(L)_2]Cl$ complexes showed peaks at (425)nm and (510)nm attributed to (d-d) transition types $^4A_{2g(F)}$ $\xrightarrow{v_3}$ $^4T_{1(P)}$, $^4A_{2g(F)}$ $\xrightarrow{v_2}$ $^4T_{1(F)}$ and $^4A_{2g(F)}$ $\xrightarrow{v_1}$ $^4T_{2(F)}$ respectively .suggesting an octahedral of geometry $^{(36\text{-}38,41)}$.and (UV-Vis) spectra of the $[Mn(L)_2]$ complex showed one broad band is observed at (505)nm due to $^6A_{1g}$ $\xrightarrow{}$ $^4T_{1(g)(G)}$ and $^6A_{1g}$ $\xrightarrow{}$ $^4T_{1(g)(G)}$ electronic transition, suggesting an Octahedral

Table (2): U.V-Visible Spectra of free Ligand and their Complexes (10^{-3}) M in DMF and Magnetic susceptibility measurements

Diam= Dia magnetic

Diam= Dia magnetic									
Transitions & Remarks		Suggested structure							
•									
1	1.71	Square							
C.T New band ${}^{2}B_{2} \longrightarrow {}^{2}A_{1}$		Pyramidal							
Eg									
		Octahedral							
i	3.126								
T _{1P}	3.120								
T_{1F} T_{2F}									
- 2F	1								
1	1.05	Octahedral							
ı(G)									
)(G) (D)									
,	5.231	Octahedral							
C.T New band ${}^5T_{2g(D)}$ $\underline{\hspace{1cm}}^5E_{g(D)}$		Octanedrai							
g(D)									
	Diam	Tetrahedral							
i									
	D:	0-4-111							
i	Diam	Octahedral							
1									
,	1.71	Square							
1	1.71	Pyramidal							
E _g									
	Diam	Tetrahedral							
i	2	Tourunourun							
	Diam	Tetrahedral							
i	1								
	Diam	Tetrahedral							
i	<u> </u>								
	Diam	Octahedral							
i	1								

of geometry $^{(32,33.36,37)}$.In [Fe(L)₂]complex the peak (460)nm is attributed to (d-d) electronic transition type $^5T_{2g(D)}$ $^5E_{g(D)}$ respectively, suggesting an Octahedral of geometry $^{(33,36-38)}$



Molar conductance value of the Cr(III)complex is (87.412) ohm⁻¹cm²mol⁻¹. the Cr(III)complex is electrolytic (1:1)⁽⁴¹⁾ or shows a certain degree of conductivity the another complexes did not show electrolytic properties (9.6-27.564) ohm⁻¹cm²mol⁻¹. (42,43)

Magnetic susceptibility measurements:

The magnetic moment value of VO(II) complexes is (1.71BM) which suggests a square pyramidal geometry (31,32) around the metals ion. The magnetic moment of

Cr(III),Mn(II)and Fe(II) complexes (3.126)BM,(1.05)BM and Diamagnetic and these CFSE=(-12Dq ,-20Dq and -24Dq) which suggests the high spin sixcoordinated Octahedral arrangement (32,33,41) of ligand molecules around the metals ion .the Zn(II),Cd(II) and Hg(II) complexes is found to be diamagnetic which suggests the Tetrahedral geometry $^{(19,26,44)}$ around the metals ion. The UO₂(II) complexes is found to be diamagnetic which suggests the Octahedral

geometry^(34,35). The physical properties and data of the Ligands (HL) and (Q)with there metal Complexes are given in (Table 3) Complex formation in solution:-

Complexes of (HL) with metal ions were studied in solution using methanol as a solvent, in order to determined the ratio of [M:(HL)], (M=VO, Cr, Mn, Fe, Hg,

and UO_2) in the complex following (Molar ratio methods). A series of solutions were prepared having a constant concentration (10^{-3}) of the metal ion and ($0.25\text{-}4\times10^{-3}$) (HL). The analytical data show that the metal to ligand ratio is (1:2) they are soluble in common organic solvents

Table (3): Physical Characteristics and analytical data for (HL), (Q) and its metal complexes

CompNo.	Suggested formula For isolated	Color	Melting Point C	Yield %	C,H,N,Cl and Metal Analyses, Found (calcd) (%)						Molar conductivity
l I	precipitate				C	Н	N	Cl	Metal	DMF	ohm ⁻¹ cm ² mol ⁻¹
HL	-	Light Yellow	189	85	69.81 (70.34)	6 (5.52	13.1 (13.67)	-	-	-	4.47
Q	-	White	73	-				-	-	-	12.258
1	[VO(L) ₂]	Yellow green	212d	60	62.01 (63.746)	4.121 (4.721)	11.435 (12.395)	Nil	7.091 (7.525)	1:2	23.121
2	[Cr(L) ₂]Cl	Brown	210d	83	62.11 (61.919)	4.21 (4.586)	10.68 (12.039)	4.76 (5.08)	6.526 (7.381)	1:2	87.412
3	$[Mn(L)_2]$	Brown	194d	72	63.98 (64.993)	5.87 (4.814)	12.897 (12.637)	Nil	7.421 (8.124)	1:2	14.05
4	[Fe(L) ₂]	Dark brown	194d	78	65.11 (64.896)	6.001 (4.807)	11.83 (12.618)	Nil	7.23 (8.262)	1:2	9.6
5	$[Hg(L)_2]$	Light orange	223d	78	52.657 (53.288)	4.56 (3.947)	10.087 (10.36)	Nil	23.12 (24.67)	1:2	25.121
6	$[\mathrm{UO}_2(\mathrm{L})_2]$	Orange	208d	67	47.879 (49.053)	4 (3.633)	9.98 (9.538)	Nil	-	1:2	17.987
7	[VO(L)(Q)]	Violet	216d	68	63.01 (62.682)	5.087 (4.252)	10.076 (10.824)	Nil	9.028 (9.854)		27.564
8	[Zn(L)(Q)]	Yellow green	263d	74	63.654 (62.718)	5 (4.269)	11.021 (10.866)	Nil	11.87 (12.631)		23.21
9	[Cd(L)(Q)]	Yellow	230d	88	56.654 (57.562)	4.87 (4.086)	10.32 (9.949)	Nil	20.939 (19.971)		21.112
10	[Hg(L)(Q)]	Dark Green	212d	90	48.03 (49.82)	4.142 (3.382)	9.32 (8.61)	Nil	32.068 (30.753)		21.98
11	[UO ₂ (L)(Q)]	Dark orange	250d	74	45.546 (44.978)	3.12 (3.054)	7.121 (7.774)	Nil	-		22.487

⁽d*) = Decomposition Q=8- hydroxy quinoline,

HL= O-hydroxybenzylidene-1-phenyl-2, 3-dimethyl-4-amino-3-pyrazolin-5-on

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تحضير وتشخيص معقدات الفندايل (II) ،الكروم(III)، المنغنيز(II)، الحديد(II)، الزئبق(II) واليورانيل (II) مع ليكاندات حاوية على (ONO) و(ON) كمجاميع واهبة

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الخلاصة

تحضير قاعدة شف الجديدة اور ثو- هايدروكسي بنزلدايين-1-فنيل-2,2- ثنائي مثل -4-امينو-3- بايروزولين -5- أون (HL) وتشخيصها تم استخدام (HL) كاليكاند لتحضير عدد من المعقدات الجديدة مع ايونات عناصر الفندايل (II)،الكروم (III)، المنغنيز (II)، الحديد (II)، الزئبق (II) واليورانيل (II) . كما استخدم 8-هايدروكسي كوينولين (II)، اليكاند (HL) التحضير معقدات حاوية على مزيج من الليكاندات مع ايونات عناصر الفندايل (II)،الخارصين (II)، الكادميوم (II)، الزئبق (II)، اليورانيل (II) . تم عزل المعقدات تشخيصها باستخدام طيف الأشعة تحت الحمراء وطيف الأشعة فوق البنفسجية-المرئية كذلك استخدمت في التشخيص تحليل العناصر (C.H.N) و محتوى الكلورو تقنية الامتصاص الذري أللهبي للعناصر إضافة إلى قياسات الحساسية المغناطيسية والتوصيلية الكهربائية . تم قياس النسبة المولية للفلز مع (HL) في المحلول وقد اعطت نتائج مطابقة مع التي تم الحصول عليها في الحالة الصلبة.