

Synthesis and Characterization of New Metal Complexes with Schiff Base of Disalicyaldenesuccinoyldihydrazide (DSSH)

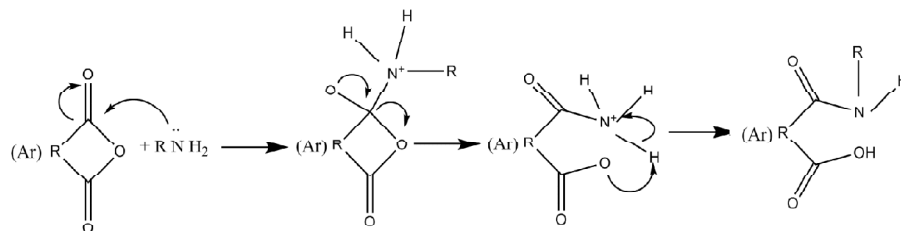
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Abstract: A new tetra dentate (N_2O_2) type Schiff base ligand disalicyaldenesuccinoyldihydrazide (DSSH) was synthesized from the condensation of succinoyldihydrazide and Salicyaldenide. Monomeric complexes of this ligand, of general formula $[Cr(DSSH)(H_2O)_2]NO_3$, $[Ni(DSSH)]H_2O$, $[Co(DSSH)]$, $[Cu(DSSH)]$ AND $[Cd(DSSH)]$ are prepared. The mode of bonding and overall geometry of the complexes were determined through FTIR, UV-Vis spectra GC-mass spectrometer, Magnetic susceptibility, microanalysis (C.H.N.), metal content and molar conductivity. These studies revealed tetrahedral geometries for the Cu(II), Co(II), Ni(II) and Cd(II), octahedral for Cr(III) complex.

Introduction

Amic acids or amidoacids can be prepared by the reaction of aliphatic amines with different cyclic anhydrides such as maleic, phthalic, succinic and citraconic anhydrides [1]. Reaction of an equimolar mixture of amines with anhydrides below $100^\circ C$ yield the acid amide quantitatively. Two factors generally determine the trend of the reaction: temperature and relative amount of the components whereas the reaction time is of minor importance [2]. The mechanism of the reaction of amic acid production involves nucleophilic attack on the carbonyl group followed by ring opening as illustrated in the following scheme.



$R = (CH_2)_2$, Aryl

$R = H$, aryl.

Considerable work has been mainly described for synthesis of amic derived from maleic anhydride and its derivatives, reaction of the anhydrides with primary amines were reported to give two isomers, while reaction of maleic or succinic anhydride with secondary amines gave one isomer only [3]. No information are available about amic acids erived from tertiary amines. In the metal complexes of Schiff bases, the presence of azomethine group with a lone pair of electrons on the nitrogen atoms provides a binding site for coordination with a metal ion [4], which can stabilize the double bond of the Schiff base [5]. Information on metal complexes with Schiff bases as a monodentate ligand are limited [6] interest seems concentrated on more coordinatate sites, Bidentate [7, 8], tridentate [9, 10] and tetradentate Schiff bases derived from condensation of θ -substituted aromatic amines, diamines and aliphatic diamines with α , β , γ -tricarboxyls substituted aromatic carbonyls were found chelating towards a wide range of metal ions [11, 12].

Experimental

All the chemicals the metal salts, reagents and solvents such as $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, succinic anhydride, pthalic anhydride, salicylaldehyde, dimethyl formamide (DMF) and ethanol were obtained from BDH, Merck and Fluka respectively.

All the above chemical have been used without further purification. Conductivity measurements of the metal complexes were recorded at 25°C for 10^{-13}M solutions of the samples in DMF, using (Hand-Held Meter LF330).

The electronic spectra of the free Schiff base ligand and its metal complexes were recorded in DMF solution, using (Shimadzu model 160A UV-Vis. Spectrophotometer).

IR spectra KBr disc in the range $(4000-400)\text{Cm}^{-1}$ were recorded by using (Shimadzu, IR-Prestige-21 FTIR 8400S) Fourier Transform Infrared spectrophotometer. Flame atomic absorption spectrophotometer were obtained by using (Shimadzu 160AA). Elemental microanalysis for (C, H and N) were carried out on (Eurovector EA3000A),

University of Babel. Magnetic susceptibility of the metal complexes at 25°C were determined with magnetic susceptibility balance (Johson Matthey) in Al-Nahrain University. Melting points were measurement by using (Gallen Kamp Melting point).

GC-Mass Spectrometer was carried out in THF solvent for the free Schiff base ligand by using (Shimadzu, Q_p 2010).

Preparation of Disalicylaldehyde Succinoyl Dihydrazide (DSSH)

1. Preparation of Succinoyl diHydrazide

Succinic anhydride (1gm, 10 mmole) was dissolved in absolute ethanol (10Cm^3) with stirring. The hydrazine hydrate (1gm, 20 mmole) was dissolved in absolute ethanol (10Cm^3) and added to the succinic anhydride solution. The mixture solution heated under reflux at temperature (60 °C) for (10 min) until precipitation of the white solid was complete. And

then the solid collected by filtration, washed with ethanol (15 $\text{Cm}^3 \times 2$), dried at temperature (50 °C) for two hours. (yield = 73%), m.p = 170°C.

2. Preparation of disalicylaldehyde Succinoyl dihydrazide

Succinoylhydrazide (1gm, 10 mmole) was dissolved in ethanol (20 Cm^3) with stirring. Salicylaldehyde (2.442gm, 20 mmole) was dissolved in ethanol (5 Cm^3) and added to the solution of Succinoylhydrazide. The mixture Solution was heated under reflux at temperature (60–70 °C) for three hours until the precipitation of the yellow solid was complete. And then the solid Collected by filtration, washed with water (10 $\text{Cm}^3 \times 3$), dried at temperature (50°C) for three hours and then recrystallized from absolute ethanol. The yellow crystals were collected by filtration and then dried at temperature 50C° for three hours.

Results and Discussion


Infrared Spectra of disalicylaldehydesuccinoyl dihydrazide (DSSH) and its metal Complexes

The infrared spectrum of the free Schiff base ligand showed characteristic the stretching vibration of (OH) of phenolic group at 3263 Cm^{-1} .

The band at 3213 Cm^{-1} assigned to the stretching vibration of (-N-H), the bands at 3043 and 2970 Cm^{-1} attributed to the stretching vibration of (-C-H) of aromatic and aliphatic respectively, the band at 1624 Cm^{-1} attributed to stretching vibration of (-C=O), the band at 1573 Cm^{-1} assigned to the stretching of (HC=N), the band at (1485-1450) Cm^{-1} due to the stretching vibration of (-C=C). the bands at 1199 and 1153 Cm^{-1} attributed to stretching vibration of (-C-O) and (-C-N) respectively.

The stretching vibration of (-C=N) band at 1573 Cm^{-1} which was shifted to a lower frequency (40-50) Cm^{-1} due to the coordination of the nitrogen of the azomethine group in all the spectra of the metal complexes [13].

The stretching vibration of (OH) was disappeared in all the metal complex formation. This band indicates the participation of the oxygen atom of the hydroxyl of phenolic group in coordination [14].

In the free Schiff base ligand, strong band was showed at 1624 Cm^{-1} due to the stretching vibration of (-C=O). since no significant change in this band was noticed for the cadmium complex which was indicated that the coordination was not occur. This band was shifted to lower frequency in the other complexes which was indicated the complex formation. The weak broad bands at 3417, 3305 and 3444 Cm^{-1} due to the stretching vibration of (-N-H) due to the intramolecular hydrogen bonding of  for the chromium, copper and cadmium complexes respectively, which was appeared very weak band at cobalt complex.

The IR spectrum of nickel complex has a broad band at region 3363 Cm^{-1} was assigned to the stretching vibration of (OH) of water indicated the presence of water molecule, which was overlapped with the band of itramolecular hydrogen bonding [15].

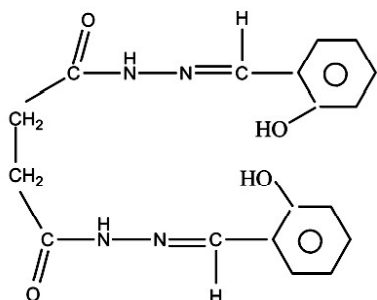
The new stretching vibration bands were observed around $(428-489)\text{cm}^{-1}$ and $(474-586)\text{cm}^{-1}$ which were assigned to the frequency of (metal-oxygen) and (Metal-nitrogen) respectively.

All the IR spectra of ligand and metal complexes are illustrated in table (1) and figures (1), (2), (3), (4), (5) and (6).

Table 1
The Infrared Spectra for the Free Ligand (DSSH) and its Complexes in (cm^{-1})

Compound	ν (OH)	ν (N-H)	ν (C-H)ar.	ν_{as} (C-H)al	ν (C=O)	ν (C=N)	ν (C-O)	ν (C-N)	ν (N-N)	ν $\nu(M-O)$ $\nu(M-N)$	
DSSH	3263	3213	-	3043	2970	1624	1573	1199	1153	1029	=
[C _r (DSSH) (H ₂ O) ₂]NO ₃	3444	-	3417	3050	2835	1604	1535	1199	1153	1018	489516
[Ni(DSSH)]H ₂ O	3363	-	-	3055	2939	1604	1539	1192	1153	1037	428586
[Co(DSSH)]	-	-	-	3032	2912	1604	1523	1192	1149	1072	428540
[Cu(DSSH)]	-	-	3305	3039	3008	1612	1531	1195	1149	1018	428497
[Cd(DSSH)]	-	-	3464	3016	2962	1624	1543	1188	1149	1037	435474

Where DSSH =



Electronic Spectra of disalicyaldenesuccioyldihydrazide (DSSH) and its Metal Complexes

The UV-visible spectra of the free Schiff base ligand and its complexes were carried out in their solutions in 10^{-3}M dimethylformamide (DMF) in the range (200-900)nm. The electronic spectrum of the free Schiff base ligand showed two intense bands, the first band observed at 304nm (32895Cm^{-1}) which was assigned to $(\pi-\pi^*)$ electronic transition of the aromatic ring [16], and the other band at 358nm (27933Cm^{-1}) assigned to $(n-\pi^*)$ electronic transition of the (C=N) chromophore [17].

The electronic spectrum of chromium (III) complex shows five bands, the first strong bands showed at 304nm (32895Cm^{-1}) assigned to $(\pi-\pi^*)$ electronic transition, and the other four bands, showed at 431nm (23202Cm^{-1}), 512nm (19531Cm^{-1}), 686nm (14577Cm^{-1}) and 741nm (13495Cm^{-1}) attributed to charge transfer (C.T.), ${}^4A_{2g} \rightarrow {}^4T_{1g}$, ${}^4A_{2g} \rightarrow {}^4T_{2g}$, ${}^4A_{2g} \rightarrow {}^2T_{2g}$

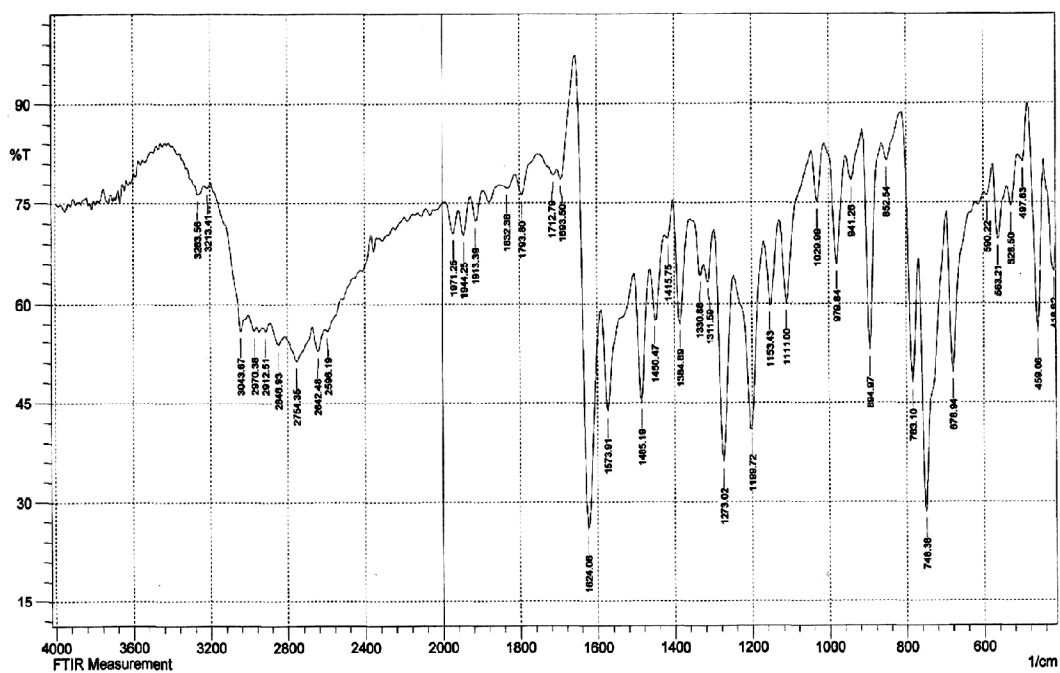


Figure 1: Infrared spectrum of disalicylaldehydesuccinoyldihydrazone (DSSH)

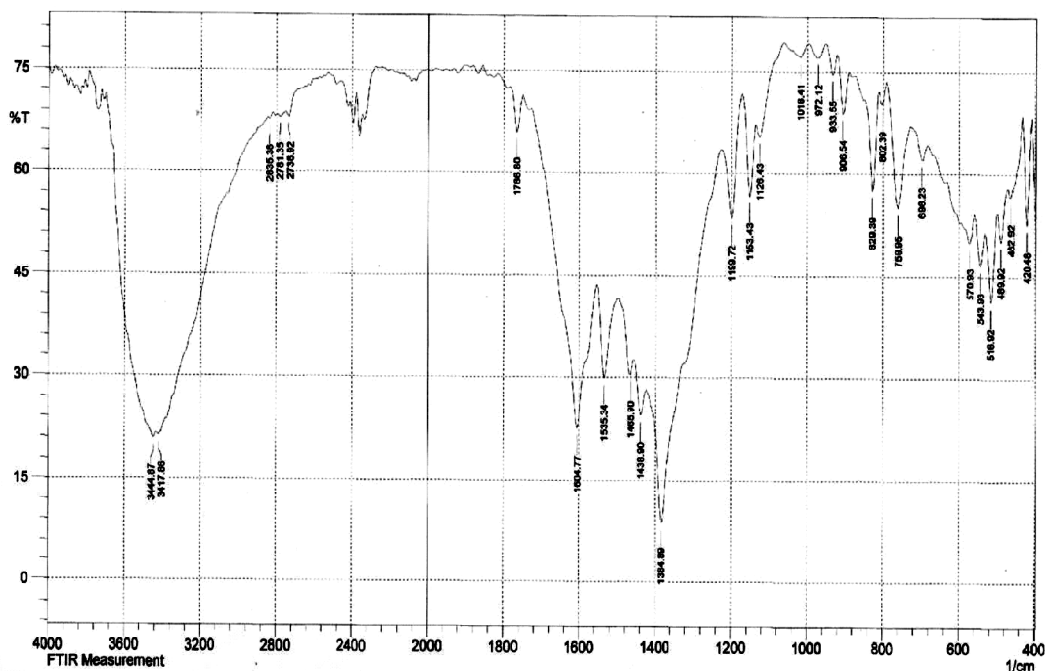


Figure 2: Infrared spectrum of (DSSH)Cr complex

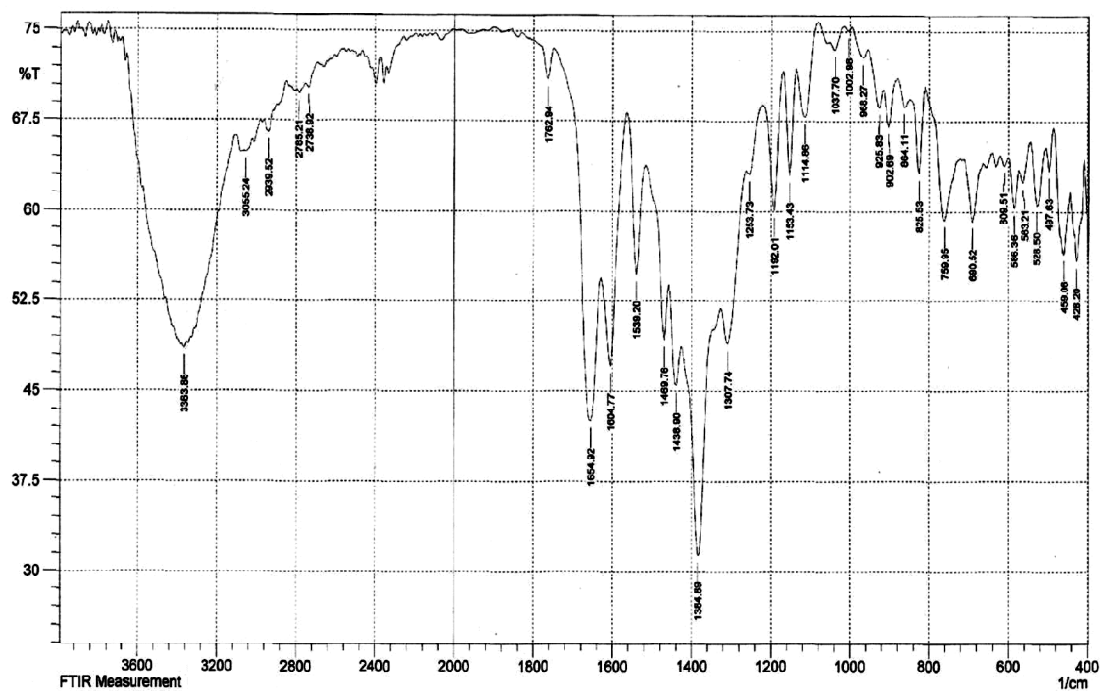


Figure 3: Infrared spectrum of (DSSH)Ni complex

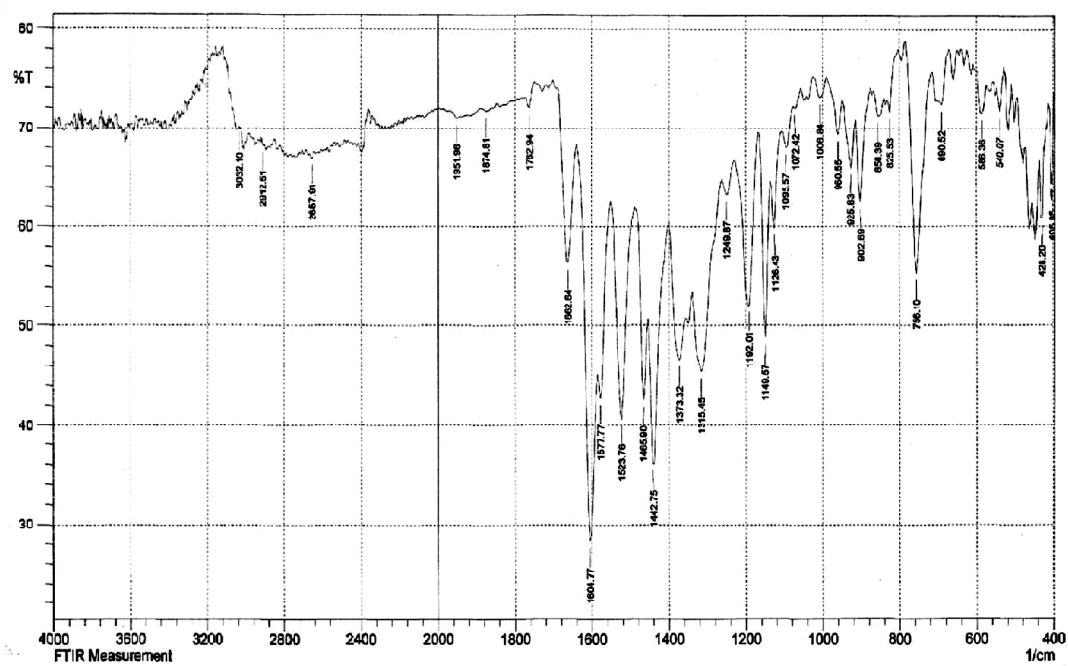


Figure 4: Infrared spectrum of (DSSH)Co complex

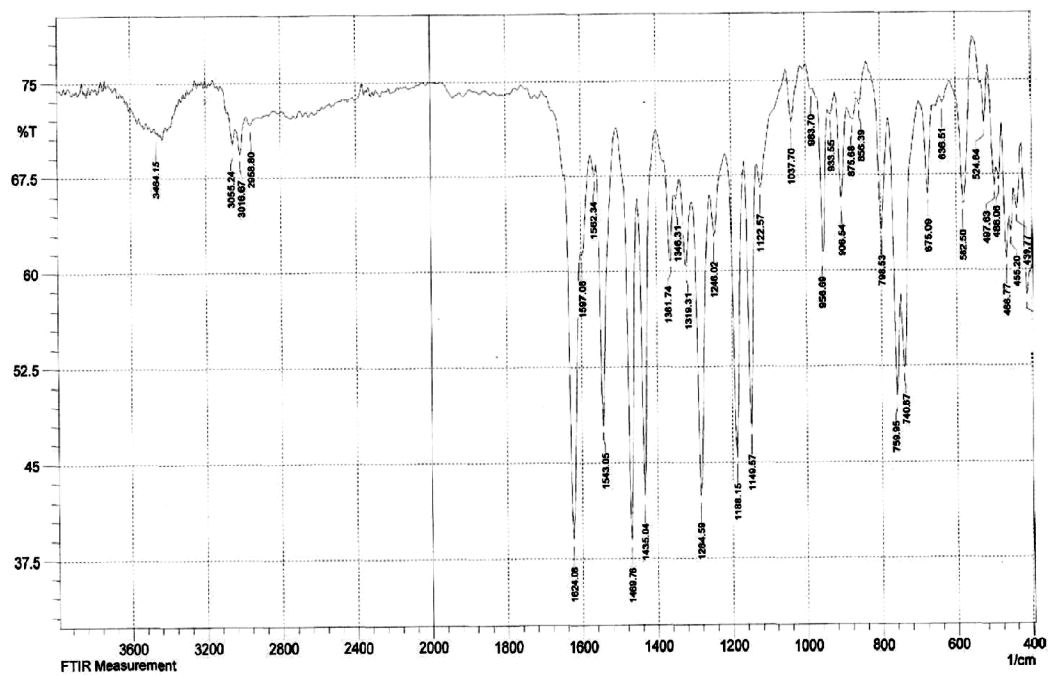


Figure 5: Infrared spectrum of (DSSH)Cu complex

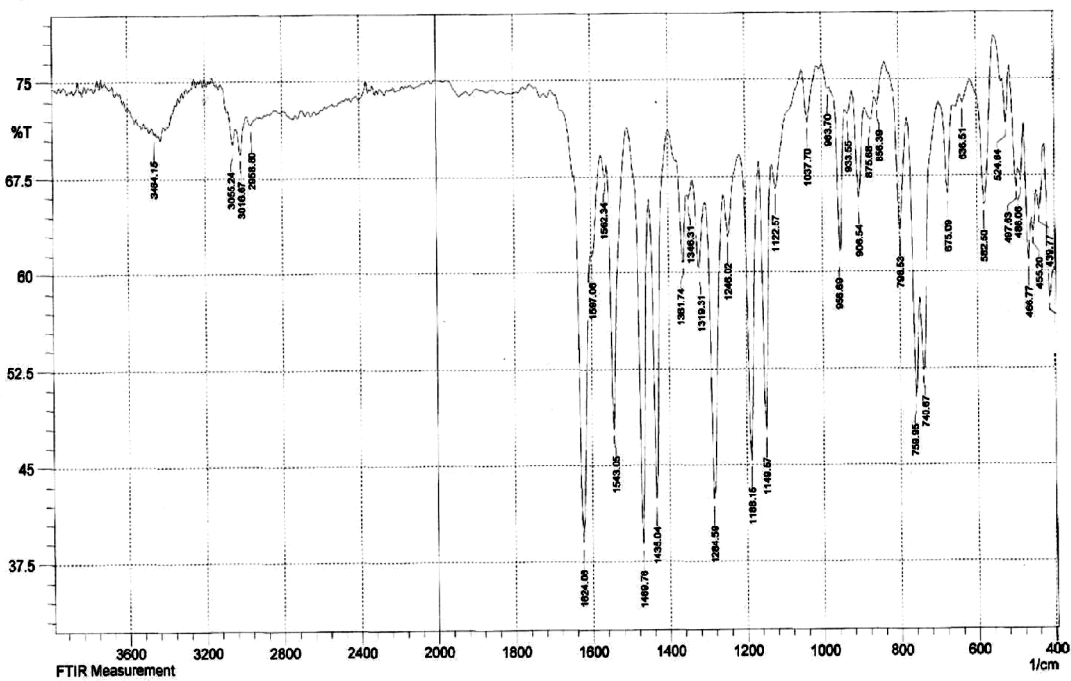


Figure 6: Infrared spectrum of (DSSH)Cd complex

Transitions respectively, from these transitions observed for chromium complex suggest the octahedral geometry of the complex [18].

The electronic spectral bands of the cobalt complex, showed four bands, one at 312nm (3205Cm^{-1}) assigned to ($\pi-\pi^*$) transition, the other three band absorption at visible region showed at 452nm (22124Cm^{-1}), 586nm (17065Cm^{-1}) and 848nm (11792Cm^{-1}) can be assigned to charge transfer (C.T.), $^4A_2 \rightarrow ^4T_1(P)$, $^4A_2 \rightarrow ^4T_1(F)$ transitions respectively. The values of these transitions were characterized to tetrahedral geometry around the cobalt ion [8].

The yellowish green of the nickel (II) complex shows three bands one of them observed at 301nm (32051Cm^{-1}) can be assigned to ($\pi-\pi^*$) electronic transition, the second at 404nm (24752Cm^{-1}) assigned to charge transfer transition (C.T.) and the third band showed at 486nm (20576Cm^{-1}) due to $^3T_1 \rightarrow ^3T_1(P)$ transition. This confirms the presence of tetrahedral geometry for nickel complex [19].

The light brown of copper (II) complex exhibited three bands having wavelength one of them showed at 304nm (32895Cm^{-1}) assigned to ($\pi-\pi^*$) transition, the other two bands observed at 407nm (24570Cm^{-1}) and 685nm (14598Cm^{-1}) attributed to charge transfer (C.T.) and $^2B_2 \rightarrow ^2E$ respectively. These transitions suggested tetrahedral geometry of copper complex [20].

The Pall yellow of cadmium (II) complex showed two bands one of them observed at 320nm (31250Cm^{-1}) exhibited to ($\pi-\pi^*$) transition, the other band at 416nm (24038Cm^{-1}) attributed to charge transfer ($M \rightarrow L$). This is because of the electronic configuration and diamagnetic of this complex which confirmed the absence of any (d.d) electronic transition. The spectra are typical of cadmium complex with tetrahedral geometry for cadmium complex [21].

In the free Schiff base, the band at 358nm which is assigned to ($n-\pi^*$) transition of the (C=N) chromophore and this band was shifted to higher wavelength in all the metal complexes, this is confirmed the coordination of the nitrogen atom of the azomethine group with the central metal ion [22].

The interaction of the metal ions with the ligand showed the absorption spectra of a peak in the range of visible region. A great bathchromic shift in the visible region was detected in the complex solutions spectra with respect to that of the free Schiff base ligand. The high shift in the (λ_{max}) gave good indication for the metal complex formation.

The table (2) and figures (7) (8), (9), (10), (11) and (12) showed a comparison between the spectra of the free Schiff base ligand and the metal complexes.

GC-Mass Spectrometer of disalicyaldenesuccinoyldihydrazide (DSSH)

The main fragmentation GC-mass spectrometer of the free Schiff base ligand (disalicyaldenesuccinoyldihydrazide which is illustrated in figures (13)). The molecular ion peak of the ligand is observed at $m/z=352[M]^+$ for $C_{18}H_{16}N_4O_4$ requires = 352.

Table 2
Electronic Spectra Data of the Free Ligand (DSSH) and its Metal Complexes

Compound	$\lambda(\text{nm})$	Wave number (cm^{-1})	Absorbance	ϵ_{max} ($\text{Mol}^{-1} \cdot \text{l. Cm}^{-1}$)	Assignment	Suggested structure
(DSSH)	304	32895	1.37	1370	$\pi-\pi^*$	-
	358	27933	1.615	1615	$n-\pi^*$	
[Cr(DSSH)(H ₂ O)]NO ₃	304	32895	1.42	1420	$\pi-\pi^*$	Octahedral
	431	23202	0.828	828	C.T.	
	512	19531	0.11	110	${}^4A_{2g} \rightarrow {}^4T_{1g}(\text{F})$	
	686	14577	0.005	5	${}^4A_{3g} \rightarrow {}^4T_{3g}$	
	741	13495	0.003	3	${}^4A_{3g} \rightarrow {}^4T_{3g}$	
[Ni(DSSH)]H ₂ O	301	33223	0.824	824	$\pi-\pi^*$	Tetrahedral
	404	24752	1.954	1954	C.T.	
	486	20576	0.11	110	${}^3T_1 \rightarrow {}^3T_1(\text{P})$	
[Co(DSSH)]	312	32051	1.989	1989	$\pi-\pi^*$	Tetrahedral
	452	22124	1.57	1570	C.T.	
	586	17065	0.022	22	${}^4A_2 \rightarrow {}^4T_1(\text{P})$	
	848	11792	0.018	18	${}^4A_2 \rightarrow {}^4T_1(\text{F})$	
[Cu(DSSH)]	304	32895	0.607	607	$\pi-\pi^*$	Tetrahedral
	407	24570	0.531	531	C.T.	
	685	14598	0.04	40	${}^2B_2 \rightarrow {}^2E$	
[Cd(DSSH)]	320	31250	0.99	990	$\pi-\pi^*$	Tetrahedral
	416	24038	1.195	1195	C.T.	

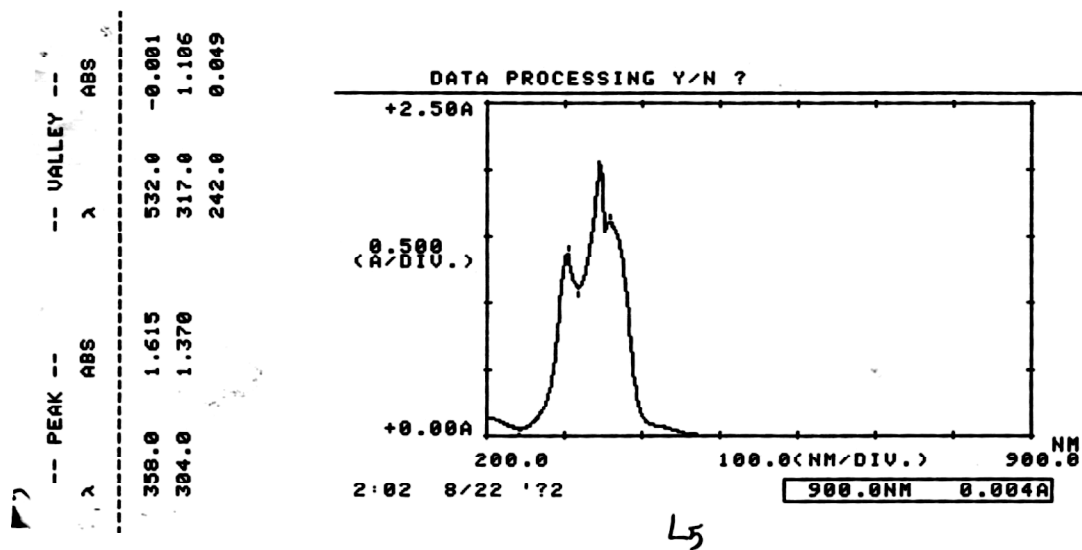


Figure 7: Electronic Spectrum of DisalicyaldineSucclnoyldihydrazide (DSSH)

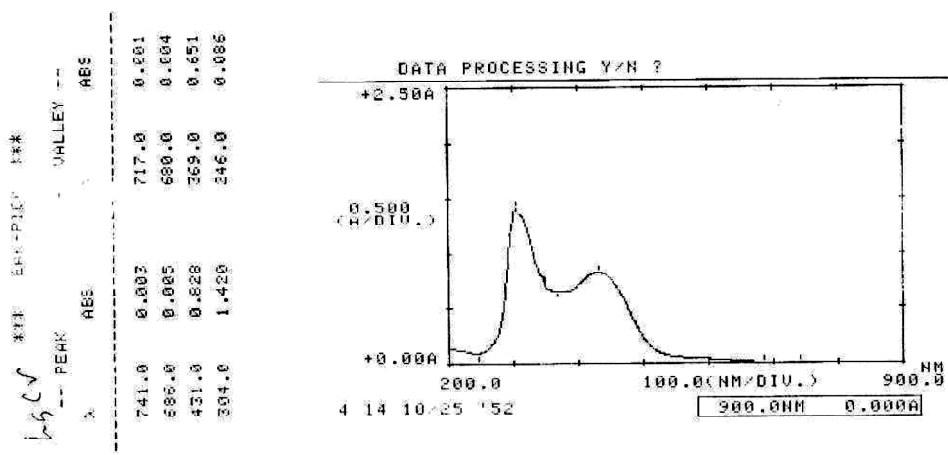


Figure 8: Electronic Spectrum of (DSSH)Cr complex.

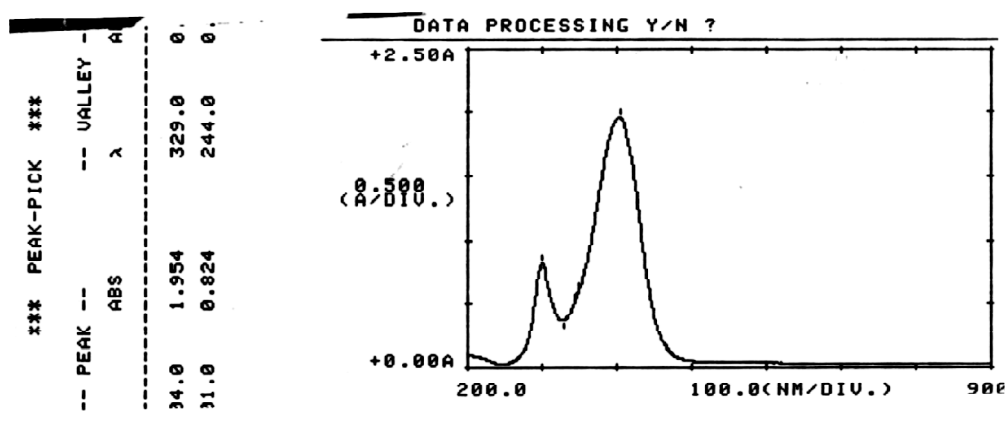


Figure 9: Electronic Spectrum of (DSSH)Ni complex.

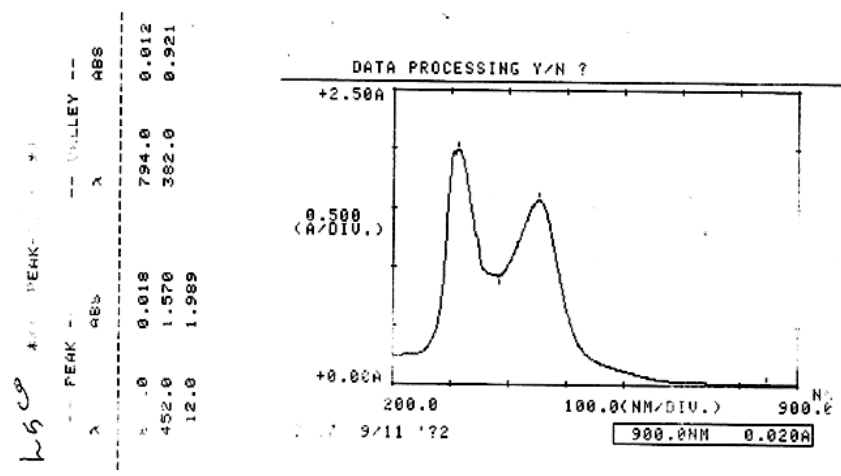


Figure 10: Electronic Spectrum of (DSSH)Co complex.

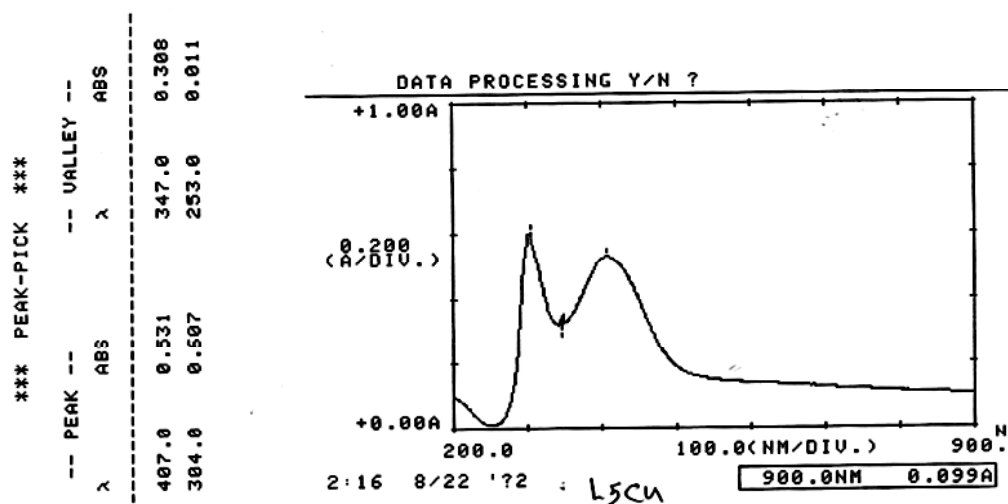


Figure 11: Electronic Spectrum of (DSSH)Cu complex.

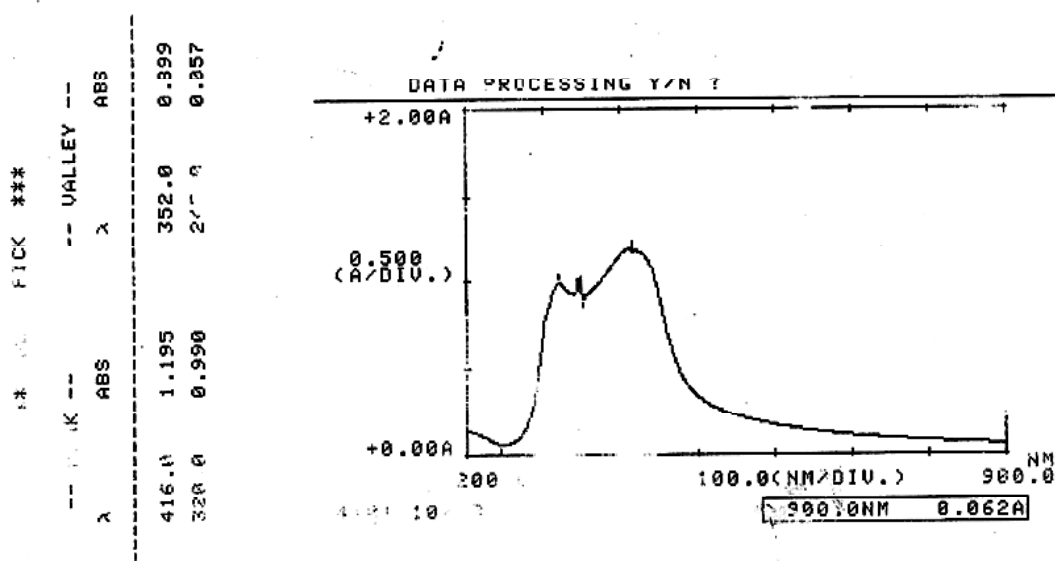


Figure 12: Electronic Spectrum of (DSSH)Cd complex.

The other peaks detected at $m/z=222$ (relative abundance is 35%), 205 (relative abundance is 100%), 177 (relative abundance is 15%), 145 (relative abundance is 15%) and 57 (relative abundance is 20%), corresponding to $[M-(8H_5+CH=CN_2)]$, $[M-(CH_3+2H^+)]$, $[M-(CO)]$, $[M-(N_2H_4)]^+$ and $[M-(C_7H_4)]$ respectively.

The fragmentation pattern of the molecular ion of the free Schiff base ligand in scheme (1).

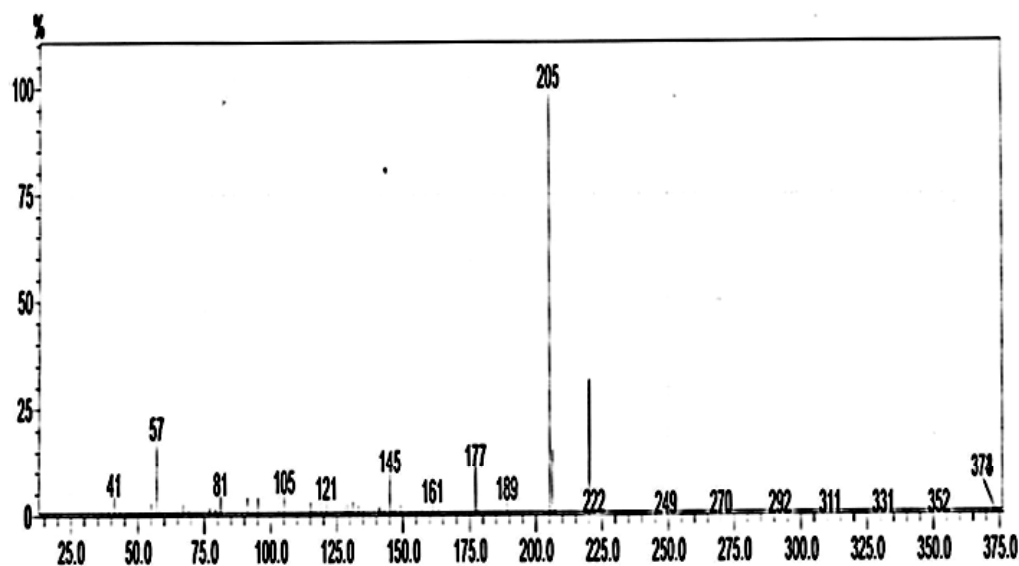


Figure 13: Gc - Mass Spectrometer of disalicyaldenesuccinoyldihydrazide (DSSH)

Physical Properties of the Free Schiff base Ligand and their Complexes

The molar conductance measurements suggested the absence of union (nitrate) outside the coordination sphere for nickel (II) complex, cobalt (II) complex, copper (II) complex and cadmium (II) complex which are indicated that metal complexes are non-electrolytic nature [23]. The molar conductance of the chromium (III) complex reveals the presence of nitrate ion outside the coordination sphere, which is indicated the chromium (III) complex are electrolytic nature. The metal forms ratio 1:1 complexes with Schiff base ligand [24].

The magnetic momentums of chromium (III) d^3 , and cobalt (II) d^3 complexes were found to be (3.70), (3.72) B.M. respectively, within the expected spin only values. Moreover, the higher value of the magnetic momentum for nickel (II) d^8 complexes was found to be (3.4) B.M. which is due to the orbital contribution [23]. the magnetic momentum for copper (II) d^9 complexes were found to be (1.48)B.M. within expected value for one electron. Finally the magnetic momentum for cadmium (II) d^{10} complexes are diamagnetic as expected from its electron configuration [3]. From the above values, indicated all the metal complexes are paramagnetic (high spin) except the cadmium complex is diamagnetic [25].

The microanalysis (C.H.N) and metal contents of the free Schiff base ligands and their metal complexes were obtained using (Euro vector EA3000 single V.3.0 single) and flame atomic absorption spectrophotometer (Shimadzu 680 A).

The data of the free Schiff base ligands and their metal complexes were in good agreements with the calculated values, which are indicated the identities and purities of the ligand and their metal complexes.

All these data are summarized in table (3).

Table 3
The Physical Properties and Microanalysis of the Ligand (DSSH) and their Complexes

Compound	yield	Color	m.p(°C)	M% (calc.) found	C%	Microanalysis (Calc.) Found	N%`	Molar conductance $\mu_s \text{ cm}^{-1}$	μ_{eff} . (B.M.)
DSSH	80%	Yellow	210	-	(61.02)	(5.08)	(15.82)	-	-
[Cu(DSSH)]	76%	Light brown	190	(15.30) 15.645	(51.98)	(3.85)	(13.47)	12.9	1.48
[Ni(DSSH)]H ₂ O	73%	Yellowish green	240	(13.64) 12.71	(50.38)	(3.73)	(13.06)	40.3	3.42
[Cr(DSSH)(H ₂ O) ₂] NO ₃	75%	Greenish brown	290	(10.36) 10.42	(43.03)	(3.98)	(11.155)	76.9	3.70
[Co(DSSH)]	69%	Dark brown	160	(14.34) 13.645	(52.56)	(3.89)	(13.63)	18.3	3.72
[Cd(DSSH)]	65%	Pall yellow	300	(24.2) 23.271	(46.51)	(3.44)	(12.06)	14.2	zero
					47.1	3.52	12.42		

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