

****Synthesis and Characterization of 2-(5-mercapto-1,3,4-thiadiazol-2-yl)isoindoline-1,3-dione (L) and It's Complexes with Some Transition Metals (Co(II) , Ni(II) , Cu(II) , Zn(II) and Cd(II))***

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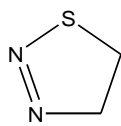
** College Of Education - Al-Qadisiya University

Haider.aj1976@gmail.com**Abstract**

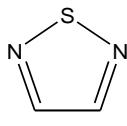
In this work ,The ligand 2-(5-mercapto-1,3,4-thiadiazol-2-yl)isoindoline-1,3-dione (L) has been synthesized by the reaction of 5-amino -1,3,4-thiadiazole -2-thiol with phthalic anhydride by using fusion method . The metal complexes of the ligand were prepared with metals chlorides of Co(II) , Ni(II) , Cu(II) , Zn(II) and Cd(II) in ethanol as solvent . The ligand (L) and its metal complexes have been characterized by elemental analysis (CHN), IR, ¹H-NMR and UV- Vis. spectra , magnetic susceptibility measurements , molar conductivity , melting points and atomic absorption . The metal-ligand ratio was determined by mole ratio method .The suggested structures for the Co(II) , Ni(II) and Cu(II) complexes are octahedral geometry and for the Zn(II) and Cd(II) complex are tetrahedral geometry .

Keyword : 1,3,4-thiadiazol , complexes of 1,3,4-thiadiazol**Introduction**

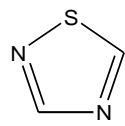
Thiadiazoles are five membered heterocyclic compound , contain two nitrogen atoms and one sulfur atom as hetero atoms. ^(1,2) They are four isomeric forms : 1,2,3-thiadiazole (I); 1,3,4-thiadiazole (II); 1,2,4-thiadiazole (III) and 1,2,5-thiadiazole (IV) ^(3,4).



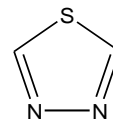
1,2,3-Thiadiazole



1,2,5-Thiadiazole



1,2,4-Thiadiazole



1,3,4-Thiadiazole

The studies of the 1,3,4-Thiadiazole isomer are more than all the other isomers combined⁽⁵⁾. Several derivatives of 1,3,4-thiadiazoles have been found biologically active such as anti-inflammatory anti-cancer , antibacterial , anti-fungal , antiviral , anti-convulsant ⁽⁶⁻⁹⁾, carbonic anhydrase inhibitor ⁽¹⁰⁾. These biologically active of 1,3,4-thiadiazole due to the presence of (=N-C-S) moiety ⁽¹¹⁾. One of applications of the 1,3,4-thiadiazole in coordination chemistry used as ligand to prepare different complexes , most of these complexes has pharmacological activity ^(12,13). Phthalimides are organic compounds as starting materials and intermediates for

***The Research is apart of on Ph.D. dissertation in the case of the third researcher**

the synthesis of many types of alkaloids .Some derivatives of Phthalimides were used in industrial fields such as drugs synthesis because they have broad spectrum of biological applications such as anti-bacterial agents , anti-inflammatory, anti-fungal agents and plant growth ⁽¹⁴⁻¹⁶⁾.

Experimental

Materials And Methods

All chemicals were Supplied from Al-Drich , Fluka and BDH The UV- Vis. spectra have been recorded in the range of (200-1000) nm using (ShimadzuU.V-165PCS spectrophotometer).Infrared spectra have been recorded in the range (400-4000 cm^{-1}) using KBr disk for the ligand and its complexes using (Shimadzu FT-IR 8400S spectrophotometer).

The $^1\text{H-NMR}$ spectra was recorded on Bruker model Ultrashild 300 MHz NMR at Al-Albyt University of Jordon. DMSO- d^6 was used as solvent and TMS as internal reference . Elemental analysis were recorded on instrument type EA-99.mth . The metal content was determined by using Shimadzu corporation model AA-6300 atomic absorption . Molar conductivity measurements were obtained by using electrolytic conductivity measuring set model Cond 3110 SET1 in DMF solvent in concentration (10^{-3}M) at 25°C . Magnetic susceptibility measurements were obtained at room temperature applying method using Balance Magnetic Susceptibility Model MSB-MKI.

Synthesis of The Ligand 2-(5-Mercapto-1,3,4-Thiadiazol-2-yl)Isoindoline-1,3-dione (L)

Compound 5-Amino-2-thio-1,3,4-thiadiazole (0.02mole , 2.66 gm) was mixed with phthalic anhydride (0.02mole , 2.96 gm) in open pyrex tube , heated over oil bath at ($170-180^\circ\text{C}$) , fusion was completed after (1hr) , the mixture was cooled and the precipitate was recrystallized from ethanol and afforded ligand (L) as dark yellow crystals (yield 84% , m.p : $240-242^\circ\text{C}$) , (Scheme 1) ⁽¹⁷⁾.

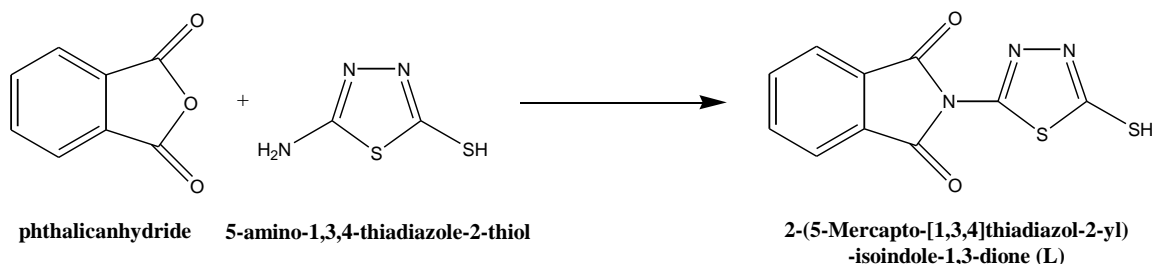
Synthesis of the Co(II) , Ni(II) and Cu(II) Complexes

The ligand (L) (0.002mol , 0.53 gm) was dissolved in 20 ml ethanol in a 100 ml round-bottom flask. A solution of (0.001mol , 0.24gm , 0.24gm , 0.17gm) of the hydrated metal chloride Co(II) , Ni(II) and Cu(II), respectively in 20 ml ethanol was added dropwise , with continuous stirring at room temperature. Reflux for 2hr. , with added a few drops of ammonia (10%) to form precipitate except complex of Cu(II) . The resulting precipitates were filtered off, washed with ethanol , dried and recrystallized from ethanol .

Synthesis of Zn (II) and Cd(II) Complexes

The ligand (L) (0.001mol , 0.26gm) was dissolved in 20 ml ethanol in a 100 ml round-bottom flask. A solution of (0.001mol , 0.22gm , 0.27gm) of the hydrated metal chloride, Zn(II) and Cd(II) respectively in 20 ml ethanol was added dropwise with continuous stirring at room temperature. Reflux for 2hr. , the resulting

precipitates were filtered off, washed with ethanol, dried and recrystallized from ethanol and dried. Preparation of Zn(II) complex was required addition a few drops of ammonia (10%) to form precipitate.



Scheme (1). Synthesis of the ligand 2-(5-mercapto-1,3,4-thiadiazol-2-yl)isoindoline-1,3-dione (L)

Results and Discussion

A new ligand 2-(5-mercapto-1,3,4-thiadiazol-2-yl)isoindoline-1,3-dione (L) was synthesized from the reaction 5-amino-1,3,4-thiadiazole-2-thiol with phthalic anhydride by using fusion method. The metal complexes of the ligand were prepared with metals chlorides of Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) in ethanol as solvent. The ligand and its complexes are soluble in polar organic solvents, such as DMSO and DMF. The physical properties and elemental analysis are shown in Table(1). The molar ratio method was followed to determine the metal to ligand ratio. This method showed that the metal to ligand ratio is (1:2) in case Co(II), Ni(II) and Cu(II) complexes and (1:1) in case Zn(II) and Cd(II) complexes.

The Infra-red Spectra

The spectrum of the free ligand (L), Figure (2), showed two bands at (1782, 1735) cm^{-1} assigned for $\nu(\text{C}=\text{O})$ of the carbonyl group. The bands at (3193), (1604) and (2661) cm^{-1} were assigned to $\nu(\text{N}-\text{H})$, $\nu(\text{C}=\text{N})$ and $\nu(\text{S}-\text{H})$ stretches respectively. Also the spectrum shows bands at (1357), (1226), (1064), (1542, 1465, 1434) and (3031) cm^{-1} attributed to the $\nu(\text{C}=\text{S})$, $\nu(\text{C}-\text{N})$, $\nu(\text{N}-\text{N})$, $\nu(\text{C}=\text{C}_{\text{aromatic}})$ and $\nu(\text{C}-\text{H}_{\text{aromatic}})$ stretches respectively⁽¹⁸⁻²¹⁾.

The carbonyl group in the free ligand was shifted to lower wave number after complexation in the range (1689-1720) cm^{-1} for Co(II), Ni(II) and Cu(II) complexes respectively^(22,23), while in complexes of Zn(II) and Cd(II), the carbonyl group was splitted and shifted into two bands in the range (1650-1789) cm^{-1} . The wave number of azomethine group of thiadiazole ring was shifted to lower and higher wave number after complexation in complexes of Zn(II) and Cd(II). This band exhibited at the range (1596-1643) cm^{-1} for these complexes^(24,25). The broad bands observed for complexes Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) in the region (3317-3417) cm^{-1} are due to the $\nu(\text{O}-\text{H})$ stretching of water molecules and ethanol molecules^(26,27).

The new bands at (571, 590, 552, 550, 591) cm^{-1} are due to the $\nu(\text{M}-\text{O})$ for Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) complexes respectively and bands at (493, 462) are due to $\nu(\text{M}-\text{N})$ for Zn(II) and Cd(II) complexes respectively^(28,29).

The shifted of the wave number of carbonyl and azomethine groups of free ligand after formation complexes and the appearance a new bands in the complexes spectral are evidence on coordination between the ligand and metals ions.

¹H-NMR spectra of The Ligand(L)

The ¹H-NMR spectrum of the ligand , Figure (1) , this spectrum was showed many signals at 6.7-6.9ppm due to aromatic protons . The proton of thiol group exhibited signal at 11.12ppm , respectively ^(25,30).

Molar Conductivity Measurements

The molar conductance values of the synthesized complexes were measured in DMF solvent and concentration 10⁻³M . The spectral data of molar conductance are shown in table (3) and in the range (10.1-86.2ohm⁻¹ . cm² . mole⁻¹).These results inducted to the complexes of Co(II) and Ni(II)are ionic in (1:1) ratio, while the complexes of Cu (II) , Zn(II) and Cd(II) are non - ionic ^(31,32).

3.4 - Electronic Spectra and Magnetic Susceptibility Measurements

The electronic spectra data and magnetic moments of the ligand and its complexes have been shown in table (3) .

The absorption spectra for of L in absolute ethanol , figure(8) gives three band at 203, 227 which these peaks due to π - π^* transition and 322 nm which can be attributed to and n- π^* transitions ^(16,19).

The electronic spectra of Co(II) complex , Figure(9) showed four peaks at (351, 701, 752 and 771nm) , the first peak attributed to charge transfer and last three peaks attributed to d-d transitions , $^4T_{1g} \rightarrow ^4T_{1g}$ (P) , $^4T_{1g} \rightarrow ^4A_{2g}$ (F) and $^4T_{1g} \rightarrow ^4T_{2g}$ (F) respectively. The magnetic moment of Co(II) complex is (5.02B.M) , these measurements suggested octahedral geometry around Co(II) ion ⁽³³⁾.

The electronic spectra of Ni(II) complex , Figure(10) showed peaks at (391 , 607 and 672 nm) , attributed to $^3A_{2g} \rightarrow ^3T_{1g}$ (P) , $^3A_{2g} \rightarrow ^3T_{1g}$ (F) and $^3A_{2g} \rightarrow ^3T_{2g}$ (F) respectively. The magnetic moment of Ni(II) complex is(3.13B.M). These measurements suggested octahedral geometry around Ni(II) ion ⁽³⁴⁾.

The electronic spectra of Cu(II) complex , Figure(11) exhibited two peaks at (361and 731nm) , the first peak may be due to charge transfer . The other peak may be assigned to $^2E_g \rightarrow ^2T_{2g}$ transition . The magnetic moment of Cu(II) complex was found (1.2 B.M). The electronic spectra and magnetic moment of Cu(II) complex are inductive to octahedral geometry around Cu(II) ion ⁽³⁵⁾.

The complexes of Zn(II) and Cd(II) , Figure(12) and (13) have been found diamagnetic . The molar ratio , molar conductance , magnetic measurements , elemental analysis and atomic absorption which suggest tetrahedral geometry^(22,36).

Conclusion

Corresponding to the results , the ligand (L) behaves as bidentate ligand and coordinates with metals ions through carbonyl oxygen atom in the complexes of Co(II) , Ni(II) and Cu(II) and the azomethine nitrogen in the complexes of Zn(II) and Cd(II). Basis on the elemental analysis , Infrared and electronic spectra , magnetic susceptibility measurements , flame atomic absorption spectroscopy and molar conductivity which suggest octahedral for Co(II) , Ni(II) and Cu(II) complexes and tetrahedral geometry to Zn(II) and Cd(II) complexes (Figure 14) .

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

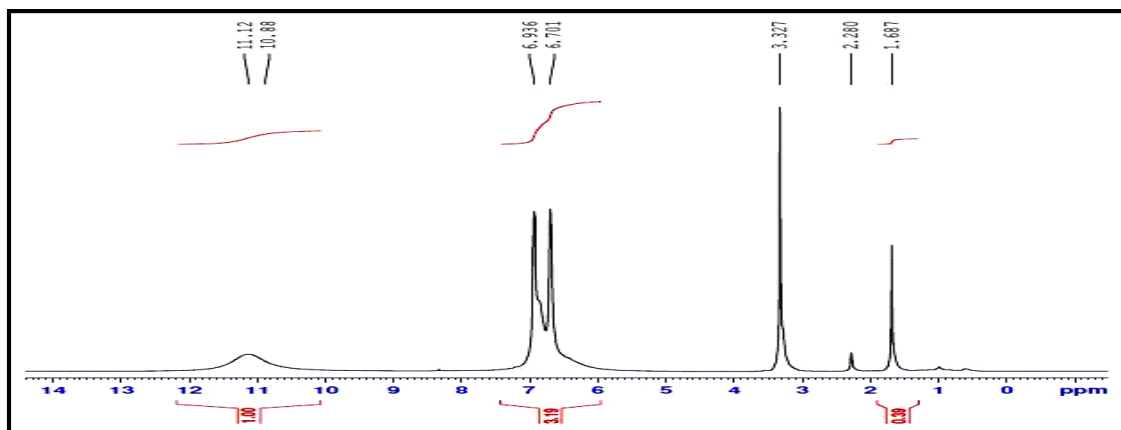
No	Compounds	Color	M.P (C°)	Yield %	Molecular Formula	Found (Calc.)%			
						C	H	N	M
1	L	Dark- Yellow	240- 242	84	C ₁₀ H ₅ N ₃ O ₂ S ₂	(45.62)	(1.91)	(15.96)	-
2	[Co(L) ₂ (H ₂ O)Cl]Cl	Dark Green	282- 284	64	C ₂₀ H ₁₂ N ₆ O ₅ S ₄ Cl ₂ Co	36.87 35.62 ()	2.09 (1.79)	13.78 (12.46)	7.79 (8.74)
3	[Ni(L) ₂ (H ₂ O)Cl]Cl	Green	258- 260	74	C ₂₀ H ₁₂ N ₆ O ₅ S ₄ Cl ₂ Ni	37.32 (35.61)	1.83 (1.29)	13.43 (12.47)	8.18 (8.71)
4	[Cu(L) ₂ Cl ₂]. C ₂ H ₅ OH	Pale green	275- 277	76	C ₂₂ H ₁₆ N ₆ O ₅ S ₄ Cl ₂ Cu	37.97 (37.37)	1.76 (2.28)	13.42 (11.88)	7.78 (8.99)
5	[Zn(L)Cl ₂]. H ₂ O	Pale Yellow	253- 255	81	C ₁₀ H ₇ N ₃ O ₃ S ₂ Cl ₂ Zn	- (28.76)	- (1.69)	- (10.06)	15.49 15.66
6	[Cd(L)Cl ₂].H ₂ O	White	298- 300	82	C ₁₀ H ₇ N ₃ O ₃ S ₂ Cl ₂ Cd	26.72 (25.85)	1.86 (1.52)	11.67 (9.04)	23.41 24.19

Table (2): The important infrared spectral bands for the synthesized complexes and ligand

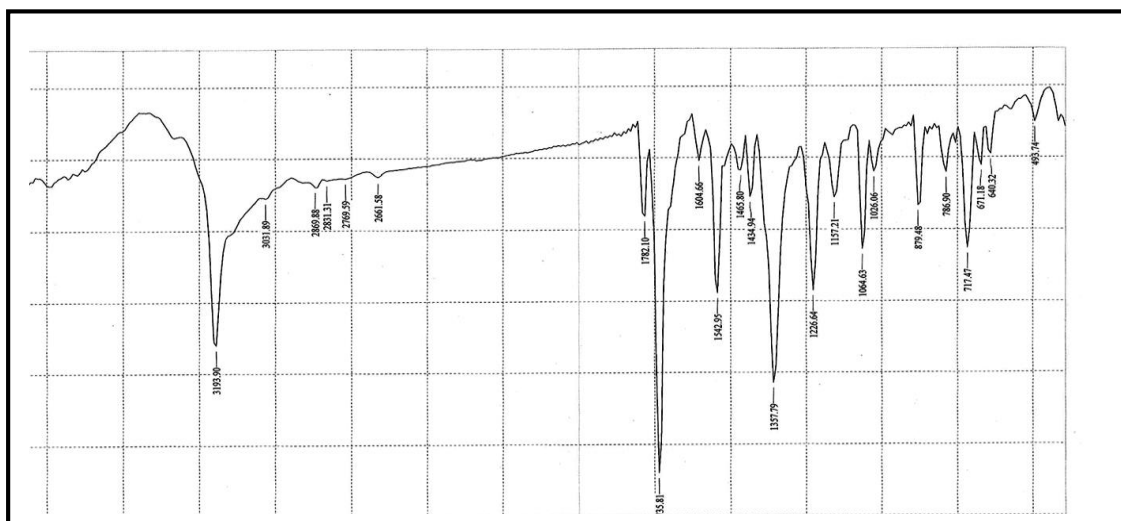
No	Compounds	$\nu(C=O)$	$\nu(C=N)$	$\nu(S-H)$	$\nu(N-H)$	$\nu(O-H)$	$\nu(M-O)$ $\nu(M-N)$
1	L	1782 1735	1604	2661	3193	-	-
2	[Co(L) ₂ (H ₂ O)Cl]Cl	1712 1697	1604	2663	3192	3402	571 -
3	[Ni(L) ₂ (H ₂ O)Cl]Cl	1712 1697	1610	2660	3188	3332	590 -
4	[Cu(L) ₂ Cl ₂]. C ₂ H ₅ OH	1720 1689	1608	2659	3204	3409	552 -
5	[Zn(L)Cl ₂]. H ₂ O	1789 1735 1704 1689	1596 1643	2653	3193	3417	550 493
6	[Cd(L)Cl ₂].H ₂ O	1789 1735 1681 1650	1602 1627	2669	3201	3317	591 462

Table (3): Electronic spectra , magnetic moments and Molar conductivity of the complexes

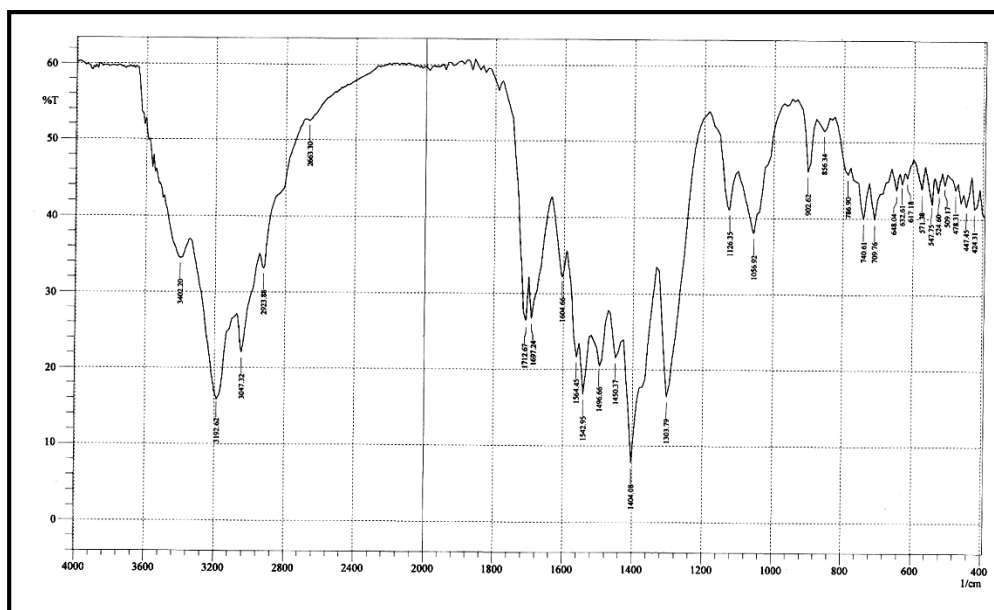
No	compounds	$\lambda(\text{nm})$	Transitions	μ_{eff} (B.M)	Conductivity $\text{ohm}^{-1}.\text{cm}^2.\text{mole}^{-1}$	Geometry
1	L	203, 227 322	$\pi-\pi^*$ $n-\pi^*$	-	-	-
2	$[\text{Co}(\text{L})_2(\text{H}_2\text{O})\text{Cl}]\text{Cl}$	351 701 752 771	Charge transfer ${}^4\text{T}_{1g} \rightarrow {}^4\text{T}_{1g}(\text{P})$ ${}^4\text{T}_{1g} \rightarrow {}^4\text{A}_{2g}(\text{F})$ ${}^4\text{T}_{1g} \rightarrow {}^4\text{T}_{2g}(\text{F})$	5.02	86.2	octahedral
3	$[\text{Ni}(\text{L})_2(\text{H}_2\text{O})\text{Cl}]\text{Cl}$	332 391 607 672	Charge transfer ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{P})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{1g}(\text{F})$ ${}^3\text{A}_{2g}(\text{F}) \rightarrow {}^3\text{T}_{2g}(\text{F})$	3.13	58.5	octahedral
4	$[\text{Cu}(\text{L})_2\text{Cl}_2].\text{C}_2\text{H}_5\text{OH}$	361 731	Charge transfer ${}^5\text{E}_g \rightarrow {}^5\text{T}_{2g}$	1.2	28.2	octahedral
5	$[\text{Zn}(\text{L})\text{Cl}_2].\text{H}_2\text{O}$	348	Charge transfer	Dia.	10.1	Tetrahedral
6	$[\text{Cd}(\text{L})\text{Cl}_2].\text{H}_2\text{O}$	286	Charge transfer	Dia	25.5	Tetrahedral



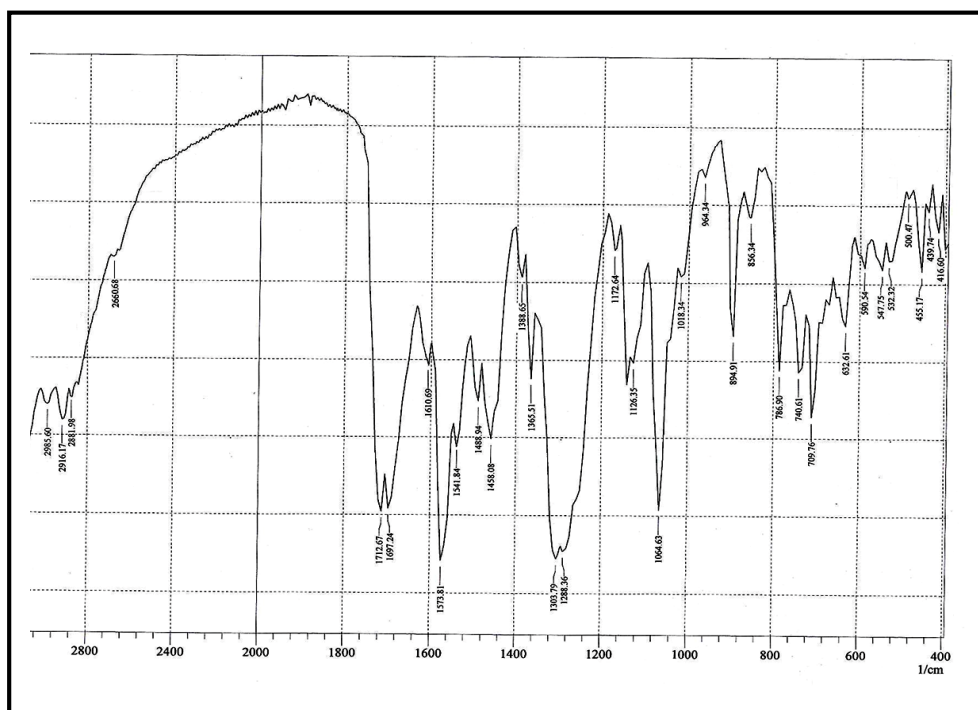
Figure(1): The ¹H-NMR spectra of the ligand (L)



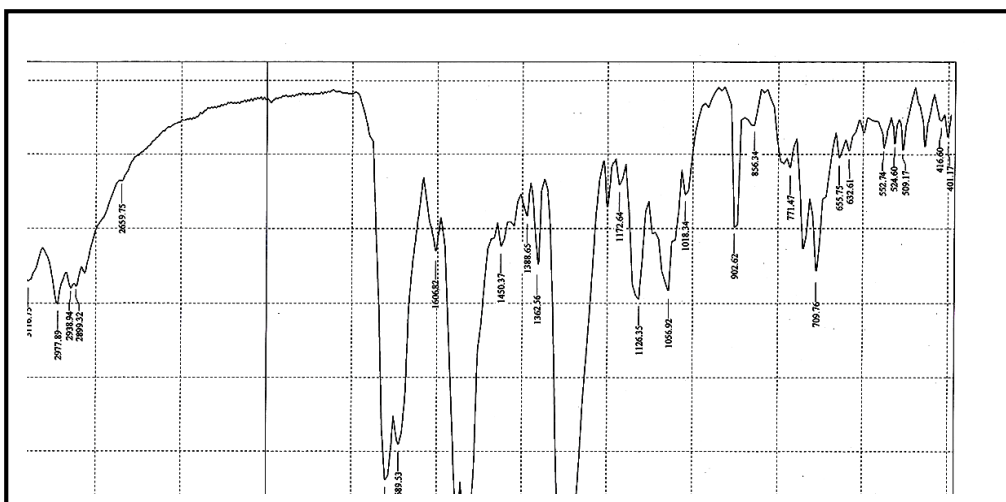
Figure(2): The FTIR spectra of the ligand (L)



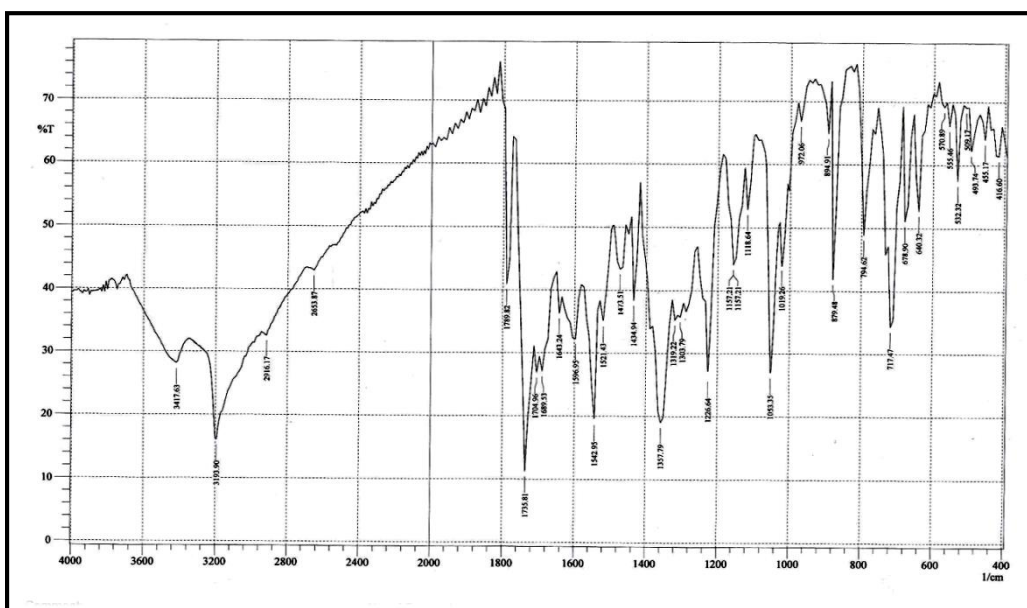
Figure(3): The FTIR spectra of the complex $[Co(L)_2(H_2O)Cl]Cl$



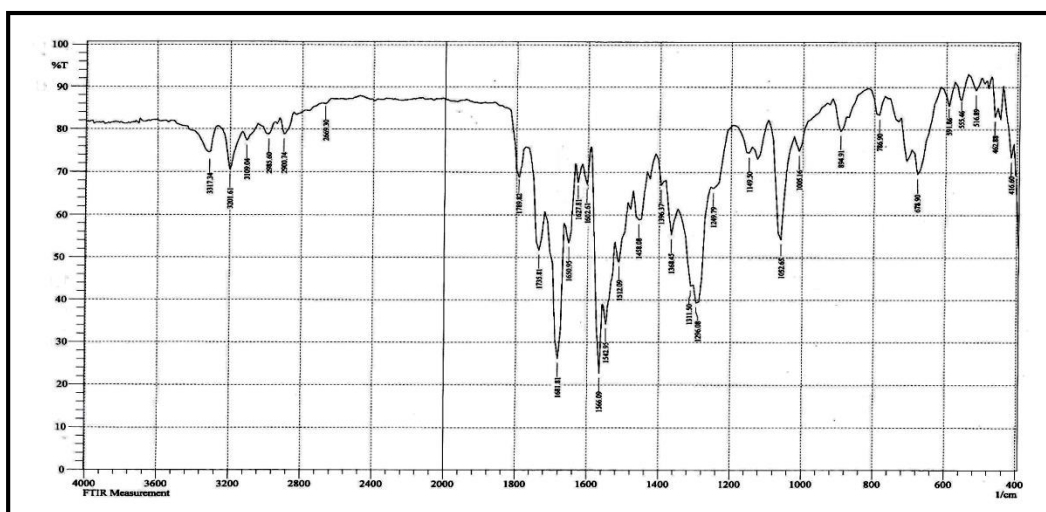
Figure(4): The FTIR spectra of the complex $[Ni(L)_2(H_2O)Cl]Cl$



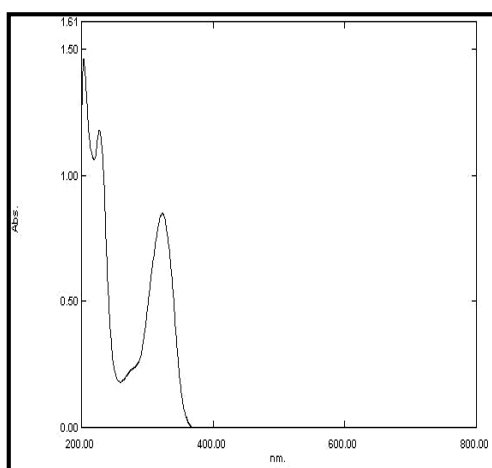
Figure(5):The FTIR spectra of the complex $[Cu(L)_2Cl_2]$. C_2H_5OH



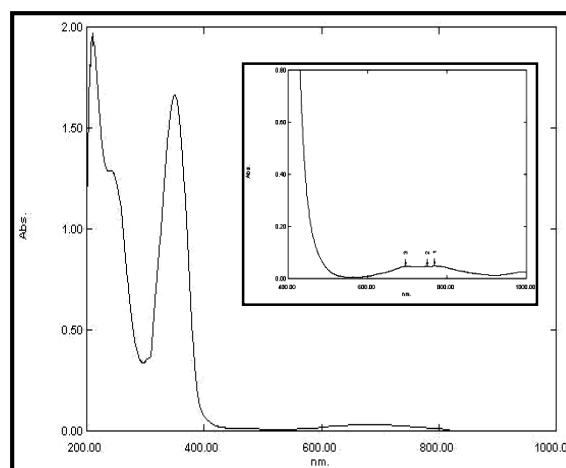
Figure(6): The FTIR spectra of the complex $[Zn(L)Cl_2]$. H_2O



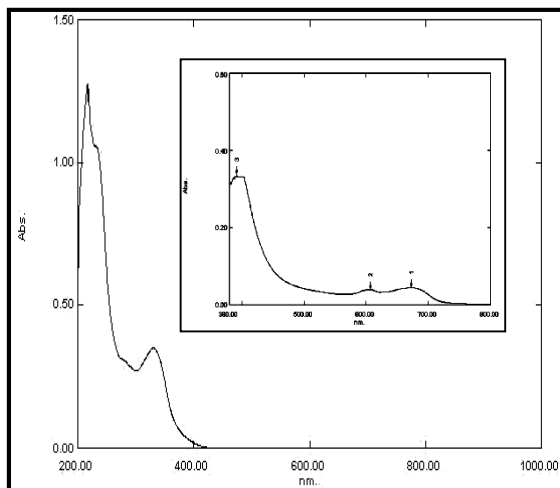
Figure(7): The FTIR spectra of the complex $[Cd(L)Cl_2].H_2O$



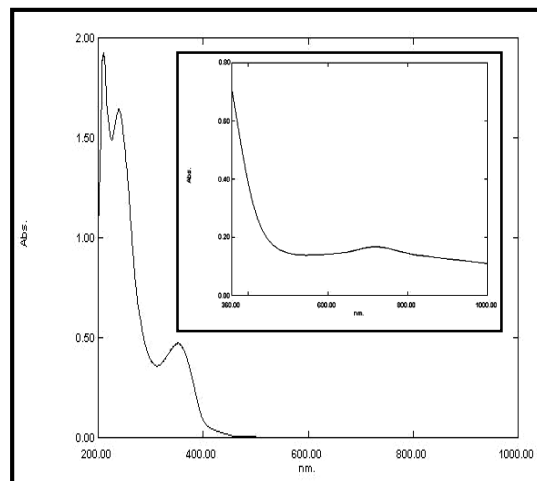
Figure(8): Electronic spectrum of the free ligand



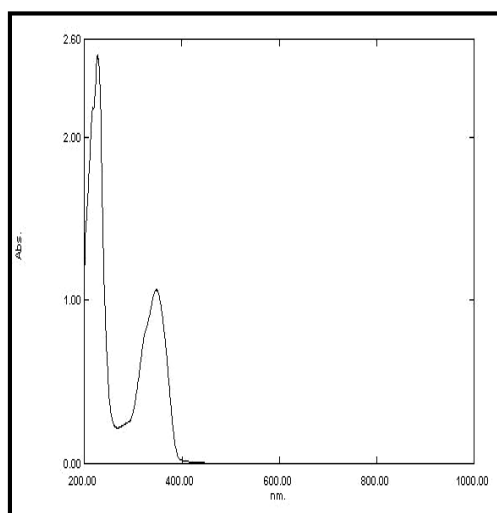
Figure(9): Electronic spectrum of complex $[Co(L)_2(H_2O)Cl]Cl$



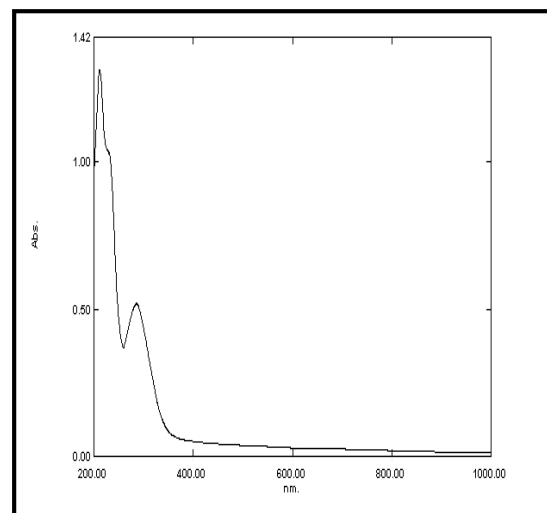
Figure(10): Electronic spectrum of complex [Ni(L)₂(H₂O)Cl]Cl



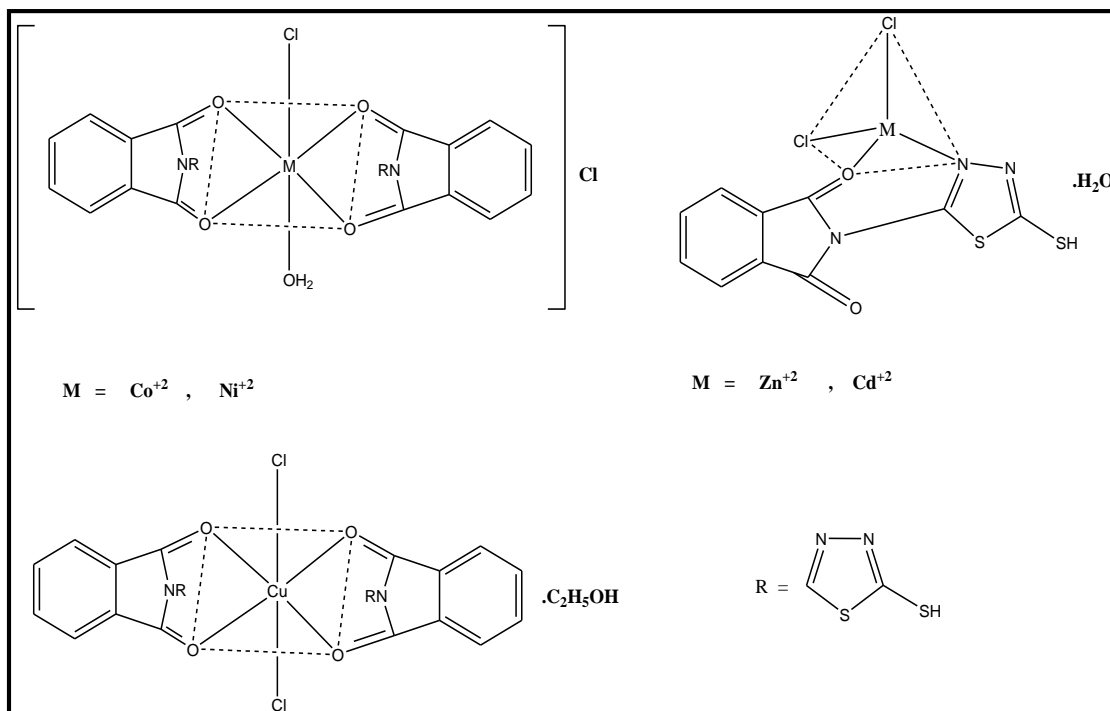
Figure(1): Electronic spectrum of complex [Cu(L)₂Cl₂]. C₂H₅OH



Figure(12): Electronic spectrum of complex [Zn(L)Cl₂]. H₂O



Figure(13): Electronic spectrum of complex [Cd(L)Cl₂].H₂O



Figure(14):The proposed chemical structure formula of the complexes

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***تحضير وتشخيص الليكاند-2(5- مركبتو-1،3،4- ثايدايازول-2- يل)
(Co(II) , Ni(II) , Cu(II), Zn(II) , Cd(II)) ايزوايندولين-1،3- دايون
ومعقداته مع بعض العناصر الفلزية**

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الخلاصة

يتضمن هذا البحث تحضير الليكاند-2(5- مركبتو-1،3،4- ثايدايازول-2- يل) ايزوايندولين-1،3- دايون من تفاعل 5-امينو-4،1،3 - ثايدايازول-2- ثايول مع فتاليك انهدريد باستخدام طريقة الصهر . حضرت المعقدات من تفاعل الليكاند مع كلوريدات الفلزات الأيونية (الكوبلت ، النيكل ، النحاس ، الزنك والكاديوم) الثنائية التكافؤ، شخص الليكاند ومعقداته باستخدام الطرق الطيفية (الاشعة تحت الحمراء ، الاشعة المرئية – فوق البنفسجية ، طيف الرنين النووي المغناطيسي) ، قياسات الحساسية المغناطيسية ، التوصيلية المولارية ، درجات الانصهار والامتصاص الذري . حددت نسبة فلز – ليكاند باستخدام طريقة النسبة المولية . الأشكال المقترحة لمعقدات الكوبلت ، النيكل والنحاس هي أشكال ثمانية السطوح أما معقدات الزنك والكاديوم تكون رباعية السطوح .

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