Synthesis and Characterization of some Transition Metal Complexes with Hydrazone ligands

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Abstract

The hydrazone ligands used in this work were: Salicyl hydrazone (SH), Salicyl phenylhydrazone (SPH), salicylaldehyde -2,4-dinitrophenylhydrazone (SDNPH), 4-hydroxy salicylaldehyde-4-nitrophenylhydrazone (4-HSNPH) .

The prepared complexes were characterized by physico-chemical and spectroscopic method. It is concluded that some of the prepared hydrazone complexes are non-electrolytes (neutrals). The ligand hydrazone coordinated to the metal ion as bidentate chelate to form six coordinated neutral complexes of the type $[ML_2Cl_2]$ $\{M=Mn^{2+},Co^{2+},Ni^{2+}andCu^{+2}\}$ possessing octahedral geometry and others form four coordinated ionic complexes of the type $[ML_2]Cl_2$ where $\{M=Mn^{2+},Co^{2+},Ni^{2+},Cu^{2+}\}$possessing a square planer or tetrahedral geometry.

Conductivity measurements indicate that the prepared complexes in dimethyl sulfoxide correspond to (1:2) electrolytes, in which ligand hydrazone coordinated to the metal as bidentate chelate to form four coordinated ionic complexes of the type $[ML_2]$ Cl_2 $\{M=Mn^{2+},Co^{2+},Ni^{2+},Cu^{2+}\}$ possessing a tetrahedral or square planer which were confirmed by $AgNO_3$ test for complexes containing chloride ions .

Keywords: Hydrazones ligands, Transitions metal complexes, Hydrazones complexes .

Introduction

Hydrazones RR'C=N–NR"R"'[R'=H, NH_2, OH,] [R"=H,CH_3,---etc] are compounds which derived from condensation of hydrazines with carbonyl compounds, namely aldehyde and ketones [1] .

Hydrazones are azomethine characterized by the triatomic grouping C=N–N and they are interesting substances because of their physiological activity like treatment of several diseases such as tuberculosis, anticancer ,antioxidant activity ,antitumor, antiviral agent, antimicrobial [2-7] ,antifungal and antibacterial activity as iron chelaters in treatment of anemia's and antiviral drugs[8],this activity is attributed to the formation of stable chelate with transition metals which catalyses the physiological process[9].They also act as herbicides, insecticides, nematocides, rodenticides, plant growth regulators, sterilants for house flies [2] .

Great interest in the complexes of transition metals with hydrazones developed as a consequence of their increasing application in medicine, analytical chemistry, and synthesis of novel heterogeneous catalysts of oxidation–reduction processes, as well as in numerous fields of science and technology. Among ligand systems, hydrazones are special because, a number of compounds express biological activity and complex formation with transition metals [10].

The interest of hydrazone complexes increased by development of bioinorganic chemistry field, since it has been recognized that many of these complexes may serve as models for biologically important species [11].

Experimental

General

IR spectra were recorded on F.T.Thermo Mattson 300 spectrophotometer in the (400-4000 cm^{-1}) range using KBr discs.Elemantal analysis for some complexes were carried out on (Al–ALBayt) university in Jordon. Electronic spectra were obtained using Philips Pu 8800 and Jenway 6405 spectrophotometers. Conductivity measurements were carried out on a conductivity meter using WTW– conductometer inolab 749-Germany. Melting points (or decomposition points) of the ligands and complexes were measured using electro thermal – IA 9000 – England /melting point apparatus .

Starting materials

All chemical used in this work were either analar or reagent grade and used without further purification.

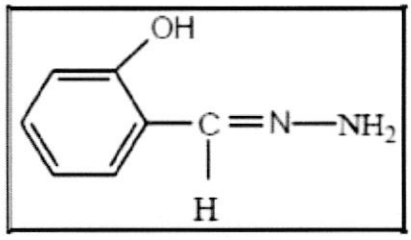
Preparation of hydrazone ligands

The hydrazones (SH) and (SPH) were prepared according to the literature method [12].by mixing (0.1mol) of an ice-cold solution of hydrazine hydrate, phenyl hydrazine in absolute EtOH with (0.1mol) of salicylaldehyde. Ice–cold water was added to the reaction with continuous stirring. The product was filtered, washed with ice –cold water and dried in vacuum over anhydrous $CaCl_2$ and recrystallized from EtOH. The hydrazones (SDNPH) and (4–HSNPH) were prepared according to the literature [13], by mixing equimolar amount of salicylaldehyde with 2, 4–dinitro phenyl hydrazine and 4–hydroxyl salicylaldehyde with 4–nitro phenyl hydrazine respectively in absolute ethanol. After heated to reflex for 1 hour a colored precipitate was obtained which was filtered off, washed with methanol and dried under vacuum .

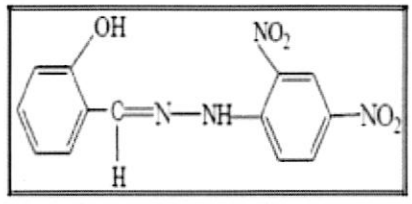
Preparation of complexes

Complexes of the type $[M(L)_2Cl_2]$ and $[M(L)_2]Cl_2$
M = Mn(II), Co(II), Ni(II) and Cu(II)

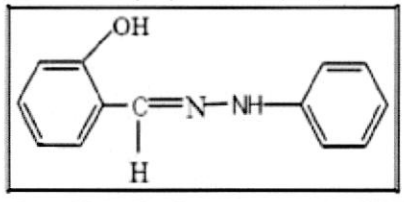
To an ethanolic solution (0.01mol,30ml) of an appropriate metal chloride salt , a hot ethanolic solution of hydrazone ligand (0.02 mol,20 ml) was added, the mixture was stirred for about 30 minutes , colored precipitate was formed , separated by filtration, washed several times with diethyl ether and dried under vacuum .



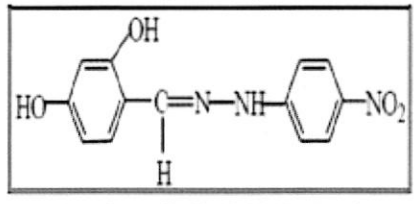
Salicylhydrazone (SH)



Salicylaldehyde–2, 4–dinitrophenylhydrazone (SDNPH)



Salicylaldehyde phenylhydrazone (SPH)



4-hydroxySalicylaldehyde–4–nitrophenylhydrazone(4-HSNPH)

The prepared hydrazone ligands

Table :(1) Elemental analysis and physical properties of some complexes

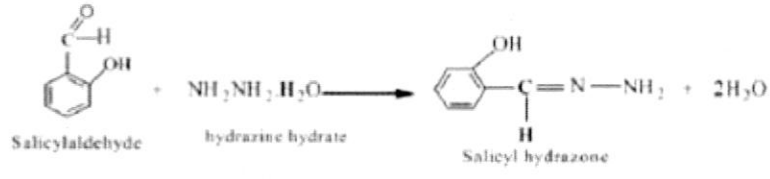
Complex	Color	Melting point (°C)	Found (calculate)%		
			C	**H**	**N**
$[Ni(SH)_2]Cl_2$	Green	>289*	41.70 (41.86)	4.10 (4.02)	12.88 (13.95)
$[Cu(SPH)_2]Cl_2$	Dark green	>194*	56.01 (55.91)	4.64 (4.34)	9.97 (10.03)
$[Ni(SDNPH)_2]Cl_2$	Reddish-orange	252	43.12 (42.56)	2.88 (2.75)	15.11 (15.27)
$[Mn(4\text{-}HSNPH)_2Cl_2]$	Red	>288*	46.54 (46.47)	3.27 (3.30)	12.46 (12.51)

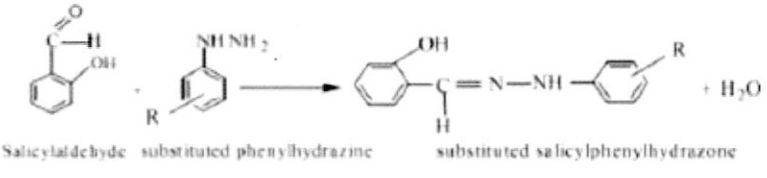
*Decomposition

Results and Discussion

Infrared spectra

The IR spectra analysis for hydrazone ligands gave information about the mode of coordination of ligands to the metallic ions. The characteristic bands are presented in Table (2). The hydrazone ligands salicylhydrazone (SH) and salicylphenylhydrazone (SPH), which derived from salicylaldehyde with hydrazine hydrate or phenyl hydrazine, respectively, were prepared according to the followings :





R=H,4-NO$_2$,2,4-NO$_2$

The donor atoms (active sites) may be:

(a) Phenolic oxygen and azomethine nitrogen.

(b) Phenolic oxygen and middle nitrogen of hydrazone ligands .

Type (a) is interested as example of coordination via non deprotonated phenolic group and azomethine nitrogen forming stable complexes of six- membered ring.

The IR spectra of all free ligands exhibit a medium broad band at the (3272-3303) cm^{-1} due to the stretching vibration of the (–NH–) group. This band is not greatly affected upon complexation [14]. The bands in the region of (939–1178) cm^{-1} are assigned to the υ (N–N) vibrational modes and they exhibit upward shift in the complexes , indicating its participation in coordination[15], and this shifting

suggests the involvement of at least one of nitrogen atoms in the bonding with the metal ions[16,17] .

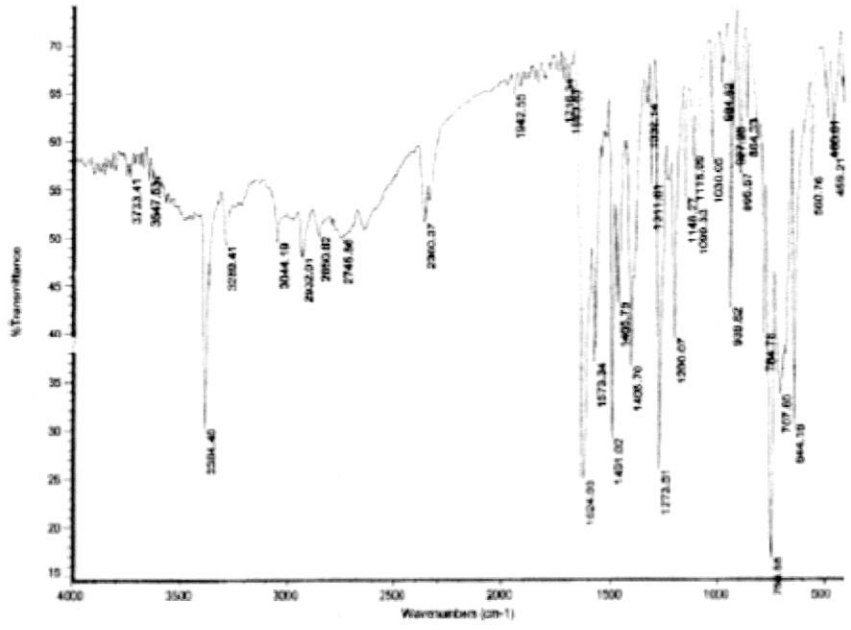
The rather strong (C=N) stretching vibration, found at ~ (1603–1631)cm^{-1} in free ligands, shifts to lower or higher wave number showing that the ligands coordinates to the metal via the imine donor atom. The shift to lower frequency has been explained as a weakening of the (C=N) bond resulting from the loss of electron density from the nitrogen to the metal atom or shift to higher frequency; indicating an increase in the force constant of C=N as a consequence of the coordination through azomethine nitrogen or sometimes as a result of coupling between C=N and C=C.The double bond character between carbon and nitrogen is increased [18, 19, 20].

The IR spectra of the hydrazones also showed a weak and broad absorption band in the region (3371–3484)cm^{-1}assigned to the stretching vibration of intermolecular hydrogen bonded υ(O–H).Upon complexation this band either shift to higher or lower frequency region by (3-71) cm^{-1} this may be due to the hydrogen bonding or its coordination to the metal which is confirmed by the appearance of new band around (405– 476) cm^{-1} due to (M–O) stretching vibration , indicating that the coordination occurs through oxygen atom of hydroxyl group[21,22]. Further more support for this coordination was obtained by shifting the phenolic υ(C–O) stretching towards higher frequencies by~ 40 cm^{-1} in the (SPH) complexes. While in (SH, SDNPH and 4-HSNPH) shifted to lower frequency. Therefore the complexation in this type of complexes occurs through nitrogen of azomethine and oxygen of hydroxyl group forming stable six membered ring complexes .

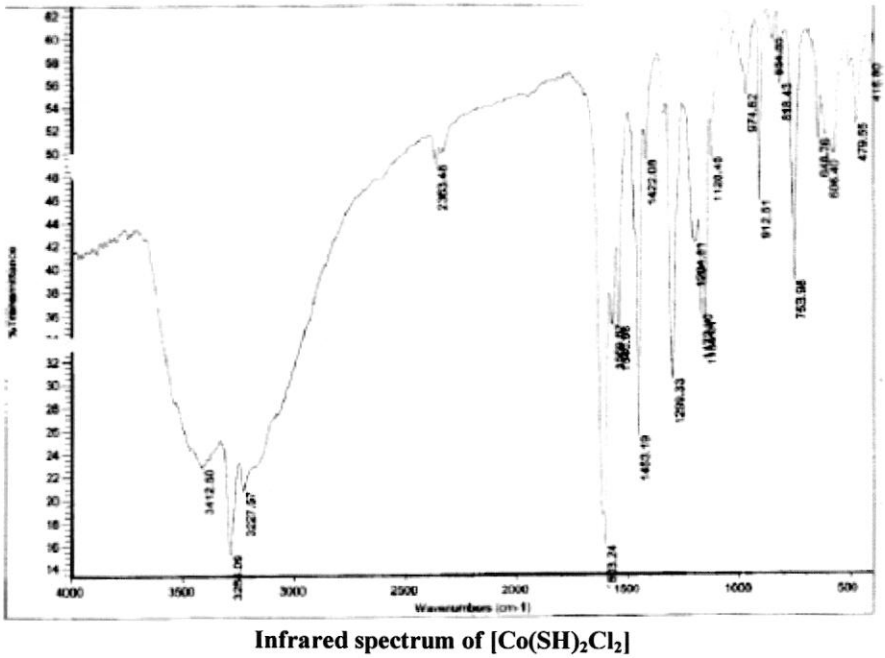
In all complexes, the new bands were observed at about (614–501) cm^{-1} and (476–405) cm^{-1}, tentatively assigned to υ(M–N) and υ(M–O) stretching vibrations, respectively as reported in literatures [23].

Table (2): Selected IR spectra bands of hydrazone ligands and their complexes

No.	Ligands and complexes	υ (N-H)	υ (C=N)	υ (O-H)	υ (C-O)	υ (N-N)	υ (M-N)	υ (M-O)
	SH	3289	1624	3384	1273	939	------	------
01	$[Mn(SH)_2]Cl_2$	3284	1573	3411	1197	983	588	421
02	$[Co(SH)_2Cl_2]$	3284	1603	3412	1204	1128	606	416
03	$[Ni(SH)_2]Cl_2$	3283	1621	3442	1272	1178	609	413
04	$[Cu(SH)_2]Cl_2$	3280	1573	3445	1206	1115	565	412
	SPH	3290	1603	3484	1278	1148	-------	-----
05	$[Mn(SPH)_2Cl_2]$	3292	1622	3430	1301	1159	501	467
06	$[Co(SPH)_2]Cl_2$	3290	1622	3478	1301	1159	504	476
07	$[Ni(SPH)_2]Cl_2$	3291	1622	3413	1301	1158	501	465
08	$[Cu(SPH)_2]Cl_2$	3290	1606	3468	1271	1150	507	471
	SDNPH	3272	1621	3371	1332	1077	-----	-----
09	$[Mn(SDNPH)_2]Cl_2$	3269	1613	3413	1273	1144	614	405
10	$[Co(SDNPH)_2]Cl_2$	3269	1613	3418	1273	1144	604	406
11	$[Ni(SDNPH)_2]Cl_2$	3269	1613	3414	1272	1144	603	405
12	$[Cu(SDNPH)_2]Cl_2$	3269	1613	3414	1273	1141	605	407
	4–HSNPH	3303	1631	3403	1292	1109	-----	-----
13	$[Mn(4\text{-}HSNPH)_2Cl_2]$	3304	1599	3409	1272	1111	577	415
14	$[Co(4\text{-}HSNPH)_2Cl_2]$	3302	1601	3408	1270	1113	537	470
15	$[Ni(4\text{-}HSNPH)_2Cl_2]$	3305	1603	3406	1270	1114	537	471
16	$[Cu(4\text{-}HSNPH)_2]Cl_2$	3346	1590	3446	1235	1121	578	412



Infrared spectrum of (SH)



Infrared spectrum of $[Co(SH)_2Cl_2]$

Electronic Spectra for hydrazone complexes

The electronic spectra of all the prepared hydrazone complexes were recorded in either absolute methanol or dimethyl sulfoxide at room temperature in ultraviolet and visible region, and they showed characteristic absorption bands around (33444–49019) cm^{-1} which attributed to aromatic transitions $\pi \rightarrow \pi^*$, or $n \rightarrow \pi^*$ and charge – transfer [24] .

In present work the hydrazones complex of Co(II), Table(3) complex No.(09) exhibit the electronic transition bands at 23364 cm^{-1} which may be attributed to ^4T_1g (F) $\rightarrow$ ^4T_1g (P) of Co(II) in an octahedral environment[25,26] while complex No. (03) showed one band at (14727) cm^{-1} which may be attributed to $^4A_2 \rightarrow {}^4T_1$ (P) of cobalt(II) in tetrahedral environment[24,27].

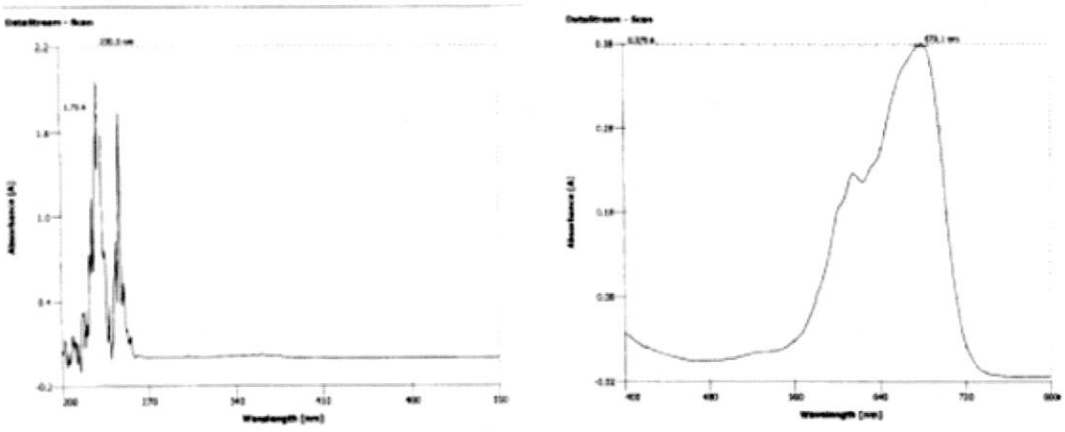
In the present work complexes No. (02, 04, 07) were consist of one or two transitions around (17271–17857) cm^{-1}, and (15151) cm^{-1} which assigned to

$^2B_1g \rightarrow {}^2Eg$ and $^2B_1g \rightarrow {}^2A_1g$ respectively for a square planer around Cu (II) .

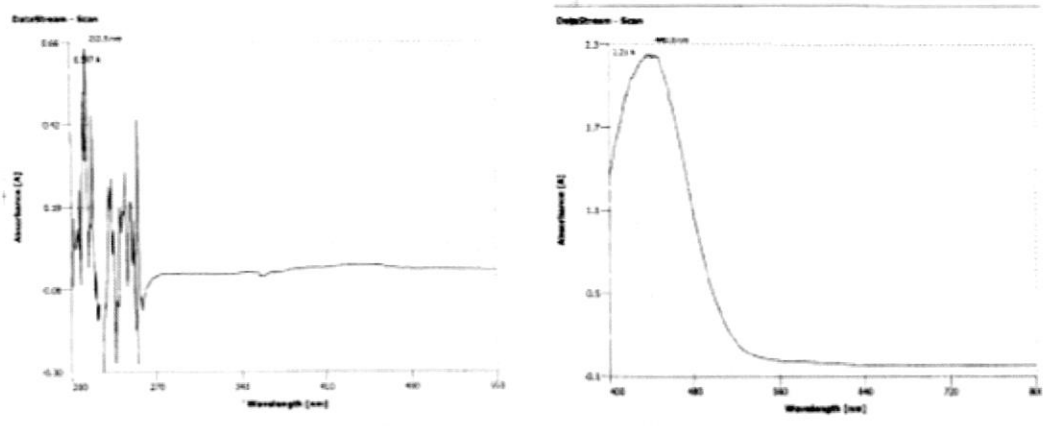
The electronic spectra of Mn(II), complex No.(08) showed a band at (28409) and (26666) cm^{-1} which assigned to $^6A_1g \rightarrow {}^4Eg$ (D) and $^6A_1g \rightarrow {}^4T_2g$ (D) transitions respectively for an octahedral geometry as compared with the reported values for octahedral Mn (II) complex [28] .

Complex No.(05) exhibit an absorption band at (17543)cm^{-1} assigned to $^6A_1g \rightarrow {}^4T_1g$(D) transition indicating the tetrahedral geometry around Mn(II).

The electronic spectra of Ni (II) , complex No.(10) shows two bands at (24630) and (28169) cm^{-1} which may be assigned to $^3A_2g \rightarrow {}^3T_1g$ and $^3A_2g \rightarrow {}^3T_1g$ (P) transitions respectively suggesting an octahedral arrangement around Ni(II) as compared with the reported value for octahedral Ni (II) complexes, while electronic spectra of Ni(II) complexes No. (01,06) exhibit one or two absorption bands (17211–17241)cm^{-1} and (25641)cm^{-1} which may be assigned to $^1A_1g \rightarrow {}^1A_2g$ and $^1A_1g \rightarrow {}^1B_1g$ transitions, suggesting a square planer geometry around Ni(II) , as compared with values of square planer Ni(II) complexes [29,30,31,32] .



Electronic spectra of $[Co(SPH)_2]Cl_2$



Electronic spectra of $[Ni(4\text{-}HSNPH)_2Cl_2]$

Table (3): Electronic spectral bands & chemical structure of some prepared hydrazone complexes

NO.	Complex	Band absorption		Assignment	Complex structure
		cm^{-1}	nm		
01	$[Ni(SH)_2]Cl_2$	42372	236	$n\rightarrow\pi^*$	**square planer**
		40485	247	$\pi\rightarrow\pi^*$	
		33967	294.4	C.T	
		25641	390	$^1A_1g\rightarrow{}^1B_1g$	
		17211	582	$^1A_1g\rightarrow{}^1A_2g$	
02	$[Cu(SH)_2]Cl_2$	46728	214	$\pi\rightarrow\pi^*$	**square planer**
		40650	246	C.T	
		15151	660	$^2B_1g\rightarrow{}^2A_1g$	
03	$[Co(SPH)_2]Cl_2$	47846	209	$\pi\rightarrow\pi^*$	**tetrahedral**
		39215	255	C.T	
		14727	679	$^4A_2\rightarrow{}^4T_1(P)$	
04	$[Cu(SPH)_2]Cl_2$	48543	206	$n\rightarrow\pi^*$	**square planer**
		47393	211	$\pi\rightarrow\pi^*$	
		38955	256.7	C.T	
		17271	579.4	$^2B_1g\rightarrow{}^2Eg$	
05	$[Mn(SDNPH)_2]Cl_2$	47619	210	$\pi\rightarrow\pi^*$	**tetrahedral**
		34013	294	C.T	
		17543	570	$^6A_1g\rightarrow{}^4T_1g$ (D)	
06	$[Ni(SDNPH)_2]Cl_2$	42372	236	$n\rightarrow\pi^*$	**square planer**
		40551	246.6	$\pi\rightarrow\pi^*$	
		33444	299	C.T	
		17241	580	$^1A_1g\rightarrow{}^1A_2g$	
07	$[Cu(SDNPH)_2]Cl_2$	49019	204	$n\rightarrow\pi^*$	**square planer**
		43290	231	$\pi\rightarrow\pi^*$	
		41152	243	C.T	
		17857	560	$^2B_1g\rightarrow{}^2Eg$	
08	$[Mn(4\text{–}HSNPH)_2Cl_2]$	46948	213	$n\rightarrow\pi^*$	**octahedral**
		45871	218	$\pi\rightarrow\pi^*$	
		40000	250	C.T	
		28409	352	$^6A_1g\rightarrow{}^4Eg(D)$	
		26666	375	$^6A_1g\rightarrow{}^4T_2g(D)$	
09	$[Co(4\text{–}HSNPH)_2Cl_2]$	49019	204	$n\rightarrow\pi^*$	**Octahedral**
		44642	224	$\pi\rightarrow\pi^*$	
		35971	278	C.T	
		23364	428	$^4T_1g(F)\rightarrow{}^4T_1g(P)$	
10	**$[Ni(4\text{–}HSNPH)_2Cl_2]$**	**47846**	**209**	$n\rightarrow\pi^*$	**Octahedral**
		46511	**215**	$\pi\rightarrow\pi^*$	
		39370	**254**	C.T	
		28169	**355**	$^3A_2g\rightarrow{}^3T_1g(P)$	
		24630	**406**	$^3A_2g\rightarrow{}^3T_1g$	

Conductivity Measurements

The molar conductivity of all prepared hydrazone (Schiff bases) complexes were measured for (10^{-3}M) in dimethyl sulfoxide (DMSO) at (room temperature ~25°C) .

The values of the molar conductance ($cm^2.ohm^{-1}.mol^{-1}$) in this solvent are given in Table (4).

The observed values in the (DMSO) are in the ranges (75–120 cm^2 $ohm^{-1}mol^{-1}$) which correspond to (1:2) electrolytes, this is also confirmed with positive $AgNO_3$ test for chloride containing complexes. Other complexes are non–electrolytes by the observing molar conductance (1.2–19.8 $cm^2ohm^{-1}mol^{-1}$) which are also confirmed with negative $AgNO_3$ test for chloride ions .

Table (4): Formula and physical properties of the prepared hydrazone complexes

No.	Complex	Color	Melting point(°C)	DMSO $cm^2ohm^{-1}mol^{-1}$	$AgNO_3$ Test
01	$[Mn(SH)_2]Cl_2$	Yellow	>230*	80.5	+
02	$[Co(SH)_2Cl_2]$	Brown	174–175	19.8	-
03	$[Ni(SH)_2]Cl_2$	Green	>289*	87.6	+
04	$[Cu(SH)_2]Cl_2$	Greenish–yellow	208	84.9	+
05	$[Mn(SPH)_2Cl_2]$	Deep yellow	124–125	1.7	-
06	$[Co(SPH)_2]Cl_2$	Pink	>153*	120	+
07	$[Ni(SPH)_2]\ Cl_2$	Greenish-yellow	>340*	93	+
08	$[Cu(SPH)_2]Cl_2$	Dark green	>194*	75	+
09	$[Mn(SDNPH)_2]Cl_2$	Red	244–245	81.8	+
10	$[Co(SDNPH)_2]Cl_2$	Red	>246*	92.2	+
11	$[Ni(SDNPH)_2]Cl_2$	Reddish-orange	252	78.3	+
12	$[Cu(SDNPH)_2]Cl_2$	Red	248	84.2	+
13	$[Mn(4–HSNPH)_2Cl_2]$	Red	>288*	1.9	-
14	$[Co(4–HSNPH)_2Cl_2]$	Deep red	>279*	1.4	-
15	$[Ni(4–HSNPH)_2Cl_2]$	Red	>291*	1.2	-
16	**$[Cu(4–HSNPH)_2]Cl_2$**	**Green**	**>326***	**89**	+

*Decomposition

Conclusion

From the previous results and discussion we concluded the following :

1. The neutral molecules of hydrazone ligands were coordinated to each of the metal ions studied as bidentate ligands via two bonding sites which are central azomethine nitrogen and OH group.

2. The complexes may have coordination number (four or six) depending on the number of coordinated sites of the ligands and the anion present .

3. According to the conductivity measurements of the complexes, some of hydrazone complexes are non electrolytes .

4. Factors such as the coordinating ability of substituents on an azomethine group, properties of central metal ions and preparative conditions may affect the coordinating mode azomethine group .

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تحضير وتشخيص بعض معقدات العناصر الانتقالية مع ليكاندات الهايدرازون

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الملخص

يتضمن هذا البحث تحضير وتشخيص سلسلة من معقدات العناصر الانتقالية الحاوية على ليكاندات الهايدرازون حيث استخدمت الهايدرازونات الاتية :

Salicyl hydrazone (SH), Salicyl phenylhydrazone (SPH), salicylaldehyde -2,4-dinitrophenylhydrazone(SDNPH), 4-hydroxy salicylaldehyde -4-nitrophenylhydrazone(4-HSNPH) .

تم دراسة خصائص المعقدات المحضرة اعتمادا على خواصها الفيزياوية والقياسات التوصيلية المولارية وطيف الاشعة تحت الحمراء (IR) والطيف الالكتروني(uv-vis) والتحليل الدقيق للعناصر لبعض المعقدات .

اشارت قياسات التوصيلية المولارية الى ان بعض معقدات الهايدرازونات غير الكتروليتية اي متعادلة وتتناسق الليكاندات بشكل ثنائى السن مكونة معقدات سداسي التناسق وتأخذ شكل ثماني السطوح من نوع [ML_2Cl_2] حيث:(M^{+2}= Mn,Co,Ni and Cu) او معقدات آيونية رباعي التناسق و وتأخذ شكل رباعي السطوح أو مربع مستوي نوع Cl_2 [ML_2] حيث: (M^{+2}= Mn, Co,Ni and Cu) .