Journal of Al-Nahrain University **Vol.19 (4), December, 2016, pp.8-15** **Science**

Synthesis and Characterization of Some Anti-bacterial Active Transition Metal Complexes of N'-[2-(2-Phenyl hydrazinyl) Phenyl] Benzothiohydrazide

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Abstract

The ligand N'-[2-(2-Phenyl hydrazinyl) Phenyl] benzothiohydrazide (L) has been prepared from the reaction of ortho amino hydrazo benzene with phenyl isothiocyanate in mole ratio (1:1) and characterized by elemental analysis (C,H,N), H^1, ^{13}C-NMR, IR and Uv-Vis Spectroscopic methods . The complexes of the bivalent ions (Co (II), Ni (II), Cu (II), Cd (II) and Hg (II)) have been prepared and characterized. The structural diagnosis were established by IR, Uv- Visible spectrum, conductivities, atomic absorption and magnetic susceptibility. Chloride ion content was also evaluated by (Mohr method) .The suggested geometry of the complexes were octahedral around the metal ion with the (N,N,S) ligand coordinated in tridentate mode. α, K_f, ε_{max} for the complexes were estimated too. β for Co- complex was calculated. In addition biological activity of the ligand and complexes against two selected types of bacteria were also examined. Some of the complexes exhibited a good anti bacterial activities.

Keywords: Synthesis, benzothiohydrazide, complexes, biological study, metal ions.

Introduction

The hydrazones constitute important class medicinal compounds due to their wide variety of pharmacological and analytical applications. They also bestow applications in floral arena as plant growth regulators [1]. Actually benzothio hydrazide are valuable for the synthesis of transition metal complexes due to their application as ion selective electrode[2]. Some complexes containing nitrogen and sulfer donor atoms are effective as stereo specific catalysts for oxidation[3], reduction[4], hydrolysis[5], biological activity[6] and other transformations[7] of organic and inorganic chemistry. The synthesis and characterization of many metal ions complexes that contain a range of ancillary ligands such as macrocyclic ligand incorporating benzimidazol, oxadiazol, triazole derivatives, orthoaminopheny thio and orthoaminophdrazobenzene [8] are reported. In this paper, we report the synthesis ligand N'-[2-(2-phenylhydrazinyl) phenyl] benzothiohydrazide contains multidonor sites, two imino-nitrogen atoms and thio-sulphur. Since metal ions of class A have the preference for coordination with nitrogen and class B metal ions have preference for coordination with sulphur.

Experimental

A: Chemicals:

All reagents were Analar or chemically pure grade by British Drug Houses (BDH).

- Materials: orthoaminohydrazobenzene ($C_{12}H_{13}N_3$), phenylisothiocynate (C_7H_5NS), Cobalt Chloride hexahydrate ($CoCl_2.6H_2o$), Nickel Chloride hexahydrate ($NiCl_2.6H_2O$), Copper Chloride dihydrate ($CuCl_2.2H_2O$), Cadimum Chloride dihydrate ($CdCl_2.2H_2O$), Mercury Chloride ($HgCl_2$), Ethanol (CH_3 CH_2 OH) 99%, Dimethyl formamide (DMF) 99%, Dimethyl Sulphoxide 99.5% (DMSO), Carbon tetra Chloride 99.5% (CCl_4), Chloroform 99% ($CHCl_3$), Toluen 99% (C_7H_8).

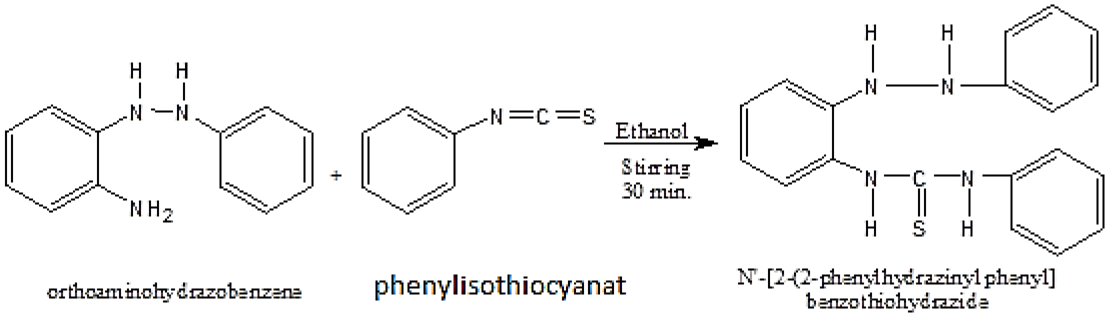
B: Instruments:

- Elemental analysis for the new ligand (L) and complexes were determined by calibration type; Linear Regression Euro EA Elemental Analysis were made in Babel University. Melting points were determined by Gallen-Kamp apparatus. ^{1}H, ^{13}C-NMR spectra were recorded in DMSO–d^6 using Burker 300 MHZ spectrometer at the A ultra university Amman-Jordan. IR spectra were recorded as KBr discs in the range (4000- 400) cm^{-}using shimadzu-FTIR. UV- visible spectra were recorded by (shimadzu-Uv-Vis-160 A) ultra violet spectrophotometer at 25c$^{\circ}$, using 1 cm quartz cell and examined at the range (200- 1100) nm at 10^{-3}M in DMSO. Atomic Absorption (A.A) technique have been measured using Ashimadzu AA 680G atomic absorption spectrophotometer were made in center of Ibn-Cina. Molar conductivity of the complexes were measured on pw 9526 digital conductivity in DMSO at 10^{-3}M. Magnetic susceptibility were recorded by magnetic susceptibility blance, model, MsB-MK_1 and made in Al- Nahrain University.

Preparation

Synthesis of N'-[2-(2-phenyl hydrazine) phenyl] benzothiohydrazide (L)

orthoaminohydrazobenzene (0.199 gm, 0.001 mol) and phenylisothiocynate (0.135 gm, 0.001mol) in ethanel (15 cm^3) was stirred for $\frac{1}{2}$hour. The precipitate was filtered and recrystallized from hot absolute ethanol, fine brown crystals were obtained, m.p 95°C, yield 82%, soluble in most organic solvents as indicated in Table (1).



Synthesis of the metal complexes

One m mole of ethanolic solution of metal salts was added to one m mole of the ligand (L). [$CoCl_2.6H_2O$ (0.24 gm. 1.00 mmol), $NiCl_2.6H_2O$ (0.24gm.1.00 mmol), $CuCl_2.2H_2O$ (0.24gm. 1.00 mmol), $CdCl_2.2H_2O$ (0.22gm. 1.00 mmol) and $HgCl_2$ (0.27gm. 1.00 mmol)] were added to (0.334 gm. 1.00 mmol) of the ligand (L). The mixture was stirred for 30 min. The product was filtered and washed with distilled water then dried under Vacuum. Color, melting point, yield percent, metal analysis and solubility of complexes are given in Table (1).

Table (1)
The physical properties of synthesized ligand (L) and its complexes.

NO	Compound Formula	no.of mole and gm	M .Wt	Color	m.p. °C or (dec.)	Yield %	Elemental analysis Found (calculated)					
							C%	H %	N %	M %	Cl% Chloride content	S%
1	(L)$C_{19}H_{18}N_4S$	1.00 0.33	(334 $g.mol^{-1}$)	Brown	95	82	68.241 (68.263)	5.387 (5.389)	16.765 (16.766)	-	-	9.562 (9.580)
2	[$CoLCI_2$].H_2O	1.00 0.24	(482 $g.mol^{-1}$)	Violet	182	78	47.300 (47.302)	4.144 (4.149)	11.611 (11.618)	12.220 (12.240)	14.693 (14.730)	6.599 (6.639)
3	[$NiLCl_{2.}$].H_2O	1.00 0.24	(481.7 $g.mol^{-1}$)	Green	202	73	47.322 (47.320)	4.150 (4.151)	11.620 (11.625)	12.166 (12.160)	14.732 (14.739)	6.630 (6.643)
4	[$CuLCl_2$] .H_2O	1.00 0.24	(415 $g.mol^{-1}$)	Blue	190	87	46.856 (46.855)	4.115 (4.119)	11.599 (11.590)	12.875 (12.870)	17.100 (17.108)	7.760 (7.710)
5	[$CdLCl_2$] .H_2O	1.00 0.22	(535.4 $g.mol^{-1}$)	Light yellow	195	76	42.580 (42.588)	3.733 (3.720)	10.455 (10.459)	20.973 (20.970)	13.250 (13.261)	5.990 (5.976)
6	[$HgLCl_2$].H_2O	1.00 0.27	(623.5 $g.mol^{-1}$)	Violet	222 (dec)	88	36.560 (36.566)	3.200 (3.203)	8.981 (8.980)	32.165 (32.166)	11.372 (11.387)	5.130 (5.132)

Dec. = Decomposition
C_7H_8 = Toluen

Results and Discussion

The synthesized ligand N'-[2-(2- phenyl hydrazinyl) phenyl]benzothiohydrazide (L) has been characterized by (C. H. N.S), ^1H, ^{13}C-NMR, FT-IR, UV-Vis spectro scopic methods.

1. ^1H- NMR spectrum for the ligand (L).

^1H- NMR spectrum of ligand (L) showed the following characteristic chemical shift (DMSO as a solvent): a multiplet signal at (δ = 7.11,7.09,7.05,7.03,6.77,6.74,6.46,6.44 and 6.42 ppm) which are due to aromatic hydrogen respectively of ligand (L) and a singlet signal at (δ= 5.43 ppm) equivalent to protons assigned to (N–H) group, the chemical shift at (δ 2.45 ppm) is due to DMSO solvent [9].

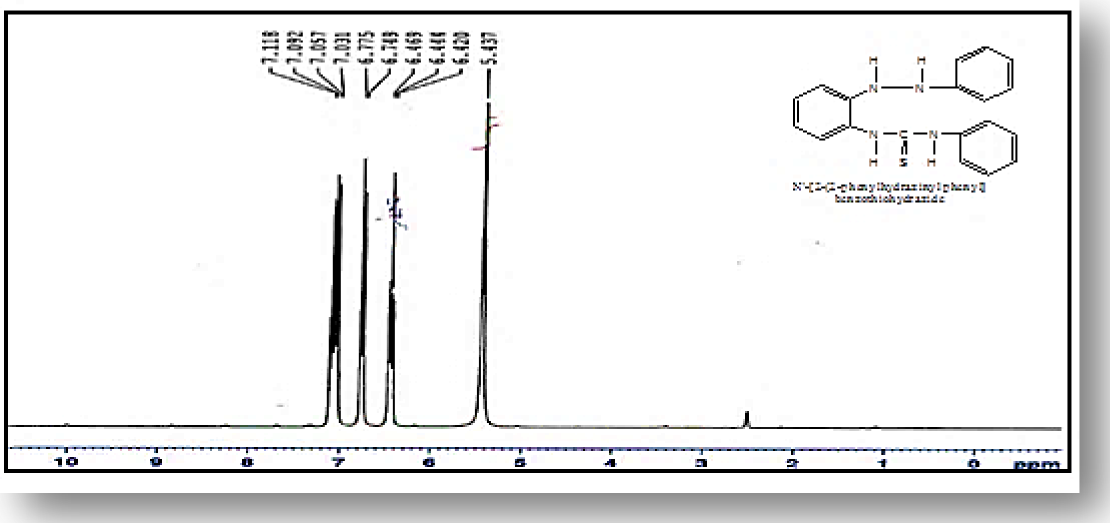


Fig.(1): The ^1H-NMR spectrum of the ligand (L).

2- ^{13}C-NMR spectrum for the ligand (L).

^{13}C-NMR of the free ligand shows asignal at (173.21 ppm)is due to(s=c-) and the signals of the $C_{aromatic}$ carbon of the benzene ring that do not bear hydrogens were observed at (δ 150.59–136.13 ppm) and signals at (δ 135.87-115.30 ppm) assigned to the carbon have one hydrogen. The chemical shift at (δ 40.33 ppm) due to DMSO solvent [10].

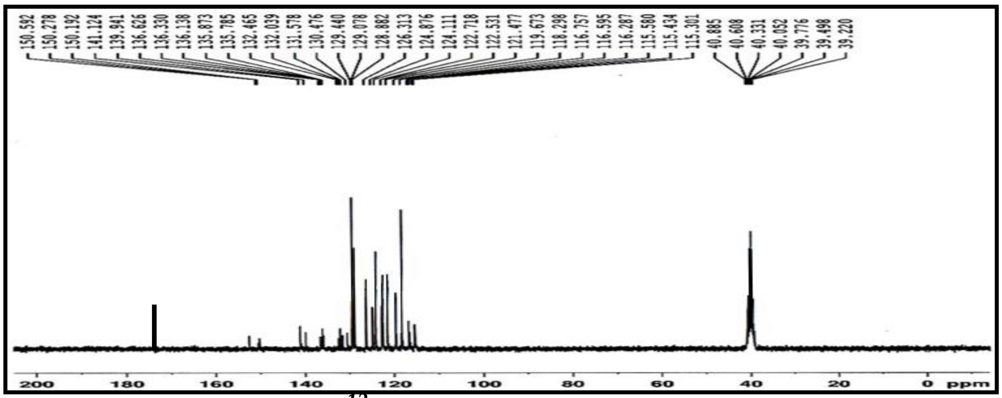


Fig. (2): The ^{13}C-NMR spectrum of the ligand (L).

3- The infrared spectra of the ligand (L) and its complexes

The infrared spectrum of the ligand showed peaks which can be assigned with reasonable certainly. From the point of view of ascertaining the donor sites of the ligand, the following peaks are importants, doublet bands (NH) at (3234, 3188) cm$^{–1}$ and (Ph–NH) at (1625) cm$^{–1}$, (1602) cm$^{–1}$. The thioamide bands I, II, III and IV have been observed at (1562) cm$^{–1}$, (1224) cm$^{–1}$, (921) cm$^{–1}$ and (744) cm$^{–1}$ in the spectrum of the ligand [11].

The infrared spectra of all complexes showed the multi bands (3286-3120)cm^{-1} are split or broader. The bands at (1615-1598)cm^{-1} has been reported .These are precluding any un ambiguous assertion of metal ligand bonding through the NH-phenyl[12]. The thio amide bands are observed at (1560-1546)cm^{-1}, (1220-1211)cm^{-1}, (925-921)cm^{-1} (756-742)cm^{-1} in the spectra of the complexes[13]. It appears that the thioamide band I broad or broad with shoulder as a result of coordination, The band II (νc=s+νc-N) shifted to (1217)cm^{-1} Co(II), (1211) cm^{-1} Ni (II), (1220)cm^{-1} Cu(II), (1217)cm^{-1}Cd (II) and (1220) cm^{-1} Hg (II) complexes. The bands III and IV are shifted to lower or higher frecuencies as a result of coordination. Bands (M-N) in the region (516-509) cm^{-1} suggesting the coordination through nitrogen atom. The bands characteristic of coordinated water and (M-O) are seen in Co(II) at (813,578)cm^{-1}, Ni (II) at (829, 582) cm^{-1}, Cu(II) at (810,582) cm^{-1}, Cd (II) in the region (825,585) cm^{-1} while Hg (II) at (810, 578) cm^{-1}[14]. Table (2) describes characteristic stretching vibration frequencies (cm$^{–1}$) located in the FT–IR of the ligand and its complexes.

Table (2)

Characteristic stretching vibration frequencies (cm$^{–1}$) located in the FT-IR of the ligand and its complexes.

No	Compound Formula	υ(N-H) cm^{-1}	υ(C-H) cm^{-1} Ar.	υ(C-N) + υ(C=S)cm^{-1}	υ(N-N) cm^{-1}	υ(C=S) cm^{-1}	υ(M-N) cm^{-1}	υ(M-S) cm^{-1}	υ(M-Cl) cm^{-1}	υ(M-O) cm^{-1}
1	(L)$C_{19}H_{18}N_4S$	3234 (m.br),3188(m.s) 1625(s.s),1602(m.s), 1310(m.s),1249(m.s)	3053(m.s)	1562(m.s) 1224(m.s)	1446(s.s)	921(s.s) 744(s.s)	–	–	–	–
2	[$CoLCl_2$].H_2O	3286 (m.s) 3170(m.s) 1598(s.s), 1311(m.s), 1273(s.s)	3080(m.s)	1550(s.s) 1217(m.s)	1456(m.s)	925(m.s) 748(s.s)	510(sh)	476(s.s)	428(s.s)	813(s.s) 578(m.s)
3	[$NiLCl_2$].H_2O	3246(m.s),3120(s.s) 1615(sh), 1309(s.s) 1276(s.s)	3090(m.s)	1546(m.br) 1211(m.br)	1444(s.s)	923(m.s) 746(s.s)	509(m.s)	449(s.s)	416(s.s)	829(m.s) 582(m.s)
4	[Cu LCl_2].H_2O	3250(sym)(m.s), 3190 1600(s.s) 1315(m.br), 1273(s.s)	3088(m.s)	1560(m.br) 1220(m.br)	1450(s.s)	923(m.s) 750(s.s)	515(m.s)	450(m.s)	455(s.s)	810(s.s) 582(m.s)
5	[$CdLCl_2$].H_2O	3269(s.s),3180(m.s) 1605(s.s) 1311(m.s), 1276(s.s)	3062(m.s)	1556(m.br) 1217(sh)	1452(m.s)	921(m.s) 742(m.s)	510(m.s)	449(m.s)	428(m.s)	825(m.br) 585(m.s)
6	[$HgLCl_2$].H_2O	3220(m.s),3175(m.s) 1612(s.s), 1317(s.s), 1273(m.s)	3059(m.s)	1552(m.br) 1220(m.br)	1450(s.s)	923(s) 756(s.s)	516(m.br)	449(m.s)	428(m.s)	810(s.s) 578(m.br)

Ar = aromatic,	***asym = asymmetric,***	***sym = symmetric,***	***sh = shoulder***
br=broad,	***m=medium,***	***s=strong,***	***w=weak***

4- The U.V- visible spectra of the ligand (L) and complexes

The UV- visible spectrum of the ligand (L) in Ethanol solvent exhibited strong, absorption bands at 295nm (33898 cm^{-1}) and 340nm (29411 cm^{-1}). This may attributed to the $\pi-\pi^*$ and $n-\pi^*$transitions [15].

The UV- visible spectrum of Co^{+2} complex showed bands in the region 800nm (12500cm^{-1}) due to $^4T_1g \rightarrow {}^4T_2g_{(F)}$,645 nm (15503 cm^{-1}) due to $^4T_1g \rightarrow {}^4A_2g$ and 520nm (19230 cm^{-1}) due to $^4T_1g(F) \rightarrow {}^2T_1g(p)$ [16].

The ratio of 19230 cm^{-1} to 12500 cm^{-1} is 1.54 which fits with Tanaba- sugano d^7 curve for E/ B = 26 and Δ_o/ B' = 16. Δ_o= 12500 cm^{-1}, B' complex = 12500/16 =781 $cm^{-1}/_1$ B for free Co^{+2} = 971 cm^{-1}.

β(nephelaxeticeffect)=B'complex/BCo^{+2}=781/ 971=0.804Which indicates of d-electron delocalization on the ligand hence a some covalent character is observed in the complex

The UV- visible spectrum of Ni^{+2} showed one band at 825 nm (12121cm^{-1}) which is due to $^3A_2g \rightarrow {}^3T_1g$. Cu^{+2} complex appeared band at 645nm (15503 cm^{-1}) due to $^2Eg \rightarrow {}^2T_2g$.

The Uv-visible spectra of Cd^{+2} and Hg^{+2} complexes showed shifted bands compared with free ligand (L) are due to charge transfer Table (3).

Table (3)
Electronic spectra (DMSO), Conductance in DMSO, Magnetic moment (B.M) of the ligand (L) and its complexes.

NO	Compound Formula	λ (nm) cm^{-1}	Assignment Bands	Λ s. cm^{-1} DMSO (10^{-3} M)	μ_{eff} (B.M)	Suggested structure
1	(L)$C_{19}H_{18}N_4S$	33898(295) 29411(340)	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	–	–	–
2	$[CoLCl_2].H_2O$	19230(520) 15503(645) 12500(800)	$^4T_1g_{(F)} \rightarrow {}^2T_1g_{(P)}$ $^4T_1g \rightarrow {}^4A_2g$ $^4T_1g \rightarrow {}^2T_2g_{(F)}$	6.56	3.83	Octahedral
3	$[NiLCl_2].H_2O$	12121(825)	$^3A_2g \rightarrow {}^3T_1g$	8.63	2.84	Octahedral
4	$[CuLCl_2].H_2O$	15503(645)	$^2T_2g \rightarrow {}^2Eg$	4.21	1.72	Octahedral
5	$[CdLCl_2].H_2O$	27932(358)	Charge-Transfer	7.82	Dia	Octahedral
6	$[HgLCl_2].H_2O$	25188(397)	Charge-Transfer	10.11	Dia	Octahedral

The molar conductance were measured in DMSO solvent and concentration (10^{-3}M) at room temperature. The molar conductance values of the synthesized complexes were at the range (10.11-4.21$ohm^{-1}.cm^2.mol^{-1}$). These results show that complexes of ($Co^{(II)}$, $Ni^{(II)}$, $Cu^{(II),}$ $Cd^{(II)}$ and $Hg^{(II)}$) are non – electrolytes [17].

The magnetic moment measure ment for the solid Co(II) complex(3.83) was indicative of three unpaired electrons per Co(II) ion suggesting consistence with their octahedral environment. Ni(II) complex showed the magnetic moment value of (2.84) B.M. (Octahedral range). The magnetic susceptibility measurements of Cu(II) complex is (1.72) B.M. which suggests the presence of one unpaired electron. Electronic spectraldata, conductance in (DMSO) and magnetic moment (B.M) of the ligand and its complexes are given in Table (3).

Study of Co^{+2} and Ni^{+2} complexes formation in solution

The complexes of the ligand (L) with selected metal ions (Co^{+2}, Ni^{+2}) were studied in solution using ethanol as solvent, in order to determine (M:L) ratio in the prepared complexes, following molar ratio method[18]. A series of solution were prepared having a constant concentration (C) 10^{-3} M of the hydrated metal salts and the ligand (L). The (M:L) ratio was determined from the relation ship between the absorption of the observed light and the mole ratio (M:L) found to be (1:1) for the complexes mentioned above . The results of complexes formation in solution are shown in Fig. (3,4).

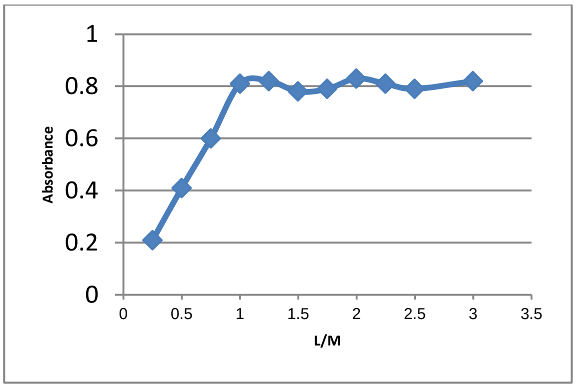


Fig.(3): The molar- ratio curve of the complex [CoLCl$_2$].H$_2$O.

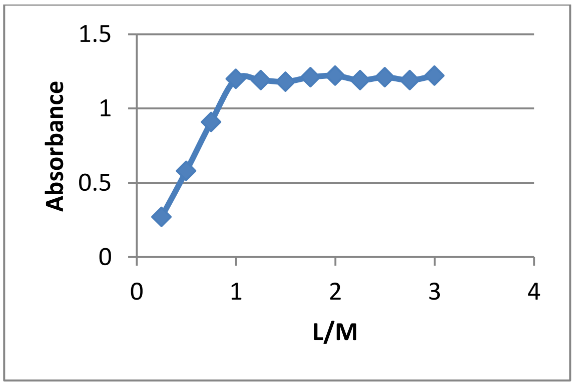


Fig. (4): The molar- ratio curve of the complex [NiLCl$_2$].H$_2$O.

The stability constant of the (K_f) was evaluated using the following equations:

$K_f = 1- \alpha / \alpha^2 c$ …. (1),

$\alpha = Am - As / Am$ ….. (2)

(α) is the degree of the dissociation, (C) is the concentration of the complex (10^{-3}M) Table (4). (Am) and (A) are the absorbance value of the partially and fully formed complex respectively. The absorbance of the solutions were measured (λ_{max}) of the maximum absorption. The molar absorptivity (ε_{max}) has been calculated using the following equation;

$A=\varepsilon_{max}$. b. C… (3), (A) is the average of three measuremens of the absorption containing the same amount of metal ion and three fold excess of ligand. (b) is the depth of the quartz cell usually equal (1) cm.

Table (4)

As, Am, K_f, ε_{max} and λ_{max} of the Co^{+2} and Ni^{+2} complexes.

No.	Compound	As	Am	α	Formation constant (K_f)	Molar absorpitivityε_{max} L.mol^{-1}.cm^{-1}	λ_{max} (nm)
1	Co-complex	0.80	0.83	0.0361	7.378×10^5	3122	301
2	Ni-complex	1.20	1.22	0.016	3.843×10^6	2925	380

Bacterial activity studies

The synthesized ligand and its complexes were screened for their antibacterial activity against staphylococcus aureus and Escherichia coli activity at (10^{-3}) mole/ liter concentration at (37C°) for (24) hrs. The zones of inhibitions the (antimicrobial activity) have been presented in Table (5). The results of antibacterial activity of ligand and its complexes show good activity against the two bacteria used.[19,20].

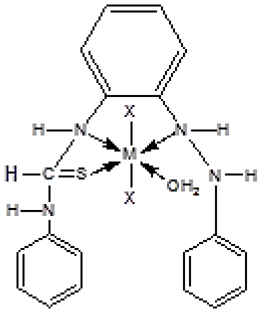
Table (5)
Effect of ligand and its complexes on gram positive and gram negative bacteria.

No.	Compound	Diameter of inhibition zone (mm) at concentration 1mg/ml	Diameter of inhibition zone (mm) at concentration 5mg/ml	Diameter of inhibition zone (mm) at concentration 1mg/ml	Diameter of inhibition zone (mm) at concentration 5mg/ml
		Staphylococcus aureus		***Escherichia. Coli***	
	Cephalosporin	25.8	27.2	26.4	28.6
1	(L)$C_{19}H_{18}N_4S$	14	14.8	14.6	15.2
2	$[CoLCl_2].H_2O$	13	13.4	13.2	13.4
3	$[NiLCl_2].H_2O$	16	16.4	16.2	16.6
4	$[CuLCl_2].H_2O$	15	15.2	15.1	15.4
5	$[HgLCl_2].H_2O$	16.6	17.2	17.6	18

Conclusion

A series of complexes of Co^{+2}, Ni^{+2}, Cu^{+2}, Cd^{+2}, and Hg^{+2} with N'[2-(2-phenyl hydrazinyl) phenyl]benzothio hydrazide (L) have been prepared and characterized.

The tridentate ligand (L) (N,N,S) is binding metal ion forming octahedral structure follow;



M= Co^{+2} , Ni^{+2} , Cu^{+2}, Cd^{+2}, Hg^{+2}
X = Cl^-

Bacterial effects of new ligand and its complexes exhibited antibacterial activity against both gram positive and gram negative bacteria.

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الخلاصة

تم تحضير الليكاند (L)، N'-(2-(2- فنيل هيدرازين يل) فنيل) بنزوثايوهيدرا زايد من تفاعل اورثوامينوهيدرازوبنزين مع فنيل ايزوثايو سيانيت وبنسبة 1:1. تم تشخيص (L) بواسطة التحليل الدقيق للعناصر (N,H,C)، طيف الرنين النووي المغناطيسي، الاشعة تحت الحمراء والاشعة فوق البنفسجية– المرئية. كما تم تحضير وتشخيص معقدات أملاح بعض ايونات العناصر الثنائية التكافؤ (Co، Ni، Cu، Cd وHg). استخدمت تقنيات تحليل العناصر (N,H,C)، الاشعة تحت الحمراء، الاشعة فوق البنفسجية– المرئية، التوصيلية الكهربائية، الامتصاصية الذرية والحساسية المغناطيسية كما تم حساب نسبة الكلوريد وأستنتج من التحاليل ان المعقدات لها شكل ثماني السطوح حول ايون الفلز مع الليكاند (L) ثلاثي السن (S,N,N) وتم تحديد قيم α، K_f,، ε_{max} لهذه المعقدات وقد تم حساب قيمة β لمعقد الكوبلت. كما تمت دراسة الفعالية البايولوجية لليكاند ومعقداته ضد نوعين من البكتريا المنتخبة، حيث أظهرت النتائج لهذه المعقدات قابلية متفاوته على قتل الأنواع المنتخبة من البكتريا.