

Synthesis and Characterization Some of Metals (II) Complexes' with Mixed Ligands of saccharinate and Isatin Schiff Base

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Abstract

This research paper includes synthesis and characterization of some complexes of Zn(II), Hg(II), Cd(II), Cu(II), Co(II), Ni(II) with mix ligands of saccharinate and Schiff base derived from Isatin. All the synthesized complexes have been characterized by elemental analysis, molar conductance, uv-visible, magnetic susceptibility, atomic absorption, infrared and ¹H NMR spectroscopy. Metal to ligand [M:L] ratio was obtained for all complexes in ethanol. The concluded chemical formulas of the complexes were [M(sac)₂(L)₂], where {M=(Zn(II), Hg(II), Cd(II))} tetrahedral geometry and for the complexes [M(sac)₂(L)(H₂O)₂], where (M=Co, Ni and Cu), were octahedral geometry, sac=saccharinate, and L=C₁₄H₁₀N₂O₂.

Key words: Schiff base, isatin, saccharin, complexes

Introduction

A large number of Schiff bases of isatin and their complexes have been studied due to their important properties. The high affinity for chelation of the Schiff bases towards transition metal ions is utilized in preparing their solid complexes[1]. These compounds have a wide range of applications such as antibacterial[2-4], antifungal[2,3,5], antimicrobial [2,6], antileukemic[7], anticancer[2,8,9]. On other hand the researchers are interested in saccharin chemistry and its complexes because it was expected to be carcinogenic[10], but after many studies it had been suggested as antidote metal poisoning [11]. Stability of some polymeric saccharin complexes for Ag(I), Tl(I) and Pb(II), give a great hope for the potential benefit of saccharin as promising molecule as chelating link [12-14], and discovered in recent years, a new biological effects of different and many of which are of a nature medical mission[15,16], examples include possession of saccharin sulfur (thiosaccharine) and some of its derivatives activity effective against germs[17].

The present paper reports synthesis and characterization of new Zn(II), Hg(II), Cd(II), Cu(II), Co(II), Ni(II) complexes with mixed ligand of isatin Schiff's base and saccharinate.

Experimental

Instrumentation

UV-Visible spectra were recorded on a (Shimadzu UV -160 A) Ultra Violet- Visible spectrophotometer, I.R- spectra were recorded in the (FT-IR (Perkin Elmer)) Fourier Transform Infrared Spectrophotometer (4000-400)cm⁻¹ range with samples prepared as KBr discs. Micro elemental analysis (C.H.N.S) conducted by using (Euro EA 3000). Molar Conductivities were measured for 10⁻³ M solution of complexes in ethanol at 22C⁰ by using (Cond7110). Melting points were obtained by using

(Melting Point Apparatus). Magnetic Susceptibility was measured by using (MSB-MKI) Magnetic Susceptibility moder) Balance. Atomic Absorption was conducted by using (Atomic absorption spectrophotometry (Aurora). The ¹H NMR spectrometer (solvent DMSO-d⁶) were recorded in the (NMR eady 60 Pro 60 MHz High resolution 17.7 bit).

Synthesis of Schiff Base Ligand C₁₄H₁₀N₂O₂ (L):

Schiff base ligand (Z)-3-((4-hydroxyphenyl) imino) indolin-2-one, was synthesizable as described in the literature [18]

Synthesis of metal complexes of Saccharin

[M(sac)₂(H₂O)₄].2H₂O

Saccharinate metal complexes of the type [M(sac)₂(H₂O)₄].2H₂O were prepared and isolated as described in the literature [19-21]

Synthesis of [Hg(sac)₂]

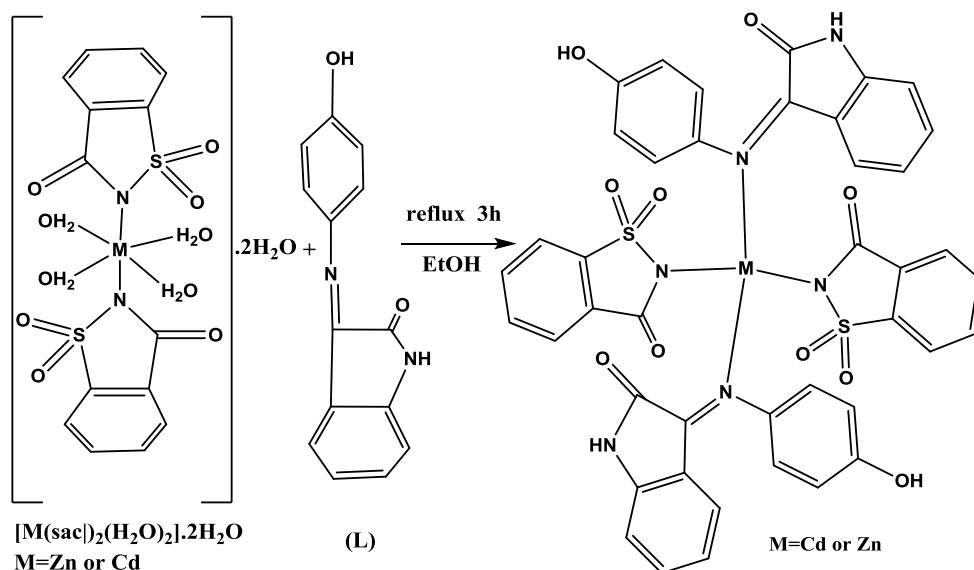
Mercury saccharin was synthesized and isolated as described in the literature [22,23]

Synthesis of complex [Hg(sac)₂(L)₂]

This complex was synthesized in molar ratio (1:2), (metal saccharin complex: schiff base ligand) using the following procedure:

A solution of schiff base ligand (0.47g, 0.002mol) in ethanol was added to a solution of metal saccharin complex(0.56g, 0.001mol) in ethanol, the mixture was refluxed for (3h), the solution formed was left for evaporation at room temperature affording an orange powder. The solid product was washed in methanol and dried in oven at 100C⁰, where the sediment weight (0.75g) and the percentage of the product 73%.

The complexes [Cd(sac)₂(L)₂] and [Zn(sac)₂(L)₂] were synthesized using similar method starting from zinc saccharinate and cadmium saccharinate: as shown in the equation below:



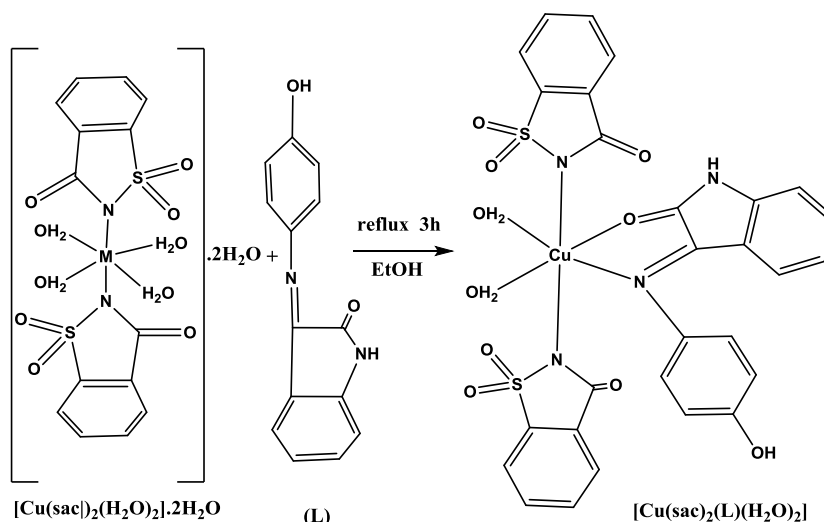
Synthesis of complex $[\text{Ni}(\text{sac})_2(\text{L})(\text{H}_2\text{O})_2]$

This complex was synthesized in mole ratio (1:1), (metal saccharin complex: schiff base ligand) using the following procedure :

A solution of schiff base ligand (0.24g, 0.001mol) in ethanol was added to a solution of metal saccharin complex (0.53g, 0.001mol) in ethanol, the mixture was refluxed for (3h), the orange solution formed left for solvent to evaporate at room temperature to give

brown solid which filtered off and recrystallized from methanol and dry at a temperature of $100\text{ }^\circ\text{C}$, product was with brown color, its weight was (0.5g) the percentage of product 73%.

The complexes $[\text{Co}(\text{sac})_2(\text{L})(\text{H}_2\text{O})_2]$ and $[\text{Cu}(\text{sac})_2(\text{L})(\text{H}_2\text{O})_2]$ were synthesized in the same way except for the use of cobalt saccharinate and copper saccharinate as shown in the equation below:



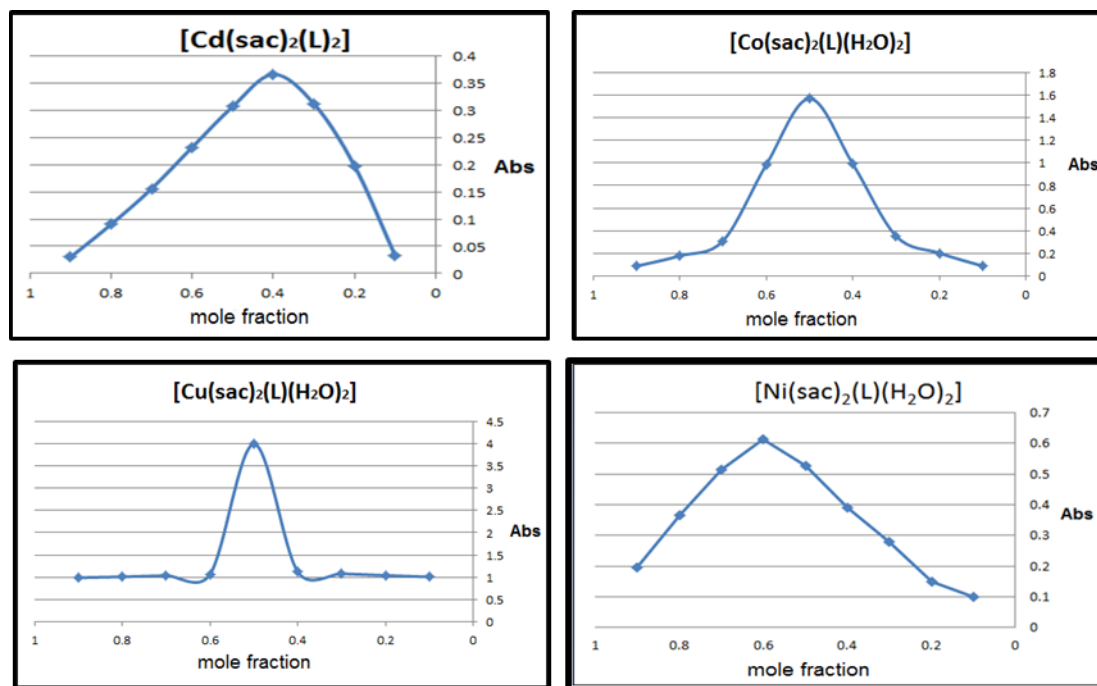
Study of complexes formation in solution by Jobs' method

The ratio of metallic ion to ligand was determined for the studied complexes using the continuous variation method.

Volumes of metal ion solution (0.5-4.5ml) with concentration of $(1 \times 10^{-4} \text{ mole} \cdot \text{L}^{-1})$ were replaced in (5ml) volumetric flask, volumes of ligand solution (0.5

We found ratio the metallic ion to ligand for complex studied by Job's method. We took the volumes ranged

(0.5-4.5 ml) from concentration $(1 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1})$ of metal ion solution and then we add volumes ranged (4.5-0.5 ml) of ligand solution $(1 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1})$, where it became the total volume of the solution (5ml) and determined absorbance of all solutions versus blank solution at the wave length the greatest for complex (λ_{max}). The [M:L] ratio was determined from the relationship between the absorption of the absorbed light and the mole ratio of [M:L].



Results and Discussion

1-Elemental micro analysis and metal content :

Elemental analysis (C.H.N.S) and metal content were found to be in agreement with calculated values .Table (1) show the values:

Table (1) Elemental micro analysis (%) , metal content ,compounds color and melting point for prepared compounds (calculated)

Seq	Compounds	C	H	N	S	M	Colors	melting point C ^o
L	C ₁₄ H ₁₀ N ₂ O ₂	70.49 (70.58)	4.46 (4.23)	11.70 (11.76)	-----	---	Orange	179-181
1	[Hg(sac) ₂ (L) ₂]	48.24 (48.44)	2.53 (2.71)	8.11 (8.07)	6.26 (6.16)	19.23 (19.26)	Orange	212-214
2	[Zn(sac) ₂ (L) ₂]	55.65 (55.67)	3.21 (3.11)	9.19 (9.27)	7.13 (7.08)	7.27 (7.21)	Orange	183-185
3	[Cd(sac) ₂ (L) ₂]	52.89 (52.92)	2.88 (2.96)	8.90 (8.82)	6.69 (6.73)	11.77 (11.79)	Orange	232-234
4	[Co(sac) ₂ (L)(H ₂ O) ₂]	48.30 (48.21)	3.21 (3.18)	8.13 (8.03)	9.17 (9.19)	8.43 (8.45)	Brown	209-211
5	[Cu(sac) ₂ (L)(H ₂ O) ₂]	47.93 (47.90)	3.09 (3.16)	8.01 (7.98)	9.03 (9.13)	9.03 (9.05)	Black	266-268
6	[Ni(sac) ₂ (L)(H ₂ O) ₂]	48.21 (48.23)	3.19 (3.18)	8.13 (8.03)	9.19 (9.20)	8.43 (8.42)	Black	139-140

2-Infrared Spectra

The prominent infrared spectral data of saccharin ,schiff base and its metal complexes given in the table (2). The IR spectrum of schiff base (Z)-3-((4-hydroxyphenyl)imino) indolin-2-one fig(1), showed a shoulder band at (3300cm⁻¹) for U(O-H) , band at (3270cm⁻¹) which assigned U(N-H) group ,another band at (3088cm⁻¹) was attributed to the aromatic U(C-H) group ,The spectrum of the ligand also reveal the presence of strong and sharp band at (1713cm⁻¹) which are assigned to U(C=O) group for isatin. In addition to that the absorption band of the isomethine group appeared at (1636cm⁻¹),[24] and this band indicates the formation of schiff base.

A comparison of the infrared spectrum of the free ligand and spectra of the prepared complexes fig (3,4)

exhibited the band of carbonyl group of saccharin in the region 1621-1663 cm⁻¹,showing shift to high wave numbers indicating that saccharin is coordinated from nitrogen to the metal ion [25],schiff carbonyl group show no change in zinc, mercury and cadmium complexes this mean it not coordinate with metal ion change in chemical shift and it shape in nickel and cobalt complexes indicated that the carbonyl grope coordinate with metal ion, copper complex shifted 25cm⁻¹ and showed smaller compared with its shape in the ligand indicating that its coordinated to the metal ion .In addition that, all metal complexes exhibited bands in the region 1582-1613cm⁻¹ which are assigned to U(C=N) group these bands are shifted to lower wave numbers, indicated that the azomethine nitrogen is coordinated to metal

ion[26]. The metal Co(II), Ni(II) and Cu(II) complexes showed broad band in the region 3241cm^{-1} to 3533cm^{-1} , which may be assigned to U(O-H) of coordinated water[27,28]. The new bands in the region of $423\text{--}483\text{ cm}^{-1}$ in the spectra of complexes

are assigned to stretching frequencies of (M-N) and bands in the range $529\text{--}538\text{ cm}^{-1}$ in the spectra of cobalt, nickel copper complexes are assigned to stretching frequencies of (M-O) bands respectively [29].

Table (2) Characteristic Stretching Vibrational Frequencies (cm^{-1}) of the ligand and its Metal Complexes

Seq	compounds	U(M-OH ₂)	U(O-H)	U(C-H)Ar	(C=O) _{sac}	C=O _{schiff}	U(C=N)	U(SO ₂) _{asy}	U(SO ₂) _{sy}	U(M-O)	U(M-N)
L	C ₁₄ H ₁₀ N ₂ O ₂	-----	3300	3046	-----	1713	1636	-----	-----	-----	-----
L	sacNa	-----	-----	3094	1649	-----	-----	1257	1150	-----	-----
1	[Hg(sac) ₂ (L) ₂]	-----	3265	3079	1659	1712	1606	1263	1155	-----	478
2	[Zn(sac) ₂ (L) ₂]	-----	3266	3077	1661	1713	1608	1269	1163	-----	480
3	[Cd(sac) ₂ (L) ₂]	-----	3266	3072	1663	1713	1613	1269	1155	-----	483
4	[Ni(sac) ₂ (L)(H ₂ O) ₂]	3241	3241	3091	1621	1709	1585	1284	1155	535	442
5	[Cu(sac) ₂ (L)(H ₂ O) ₂]	3533	3533	3091	1641	1738	1608	1266	1163	538	423
6	[Co(sac) ₂ (L)(H ₂ O) ₂]	3458	3338	3115	1621	1715	1582	1284	1158	529	481

3. Electronic Spectra, Magnetic Moment and Conductance Studies:

The UV spectrum of schiff base (L) showed intense bands at 33222 nm, 22573 nm, assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions respectively[31], table 3 gives data of the various electronic transition obtained for the complexes. The absorption spectrum of Co(II) complex showed one transition band at 15230 cm^{-1} , this transition may be assigned to ${}^2\text{Eg} \rightarrow {}^2\text{T}_{2g}$. The transition correspond to the octahedral geometry of the complex[32]. The magnetic moment of the complex 1.75 B.M indicating a low spin octahedral geometry[33]. The electronic spectrum of Ni(II) complex exhibited three bands at 12745, 19455 and 27786 cm^{-1} , which are attributed to ${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{2g(\text{F})}$, ${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{F})}$ and ${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{P})}$ transitions, respectively the transitions correspond to the octahedral geometry of the complex[34,35]. The

magnetic moment of the complex (2.97 B.M) indicating octahedral geometry[36]. The electronic spectrum of Cu(II) complex showed one band at 15723 cm^{-1} attributable to ${}^2\text{Eg} \rightarrow {}^2\text{T}_{2g}$ transition in the distorted octahedral geometry[37,38]. The magnetic moment value was 1.97 B.M corresponds with one unpaired electrons, table 3.

The UV-Vis spectra of Zn(II), Hg(II) and Cd(II) complexes showed no absorption bands at range 380-1000 nm and that indicates that these complexes have no d-d electronic transitions (d^{10}) system, but bands at 23809 for Hg(II) complex, 24213 for Zn(II) complex and 25380 cm^{-1} for Cd(II) complex were observed, indicated to the ligand bands which shifted to the lower wave number[39,40]. The lower value of molar conductivity in DMSO indicates to the nonelectrolytic nature of these metal complexes.

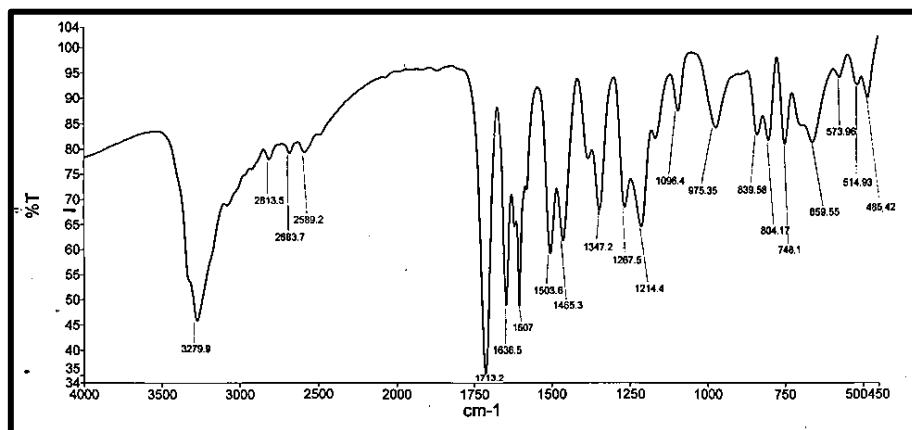
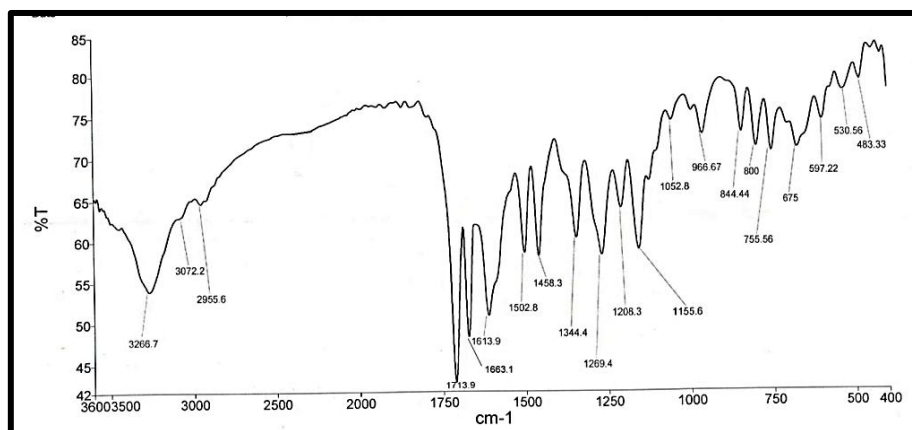
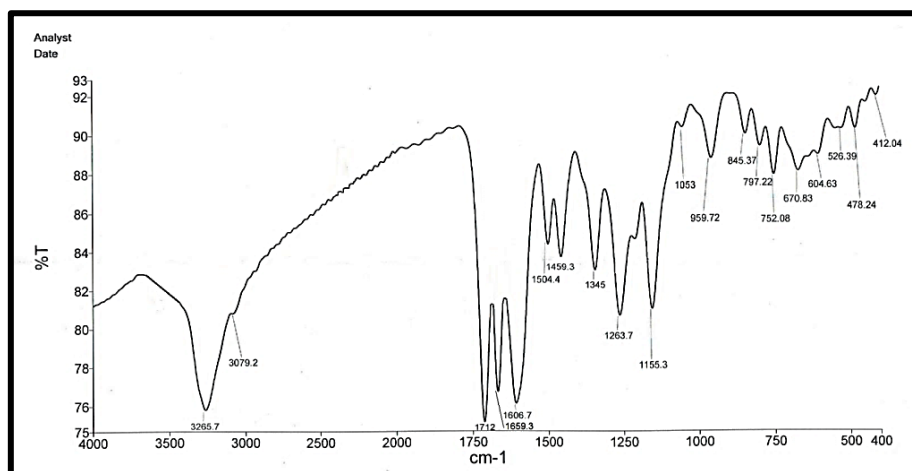
Table 3: Electronic transitions, Conductance (in DMSO) and Magnetic Moment (B.M) for (L) and Its Metal Complexes.

Compounds	Bands cm^{-1}	Assignment	Molar Cond. $\text{Ohm}^{-1} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$	μ_{eff} B.M
C ₁₄ H ₁₀ N ₂ O ₂ (L)	33222 22573	* $n \rightarrow \pi$ $\pi \rightarrow \pi^*$	-----	---
[Hg(sac) ₂ (L) ₂]	23809	L → M (C.T)	6.7	---
[Zn(sac) ₂ (L) ₂]	24213	L → M (C.T)	3.8	---
[Cd(sac) ₂ (L) ₂]	25380	L → M (C.T)	3.1	---
[Co(sac) ₂ (L)(H ₂ O) ₂]	15230	${}^2\text{Eg} \rightarrow {}^2\text{T}_{2g}$	6.3	1.75
[Ni(sac) ₂ (L)(H ₂ O) ₂]	12745 19455 27786	${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{2g(\text{F})}$ ${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{F})}$ ${}^3\text{A}_{2g(\text{F})} \rightarrow {}^3\text{T}_{1g(\text{P})}$	2.2	2.97
[Cu(sac) ₂ (L)(H ₂ O) ₂]	15723	${}^2\text{Eg} \rightarrow {}^2\text{T}_{2g}$	11.7	1.97

4-¹H-NMR spectra for Ligand and complexes

The H-NMR spectrum of the ligand (Z)-3-((4-hydroxyphenyl)imino) indolin-2-one fig(5) showed singlet at δ H(4.30ppm)[41], from the chemical shift and the integration this signal is assigned to (NH) proton, a multiple signal in the range δ H(6.40-7.44 ppm) from the chemical shift and the integration equivalent eight protons attributed to the aromatic ring. Also abroad single signal at δ H(10.84ppm) which is attributable to (OH) proton. The spectra of the complexes Zn(II) fig (6),Hg(II) fig (8) and Cd(II) fig (7) showed multiple signals in the range δ H(6.68-

7.61 ppm), δ H(6.69-7.81ppm) and δ H(6.83-7.59 ppm) respectively ,the integration showed that its equivalent twenty four protons ,which are attributed to aromatic ring protons (p-aminophenol, Isatin and saccharin)[42-44], these signals are overlap because its protons frequencies within convergent shifts. Single signals were observed at δ H (9.51)ppm, δ H (9.48-9.50)ppm and δ H (9.55) ppm in the spectra of the complexes Zn(II),Cd(II) and Hg(II) respectively, these signals are attributable to (NH) protons[43]. Single sign at δ H=(10.76-10.88 ppm) indicated to (OH)proton from the shift and integration.

Fig.(1) :FT-IR. Spectrum of (L) ligand (C₁₄H₁₀N₂O₂)Fig.(2) :FT-IR. Spectrum of [Cd(sac)₂(L)₂]Fig.(3) :FT-IR. Spectrum of [Hg(sac)₂(L)₂]

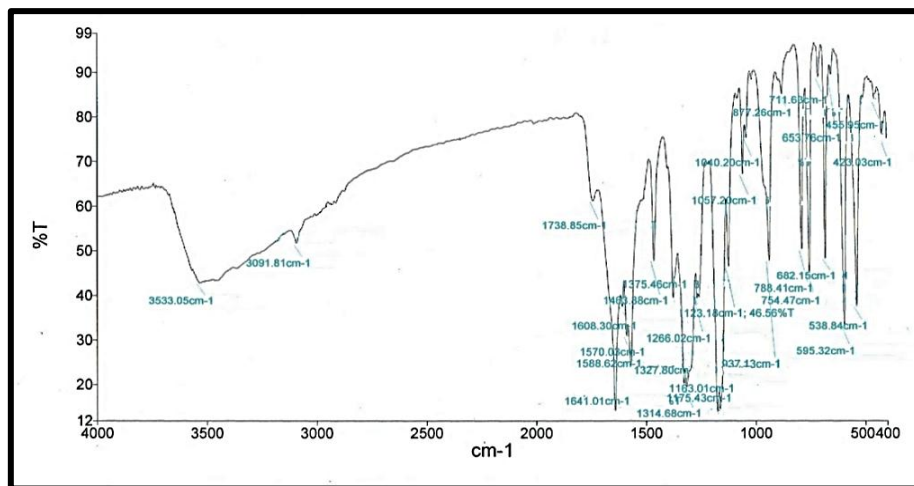


Fig.(4) :FT-IR. Spectrum of $[Cu(sac)_2(L)(H_2O)_2]$

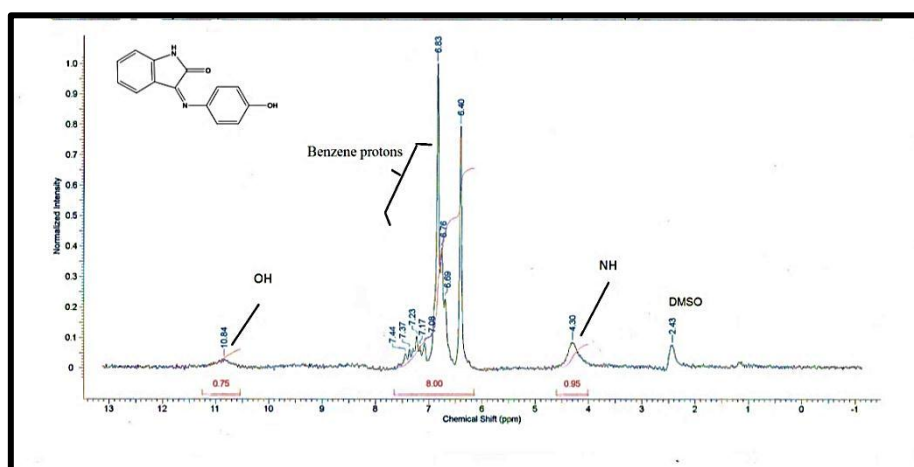


Fig.(5) : 1H nmr. Spectrum of (L) ligand ($C_{14}H_{10}N_2O_2$)

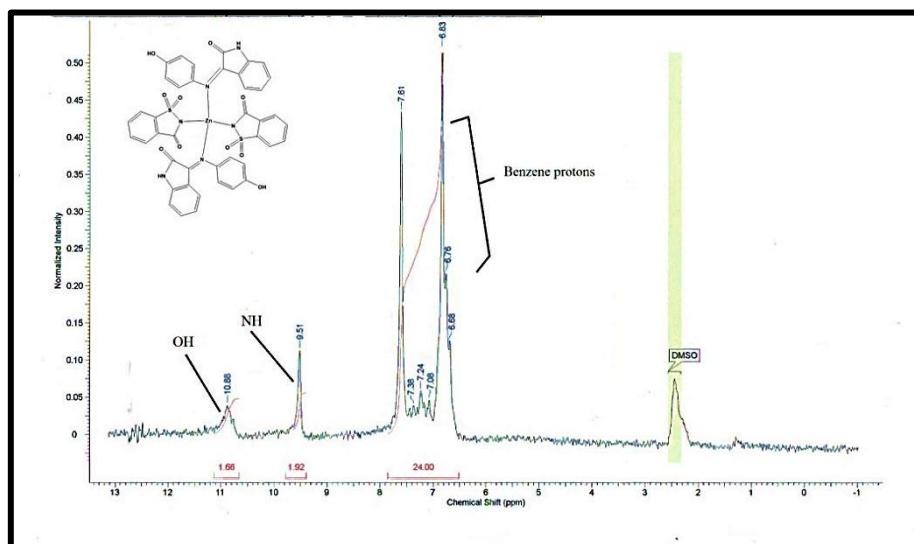


Fig.(6) : 1H nmr .Spectrum of $[Zn(sac)_2(L)_2]$

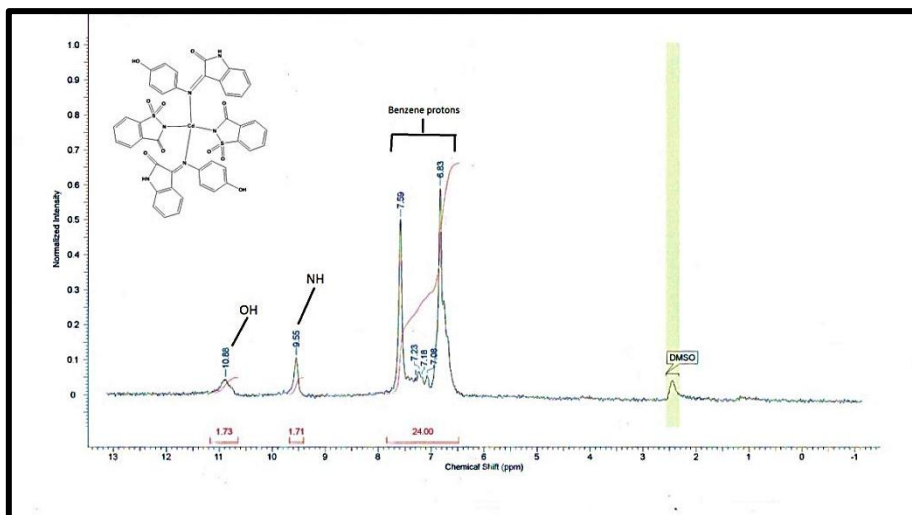


Fig.(7):¹Hnmr. Spectrum of [Cd(sac)₂(L)₂]

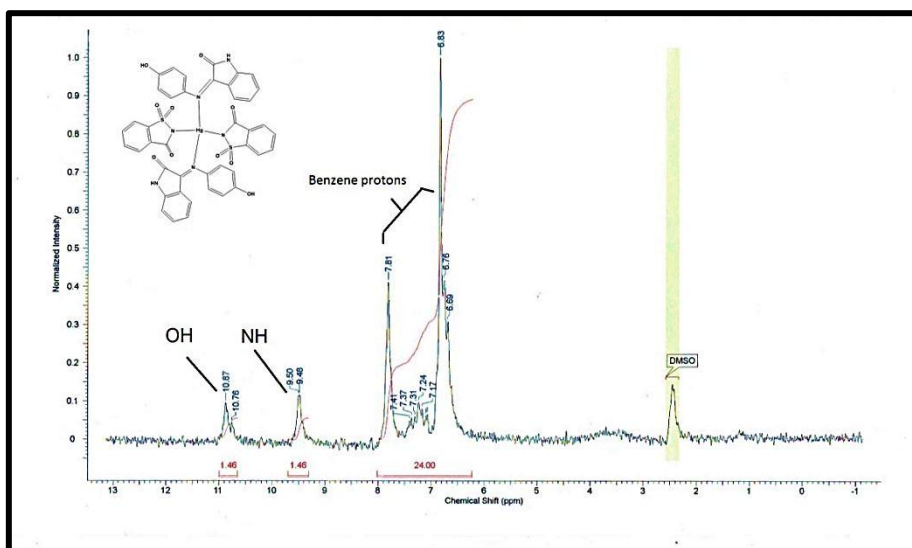
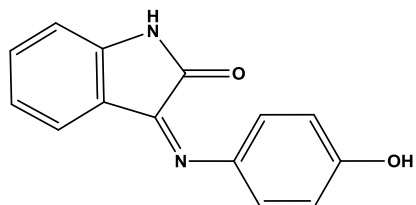


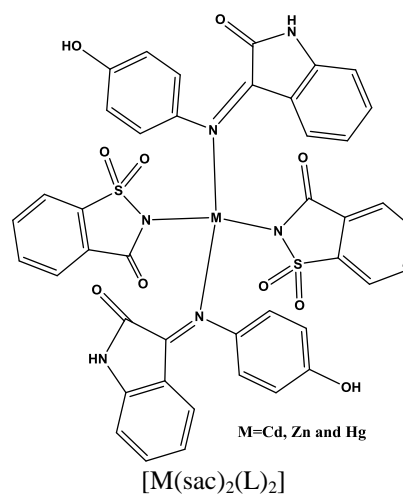
Fig.(8):¹Hnmr.Spectrum of [Hg(sac)₂(L)₂]

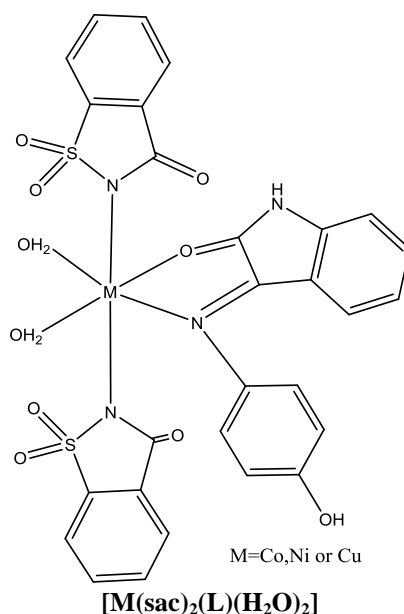
Suggested Structure for Schiff Base (L) and Its Metal Complexes

According to the results obtained from elemental and spectral analysis as well as magnetic susceptibility and conductivity measurements and ¹Hnmr spectroscopy, the suggested structures of the above mentioned compounds can be illustrated as follow :



(Z)-3-((4-hydroxyphenyl)imino)indolin-2-one





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تحضير وتشخيص عدد من معقدات العناصر (II) مع مزيج من ليكاندات السكارينيت وقاعدة شف المشتقة من الإيساتين

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الملخص

يتضمن البحث تحضير معقدات جديدة لبعض العناصر ثنائية التكافؤ مثل (Zn(II), Hg(II), Cd(II), Cu(II), Co(II), Ni(II)) مع مزيج من الليكاندات قاعدة شف المشتقة من الإيساتين والسكارين. شخضت المعقدات المحضرة بواسطة تقنية تحليل العناصر C.H.N.S، الموصلية المولارية، الأشعة فوق البنفسجية- المرئية، الأشعة تحت الحمراء، قياس الحساسية المغناطيسية، تقنية الامتصاص الذري اللهي للعناصر وطيف الرنين النووي المغناطيسي ¹Hnmr. حددت نسبة M:L لجميع المعقدات في الايثانول المطلق، واعتمادا على هذه القياسات اقترحت الصيغة التركيبية ثماني السطوح للمعقدات، $[M(sac)_2(L)_2]$ ، حيث Zn(II)=M و Hg(II) و Cd(II) و $[M(sac)_2(L)(H_2O)_2]$ ، حيث Co(II)=M و Cu(II) و Cu(II).

الكلمات المفتاحية: قاعدة شف، الإيساتين، السكارين، معقدات.