

Preparation and Characterization of tetra dentate Schiff- base ligand with some its complexes derived from thiosemicarbazone and β -[P-(2-pyrimidyl Sulhamyl) phenyl azo] Ketonilide (SDPA)

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Abstract

A new series of transition metal complexes of Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Hg(II) have been prepared and characterized using a new Schiff base (SDPAT) derived from a new azo dye. The β [- (2-Pyrimidyl Sulphamyl) phenylazo] ketoanilid and thiosemicarbazide were characterized by several techniques, including elemental analysis (C.H.N), molar conductance, magnetic measurements, electronic spectral, IR spectral studies and ^1H NMR. The data show that these complexes have a composition of $[\text{ML}]\text{X}_2$ type. The electronic spectral, and magnetic susceptibility data of the complexes suggest square planar geometry for all complexes, and the ligand (SDPAT) behaves in a bidentate and tetradentate manner, by coordination through the azomethane nitrogen atoms and sulfur groups of the thiosemicarbazide.

Key words: Schiff base (azo-azomethine Dye), metal chelate complexes, characterization

1. Introduction

Schiff base metal complexes have played a major role in the development of bioinorganic chemistry because many of these complexes provide biological models in understanding the structure of bimolecular and biological processes⁽¹⁻⁵⁾. So, they still have been the most well-known resources in the scope of heterogeneous catalysis, molecular electronics, single molecule based magnetism and, above all, photochemistry⁽⁶⁾. A large number of reports are available on the biochemistry and the microbial activities of transition metal complexes containing O,N^(7,8) and S, N⁽⁹⁻¹¹⁾ donor atoms. The transition metal complexes having sulfur and nitrogen donor Schiff base possess unusual configuration, structural lability and are sensitive to molecular environment⁽¹²⁾. In this paper, we report the preparation of a new type of tetradentate ligand formed by the condensation of β -[P-(2-Pyrimidyl sulphamyl) phenylazo] ketoanilide with thiosemicarbazide. This ligand system has both nitrogen and sulfur donor sites.

Its coordination with metal ion is through the enolisable sulfur group of the thiosemicarbazone moiety and the azomethine nitrogen atoms of the Schiff base.

2. Experimental

2.1. Materials and measurements

All chemicals used in the present work Sulphadiazine, acetoacetanilide, metal chlorides and solvents were supplied from BDH, Aldrich and used without further purification.

The melting point of ligand and its metal complexes were determined by Electro thermal melting point apparatus. Elemental analysis was performed by micro analytical unit of EA3000 A C.H.N Elemental analyzer, Al- Albat university Jordan. FTIR spectra were recorded using (KBr pellets) $4000\text{-}400\text{ cm}^{-1}$ on FTIR testscan Shimadzu 8000 series. Electronic spectra were recorded

in ethanol using Shimadzu uv-vis 1700 spectrophotometer. The ^1H NMR spectra were obtained in DMSO solution using (Bruker, Ultra Shield 3000 MKZ, Switzerland). Electrical conductivity was measured by Digital conductivity meter Alpha-800 with solute concentration (10^{-3}) in DMSO. The magnetic Susceptibility were measured on powder samples using Faraday method for this purpose, Balance magnetic (MSB-MKI) had been employed. The diamagnetic corrections were made by pascals constant⁽¹³⁾.

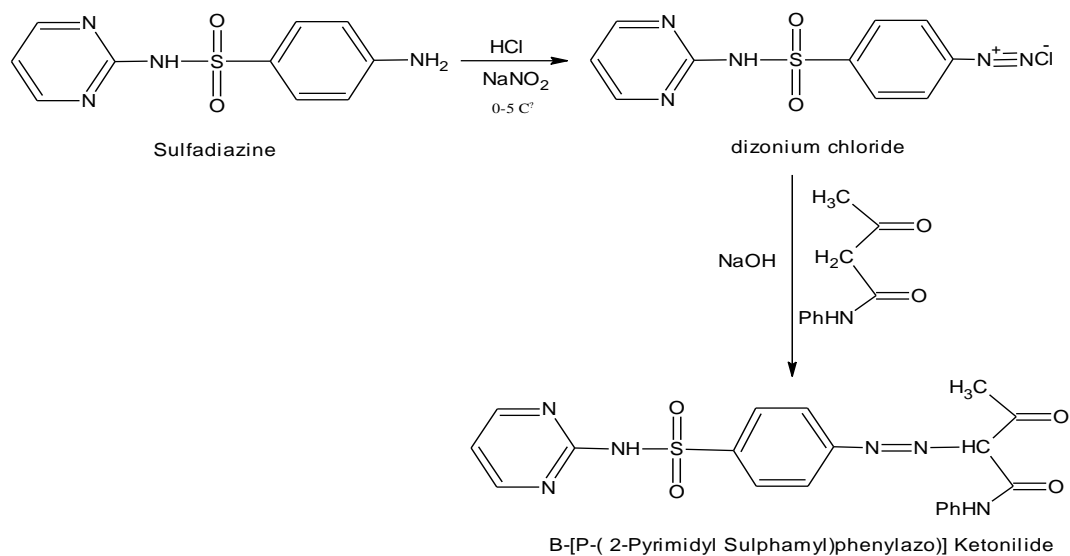
2.2. Preparation of Schiff base ligand (SDPAT)

The method of preparation was as follows:

a) Preparation of the new diazo dye.

The new diazo dye (SDPA) was prepared using the following:

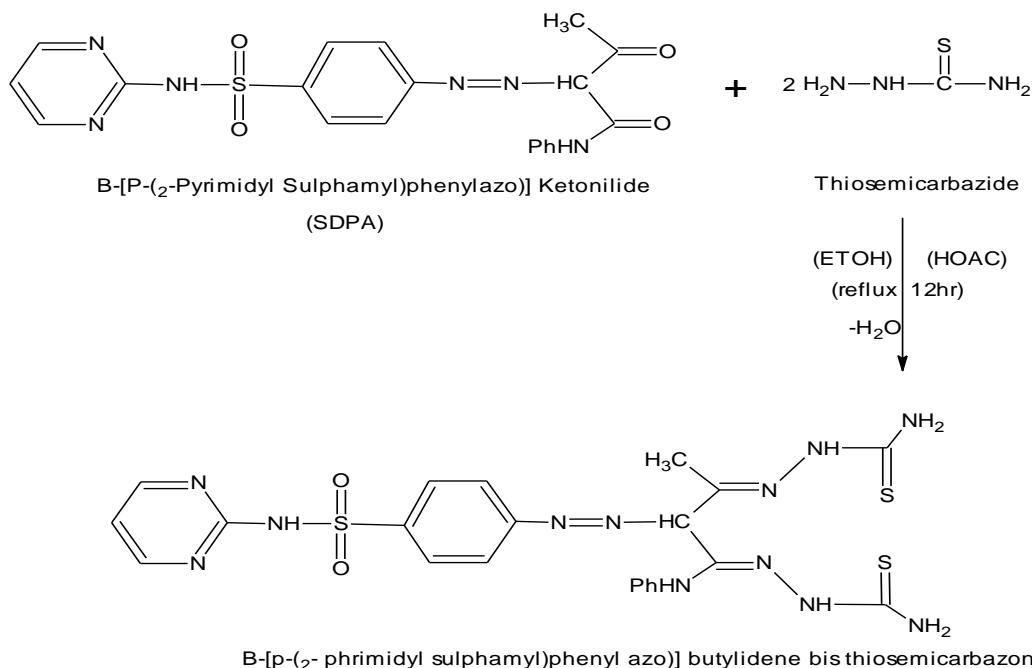
Diazoinium chloride solution was prepared by dissolving (2.56 g, 0.01 mol) of Sulphadiazine (SD) in 50 ml of water and 4 ml of concentrated HCl. The solution was treated with 5 ml of aqueous (1 M) sodium nitrite dropwise and stirred for 30 min at 0C° , acetoacetanilide (1.77 g, 0.01 mol) was dissolved in 200 ml of ethanol, and 80 ml of 10% sodium hydroxide was added the diazoinium chloride solution prepared above was then added dropwise for coupling. After the mixture had been stirred for (3h) at $0-5\text{C}^\circ$. The product was filtered, air dried and recrystallized twice from hot ethanol and then dried in the oven at 80C° for two hours, m.p (204-206 d). Scheme 1.



Scheme.1:Preparation of the azo dye (SDPA)

b) Preparation of the anew Schiff base ligand (SDPAT).

The new Schiff base ligand (SDPAT) was prepared by condensation of dye (SDPA) with thiosemicarbazide in equimolar (1:2) ratio, in absolute alcohol. Few drops of glacial acetic acid were added to the reaction mixture and refluxed for (12h) while refluxing the orange solution mixture charges into red in colored. The product was recrystallized from ethanol, and dried over anhydrous CaCl_2 . The reaction mixture gave single product, m.p ($165-167\text{C}^\circ$) Scheme 2.



Scheme.2:Preparation of the ligand (SDPAT)

2.3. Preparation Schiff base metal complexes

The Schiff base ligand (1mmol) dissolved ethanol absolute (25 ml) was mixed with salts of Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Hg(II) ions (1mmol) dissolved in ethanol (25 ml). The product was isolated after reduction of volume by evaporation. It was filtered off, washed with ethanol and dried under vacuum. The complexes thus obtained are listed in Table 1.

3. Results and discussion

The ligand is red crystal which is soluble in common organic solvents. The reaction of the ligand with metal ions mentioned above gives vary in color depending of metal ion. All complexes are quiet air-stable, insolable in water, but its soluble in most organic solvents some physical and chemical properties for ligand and its complexes are listed in table 1. The elemental analysis and metal contents data are show in table 1. In all cases (1:1) (metal:ligand) solid complexes are isolated, that is agreement with the stoichiometric ratio found using molar ratio methods. The higher conductivity values of the chelates complexes support the electrolytic nature of the metal complexes. Attempts suggest the structure of the isolated complexes come from full investigation using the following studied.

3.1. Microanalysis

The elemental analysis data the complexes as shown in table (1). it was found that the theoretical values are in a good agreement with the found data. The purity of the Schiff base ligand were tested by TLC technique and C, H, N elemental analyses.

3.2. Infrared spectra

The important infrared bands of free ligand and complexes are summarized in table (2). The weak band in the spectrum of free ligand observed at $(3268)\text{cm}^{-1}$ due to $\nu(\text{NH}_2)$ remained unaffected after complexation^(14,15). The spectrum of the free ligand and exhibited abroad band at $(3369)\text{cm}^{-1}$ assignable to the $\nu(\text{NH})$ group^(16,17). No change was observed for this band in the

spectra of complexes. The ligand band at (1647, 1620) cm^{-1} due to $\nu(\text{C}=\text{N})$ of imine nitrogen shifted to lower wave numbers on complexation suggests involvement of unsaturated nitrogen atoms of azomethine group in bonding with the metal ion other band observed disappearance in some the complexes^(18,19).

The $\nu(\text{C}=\text{S})$ stretching vibration appears at (1001) cm^{-1} in the spectrum of free ligand, was moved to a lower frequency after complexation, which means that the shifts are due to coordination of ligand to metal ions by the thione sulfur group^(20,21). The $\nu(\text{N}=\text{N})$ stretching vibration appears at (1436) cm^{-1} in the free ligand spectra⁽²²⁾, this band remains in the same region in the complexes. The spectrum of free ligand show two absorption bands at (1317) and (1163) cm^{-1} due to symmetrical and asymmetrical vibration of $\nu(\text{O}=\text{S}=\text{O})$ group^(23,24), These bands are stable in position and intensity in free ligand and its metal complexes. The spectra of chelate complexes showed new weak bands in the region (529- 430) cm^{-1} , these bands did not present in the spectrum of ligand may be attributed to vibration $\nu(\text{M}-\text{N})$ and (M-S) provide evidences concerning the bonding of nitrogen and sulfur the metal ions⁽²⁵⁻²⁶⁾. Representative example for their spectra is given in Fig.1.

3.3. Magnetic measurement and electronic spectra

The magnetic moments and electronic spectral bands of the complexes are summarized in table (3). The electronic spectrum of Co(II) complex shows two absorption bands at (32258 cm^{-1}) and (23584 cm^{-1}), there are assigned to ILCT and $^1\text{A}_{1g} \rightarrow ^1\text{B}_{1g}$ transition respectively, which are characteristic of square- planar stereo chemistry⁽²⁷⁾. The magnetic moment value of the cobalt (II) complex (3.41 B.M) which may suggesting square- planar structure⁽²⁸⁾.

The electronic spectrum of Ni(II) complex exhibit bands (25316 cm^{-1}) and (23148 cm^{-1}) assignment to $^1\text{A}_{1g} \rightarrow ^1\text{A}_{2g}$, and $^1\text{A}_{1g} \rightarrow ^1\text{B}_{1g}$ transition respectively, the observed zero magnetic moment confirms the square- planar complexes for the Ni(II) complex in conformity with the fact that all known square- planar complexes of nickel(II) are diamagnetic^(29,30). The magnetic moment value of the copper(II) (1.74 B.M) which may suggest an square –planar structure^(30,31). The electronic absorption band to the copper(II) shows abond central at (16556 cm^{-1}) which may assigned to $^2\text{B}_{1g} \rightarrow ^2\text{A}_{1g}$ transition in square- planar environment⁽³²⁾. No transition were observed in the visible region for Zn(II), Cd(II) and Hg(II) complexes consistent with d^{10} configuration of the Zn(II), Cd(II) and Hg(II) ions and the electronic spectra of this complexes do not show any d-d transition band, its diamagnetic moment⁽³¹⁻³³⁾. According to these results the following structure formal of these chelate complexes may be proposed in Fig (2).

3.4. $^1\text{HNMR}$ Spectrum Studies

The $^1\text{HNMR}$ spectra of the ligand was recorded in DMSO- d_6 . The $^1\text{HNMR}$ spectrum of the ligand shows the following signals: phenyl multiples at (6.8-7.8) δ rang, -C-NH at (4.0) δ , =N-CH at (1.5) δ , NH_2 at (8.5) δ , and CH of primidine at (6.9-8.4) δ rang as show in Fig(3).

3.5. Conductivity measurement

All chelate complexes prepared in this work showed conductivity values ranged between (72-87) S. $\text{mol}^{-1}.\text{cm}^2$ in DMSO at room temperature these values indicating that high conductivity of the complexes^(27,34).

4. Conclusion

This paper report including flowing:

- 1- Preparation and identification of new tetra dentate schiff base and its complexes with Co(II), Ni(II), Cu(II), Zn(II), Cd(II),and Hg(II) metal ions.
- 2- The isolated products were characterized by available techniques.
- 3- All the complexes are stable and nature and the geometry is proposed for complexes show square planar stereo chemistry.

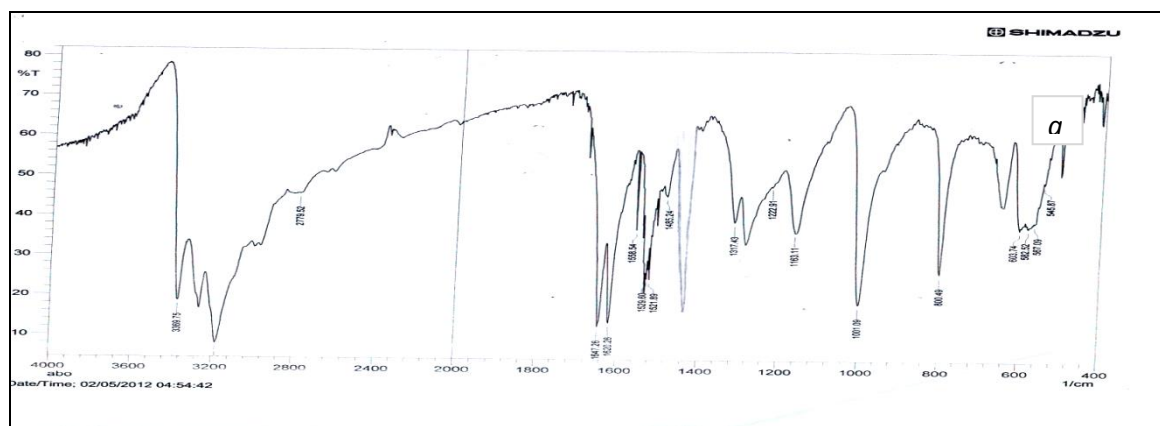
According to these results the structural formulas of these complexes may be proposed in Fig.4.

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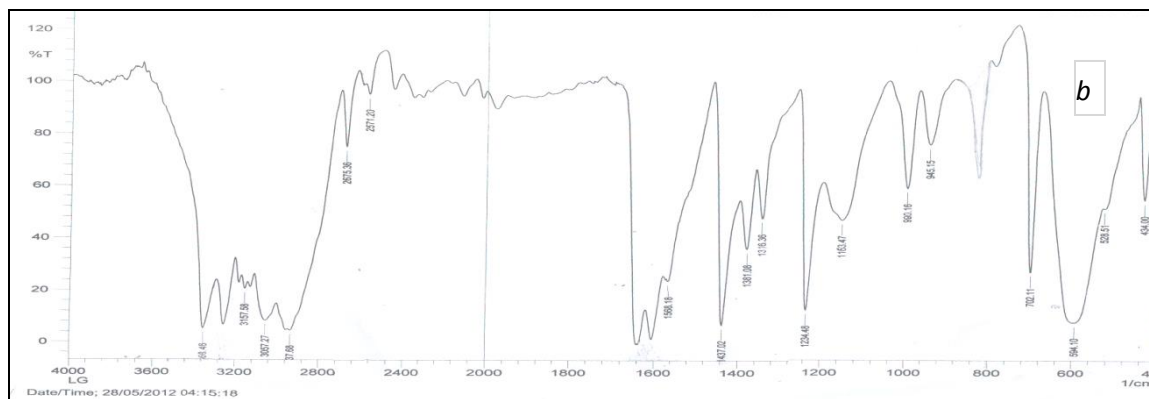


Fig.(1): IR spectra of (a) the ligand (SDPAT) & (b) [CoL]Cl₂

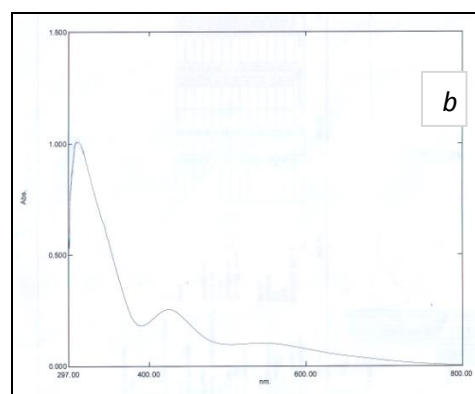
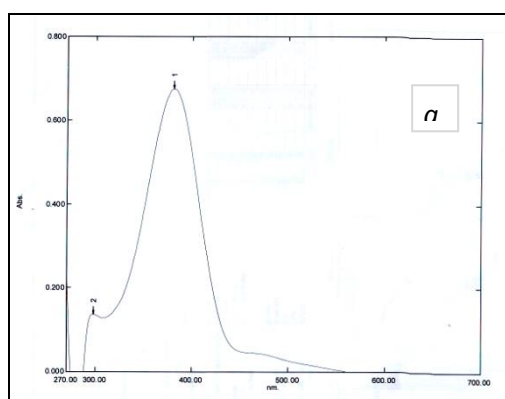


Fig.(2). Electronic spectrum of : (a) the ligand (SDPAT) & (b) [CoL]Cl₂

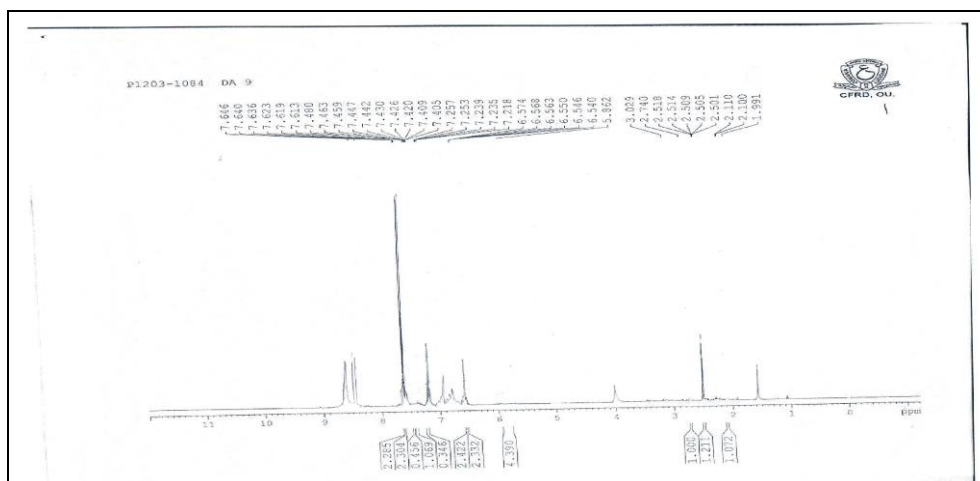
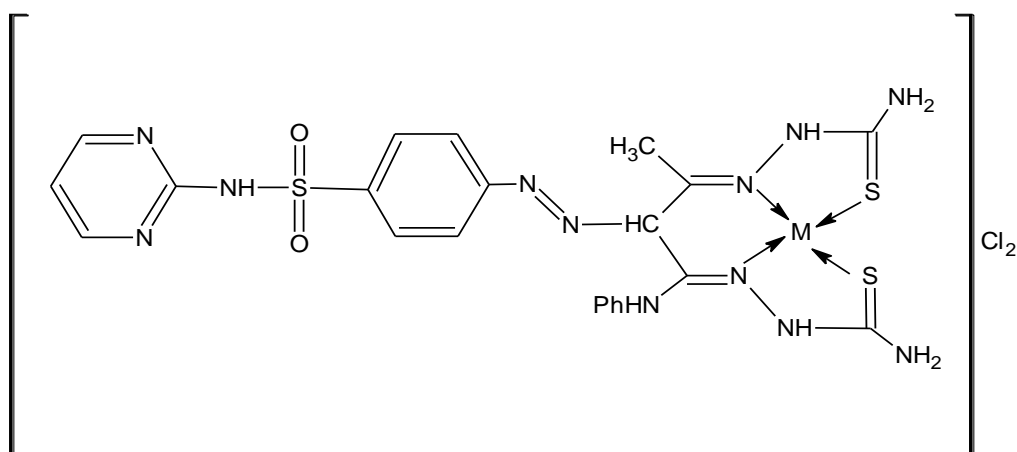


Fig.(3): ^1H NMR spectrum of the ligand (SDPAT)



M= Co(II), Ni(II), Cu(II), Zn(II), Cd(II),and Hg(II)

Fig.4: The proposed structural formula of prepared chelate complexes.

Table.1: Physical data and analysis of ligand and its complexes.

No.	Compound	Colour	M.P °C	Yield %	Formula	Found,(Calc.)%			
						C	H	N	M
1	L= Ligand	Red	(165-167 °C)	75	[C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃]	45.0 (45.19)	4.17 (4.14)	28.50 (28.75)	---
2	[CoL]Cl ₂	Dark Brown	(197-198 °C)	67	[Co(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃)]Cl ₂	36.66 (36.97)	3.30 (3.36)	23.48 (23.53)	7.98 (8.25)
3	[NiL]Cl ₂	Pale green	250 d	78	[Ni(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃) Cl ₂	36.13 (36.99)	3.25 (3.36)	23.15 (23.53)	7.89 (8.22)
4	[CuL]Cl ₂	Green	242 d	82	[Cu(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃)]Cl ₂	35.88 (36.74)	3.24 (3.34)	23.40 (23.38)	8.15 (8.84)
5	[ZnL]Cl ₂	Colour less	(260-262 °C)	76	n(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃)]C l ₂	36.00 (36.64)	3.27 (3.33)	23.45 (23.32)	9.18 (9.07)
6	[CdL]Cl ₂	Colour less	(235-236 °C)	68	[Cd(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃)]Cl ₂	36.87 (34.40)	2.97 (2.86)	22.45 (21.89)	15.78 (14.64)
7	[HgL]Cl ₂	Colour less	(270-272 °C)	72	[Hg(C ₂₂ H ₂₄ N ₁₂ O ₂ S ₃)]Cl ₂	31.76 (30.85)	2.93 (2.80)	20.98 (19.63)	23.13 (23.44)

L =ligand, d =Complex metal with decomposition

Table.2 : Electronic spectra and magnetic moment data of the complexes.

<i>Conductivity</i> <i>S.mol⁻¹. cm²</i>	<i>μ_{eff}(B.M)</i>	Transition	λ_{max} nm (cm⁻¹)	Complexes
---	---	ILCT π→π* ILCT n→π*	297 (33670) 380 (26315)	Ligand
72	3.41	ILCT ¹ A _{1g} → ¹ B _{1g}	310 (32258) 424 (23584)	[CoL]Cl ₂
85	Dia	¹ A _{1g} → ¹ A _{2g} ¹ A _{1g} → ¹ B _{1g}	395 (25131) 432 (23148)	[[NiL]Cl ₂
78	1.74	² B _{1g} → ² A _{1g}	604 (16556)	[CuL] Cl ₂
82	Dia	ILC T	330 (30303)	[ZnL] Cl ₂
87	Dia	IL C T	343 (29154)	[CdL] Cl ₂
74	Dia	IL C T	347 (28818)	[HgL] Cl ₂

Tabale.3: IR spectra frequencies for the ligand and its metal complexes in cm⁻¹

<i>v(M-S)</i>	<i>v(M-N)</i>	<i>v(O=S=O)</i>	<i>v(N=N)</i>	<i>v(NH-C=S)</i>	<i>v(C=S)</i>	<i>v(C=N)</i>	<i>v(NH)</i>	<i>v(NH₂)</i>	<i>Compound</i>
---	---	1317 1163	1436	800	1001	1647 1620	3369	3302	L=(SDAA T)
434	528	1316 1163	1437	802	990	1639 1600	3365	3305	[CoL]Cl ₂
430	524	1318 1162	1435	800	991	1620 1610	3366	3303	[NiL]Cl ₂
468	528	1320 1160	1436	804	989	1631 1600	3368	3302	[CuL]Cl ₂
432	524	1318 1162	1438	802	990	1620 1610	3366	3304	[ZnL]Cl ₂
440	525	1317 1161	1435	803	990	1618 1611	3369	3305	[CdL]Cl ₂
445	529	1315 1162	1435	800	991	1630 1600	3367	3302	[HgL]Cl ₂

تحضير وتشخيص ليكاند قاعدة شف رباعية المخلب مع بعض معقداته مشتقة من
ثايوسيميكاربازون

و B-[P-(2-pyrimidyl Sulhamyl) phenyl azo)] Ketonilide(SDPA)

الخلاصة

تضمن البحث تحضير وتشخيص بعض معقدات الايونات الانتقالية Co(II), Ni(II), Cu(II), Zn(II), Cd(II), and Hg(II), مع ليكاند قاعدة شف الجديدة المشتق من صبغة الازو β [- (2-Pyrimidyl Sulphamyl) phenylazo] ketoanilid مع الثايوسيميكاربازون. وقد شخّصت المعقدات من قياسات التحليل الدقيق للعناصر والعزوم المغناطيسية والتوصيلية المولارية واطياف الاشعة فوق البنفسجية واطياف الاشعة تحت الحمراء وبروتون الرنين النووي المغناطيسي. كما بينت الدراسة ان الصيغ التركيبية للمعقدات هي من نوع $[ML]X_2$. من نتائج طيف الاشعة فوق البنفسجية والحساسية المغناطيسية اقترح الشكل المربع المستوي لجميع المعقدات الصلبة. كما تبين ان الليكاند يسلك كليكاند غير مشحون رباعي المخلب يرتبط مع الايونات الفلزية من خلال ذرتي النيتروجين للازوميثين وذرتي الكبريت لثايوسيمكاربازون.