

Preparation , characterazition and biological activity of some complexes of potassium 2-carbomethoxy amino-5- trithiocarbonate 1,3,4-thiadiazole

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Abstract :

In this study a new ligand ,(potassium 2-carbomethoxy amino-5-trithiocarbonate 1,3,4-thiadiazole) (L) has been prepared from 2-carbomethoxy amino-5-mercapto 1,3,4-thiadiazole with CS₂ in alkali media . The product has been isolated and characterized by appropriate physical measurements, vibrational and electronic spectroscopy. The ligand was used to prepare a number of complexes with some metal ions Co(II), Ni(II) and Cu(II). These complexes have been characterized by FT-IR, UV-Vis spectra, molar conductivity, magnetic susceptibility, melting points and atomic absorption measurements. The nickel and copper complexes have an octahedral geometry while cobalt complex has a tetrahedral geometry. The nature of bonding between the metal ion and the donor atom of the ligand was demonstrated through the calculated Racah parameter and other ligand field parameters, which have been calculated by using a suitable Tanaba-Sugano diagram The biological activity of the ligand and it's complexes have been examined against two selected microorganisms *Pseudomonas aeruginosa* and *Staphylococcus aureus* using (10mM) and (5mM) in nutrient agar medium. The results were showed enhancement of activity of some complexes compared to that of the respective ligand, which were attributed to the synergetic effect between the metal ion and the ligand in addition to the difference in the structural varieties.

Key words : Thiadiazole derivatives, Thiadiazole complexes, Thiadiazole complexes, biological activity, Transition metal complexes.

Introduction :

The aromatic thiadiazole nucleus is associated with a variety of pharmacological action, such as fungicidal [1] and leshmanicides [2] activities . These activities are probably due to the presence of the $-N=C-S$ group [3]. Trithiocarbonates are used as antioxidant additives to extreme pressure lubricant oil and greases [4].Also activity of the thiocarbonate and thiocarbamate against human tumor [5]. Trithiocarbonate complexes have received attention because of the dual

nature of metal-CS₃ moiety as an electrophilic and nucleophilic reagents, which makes them versatile intermediates for the synthesis of other oil thio species Although the main application is the treatment of a variety of rheumatic diseases, some of these compounds have shown to have antileishmanial activity in vitro inhibitory effect on HIV or activity tumor cell [6,7]. Organotrithiocarbonates have found many applications in various fields such as in analysis, organic synthesis,

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medicine, industry and agriculture, some of these applications are as flotation agents, vulcanization accelerators, pesticides, plant defoliants, rust inhibitor, lubricating oil additives, and some have recently reported to possess activity as anti-radiation drugs [8,9]

Materials and methods :

A-Materials, physical measurements and analysis :

The chemicals used in this work were obtained from BDH. and Fluka, they were pure grade reagents . A Flame emission spectrophotometer type Perkin Elemer 5000 was used for metal content determination, FTIR spectra were recorded using Shimadzo-8000 spectrophotometer using CsI discs in the rang (4000-200) cm^{-1} . Electronic spectra were recorded by using Shimadza UV-Visible (160A) ultra violet spectrophotometer at wave length in the rang (1100-200)nm . Electrical conductivity was measured by using Coring conductometer and magnetic susceptibility was measured by Johnson Matting Catalytic System Division and melting point was measured by using Gallencamp M.F.B 600.01 .

B-Preparation of the ligand :

1-Preparation of 2-amino-5-mercapto-1,3,4-thiadiazole (A) :

This compound has been prepared according to Petrow and co-workers [10-12] by the reaction of the thiosemic- carbazide with anhydrous sodium carbonate and carbon disulfide, the mixture acidified with concentrated HCl to get the final pale yellow product .

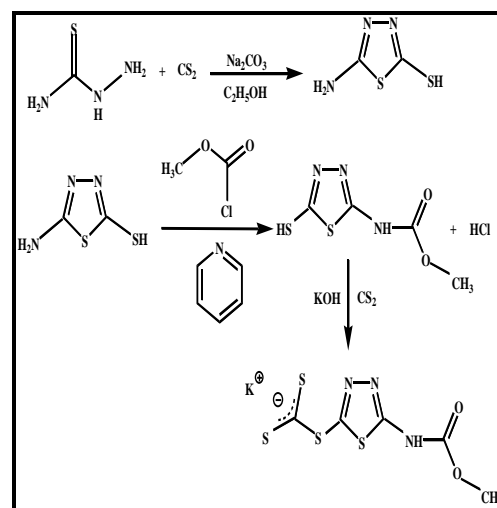
2-Preparation of 2-carbomethoxy amino-5-mercapto-1,3,4-thiadiazole (B) :

This compound has been prepared by the reaction of methyl chloroformate with compound (A) in presence of

pyridine [13], the mixture has been diluted by distilled water and acidified with concentrated hydrochloridacid to get pH=3 .The final product (B) was a yellow crystals.

3-Preparation of potassium-2-carbomethoxy amino -5-trithiocarbonate-1,3,4-thiadiazole (L) :

A Carbon disulfide(1.5ml) was added to potassium hydroxide(0.01mole), This mixture was added to the solution of compound (B) (0.01mole) in (20ml) ethanol, the mixture was refluxed for 3 hours, then the solvent was evaporated and the precipitate was filtrated and recrystalized from ethanol .The physical properties of the products (A) , (B) and (L) are listed in table (1).



Preparation of potassium 3-carbomethoxy amino-5-trithiocarbonate-1,3,4-thiadiazole

C-Preparation of Co(II), Ni(II) and Cu(II) complexes :

Ethanol solution of the selected metal ion salts (1mmole,5ml) of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ was added to ethanol solution (1mmole,10ml) of (L) for copper and cobalt and (2mmole,10ml) for nickel complex. The mixture was refluxed with stirring for one hour . The produced complexes were filtered

and washed with hot ethanol .The physical properties of these complexes are listed in table (1).

D- Biological activity of the ligand and it's metal complexes :

The biological activity of the ligand (L) and it's complexes was examined against two types of bacteria, *Pseudomonas aeruginosa* as grame-negative bacteria and *Staphylococcus aureus* as grame-positive bacteria, were cultivated in nutrient agar medium All samples were freshly prepared by dissolving them in DMSO to obtain a final concentration of 5mM and 10mM. The antibacterial test was performed according to disc diffusion method [14], which involves the exposure of micro-organism on agar

plate .The plates were incubated for 24 hours at 37 C°, the zone of inhibition of bacterial growth around the disc was observed, the results are shown in table (4).

Results and Discussion:

1-Metal Analysis :

The physical and analytical data of the metal complexes are given in table(1).The results obtained from metal analysis are in a satisfactory agreement with the calculated values .The suggested molecular formula was also supported by spectral as well as magnetic moments measurements.

Table(1): Physical properties of the ligand and it's metal complexes .

Compounds	Colour	m.p(C°)	Yield(%)	metal content(%)		Suggested formula
				Cal.	Found	
A	Pale yellow	230-233	72	-	-	C ₂ H ₃ N ₃ S ₂
B	Light yellow	180	68	-	-	C ₄ H ₅ N ₃ O ₂ S
L	Yellow	210	62	-	-	C ₅ H ₄ N ₃ O ₂ S ₄ K
CoL	Dark green	230d	64	10.83	10.79	[CoLH ₂ OCl].3H ₂ O
NiL	Green	260d	48	6.30	6.40	[NiL ₂ Cl ₂].2H ₂ O
CuL	Green	253d	60	13.3	12.8	[Cu ₂ L ₂ Cl ₂ (H ₂ O) ₂].3C ₂ H ₅ OH

d= decomposed

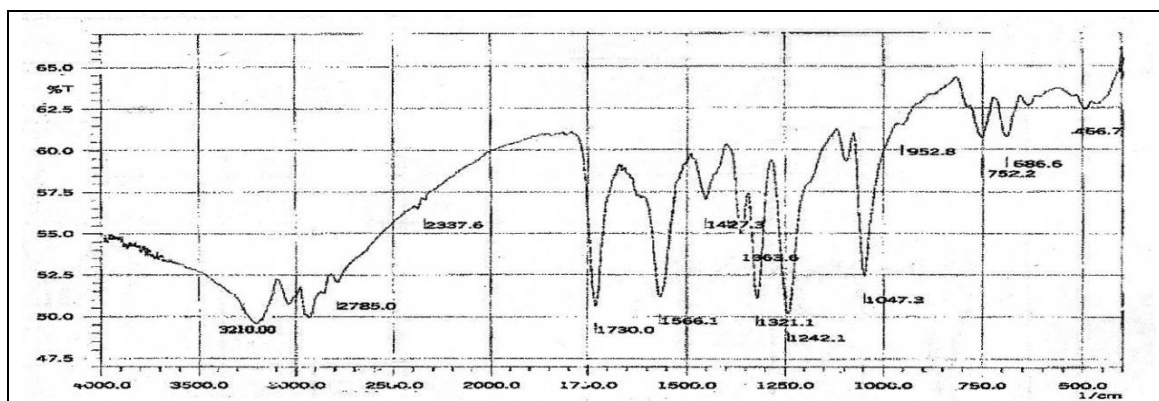
2-Infrared spectroscopic study :

The most characteristic I.R spectral bands of trithiocarbonate are those of SCS₂ moiety, the changes in their position upon the complexation of the free ligand gave a good idea about structural type for such compounds, in addition to the spectral vibrational bands of metal-sulfur band ,it can be concluded that partially delocalized C-S double bond is the main contributor spectral I.R . The band at 752cm⁻¹ is attributed to single bond stretching for the CS₂ group .Table (2) shows the most dignostic bands for the ligand and it's complexes. The spectra of cobalt and copper complexes showed, firstly:

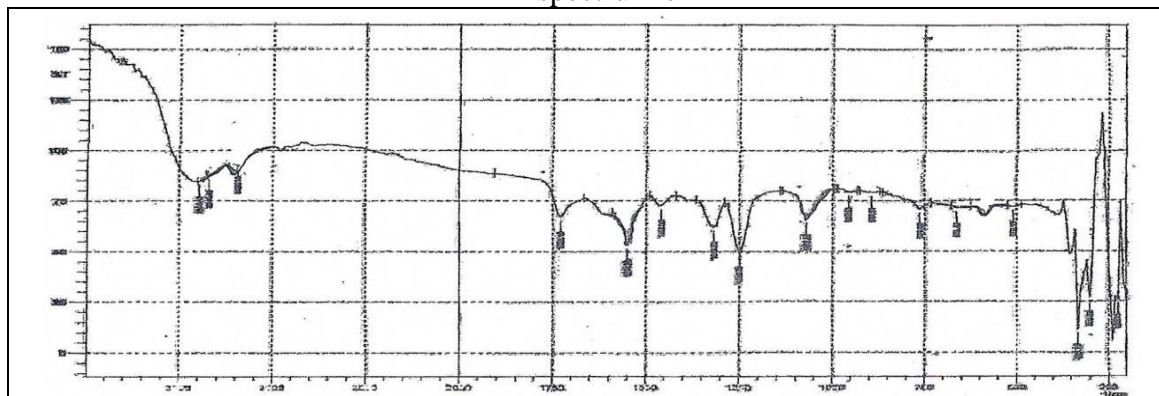
splitting of the band C-S into three weak bands lowering frequency for Co(II) complex (941,943,948)cm⁻¹ and for Cu(II)complex (940,942,948)cm⁻¹, the νC=S undergos lower frequency by 7 cm⁻¹, displayed and shifted νC-S to higher frequency by 16 cm⁻¹and there is no change in the absorption bands of the νC=N, νNCS and νCOC mode in these complexes,this excluded the possibility of nitrogen and oxygen atoms participation in coordination, therefore the ligand may be considered to behave as bidentate through the (S-S) . The IR spectrum of nickel complex showed that the stretching frequency of νC=N and νNCS bands

undergo shifting to higher frequency with low intensity by 24cm^{-1} , and the absorption bands of νCOC mode and carbonyl group shifting to lower frequency by 10cm^{-1} , this indicates that the participation of nitrogen of the ring and oxygen of the acetate coordination [15,16], beside that there is no change in SCS_2 group in this complex, this excludes the possibility of thial group participation in coordination. This

ligand may be considered to behave as bidentate (N and O). These absorptions were further indicated by appearance of $\nu\text{M-N}$, $\nu\text{M-O}$ and $\nu\text{M-X}$ respectively. A broad band was observed in the spectra of the prepared complexes, assigned to νOH and suggested the presence of water or ethanol molecules in crystal lattice of the complexes [16].



FTIR spectrum of L



FTIR spectrum of CoL

Table(2): Selected bands of thiadiazole trithiocarbonate complexes .

Comp.	νOH	$\nu\text{N-H}$	$\nu\text{C=N}$	νNCS	$\nu\text{C=S}$	$\nu\text{C-S}$	$\nu\text{C---S}$	$\nu\text{C=O}$	νCOC	$\nu\text{M-N}$	$\nu\text{M-S}$	$\nu\text{M-X}$
L	-	3210	1566	1427	1047	752	952	1730	1083	-	-	-
CoL	3394	3210	1566	1427	1041	763	941-943-948	1730	1080	-	435	M-Cl 380 M-O 483
NiL	3409	3210	1590	1450	1047	752	952	1720	1073	501	-	M-O 480
CuL	3363 3448	3205	1566	1427	1040	768	940-942-984	1730	1080	-	455	378

3-Electronic spectra, Magnetic susceptibility and Conductivity measurements :

As further structural tools, electronic spectra, magnetic and conductivity studies were used to confirm the geometry of the prepared complexes. The electronic spectra of the cobalt, nickel and copper complexes were recorded for their solution in DMSO in the range (200-1100)nm.

CoL complex :

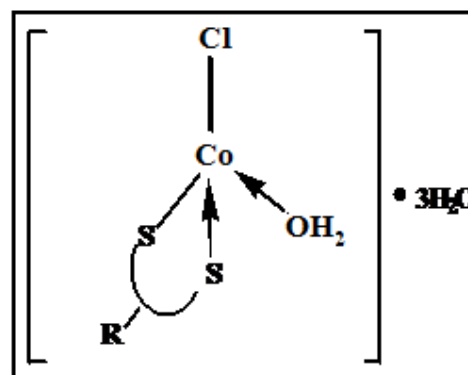
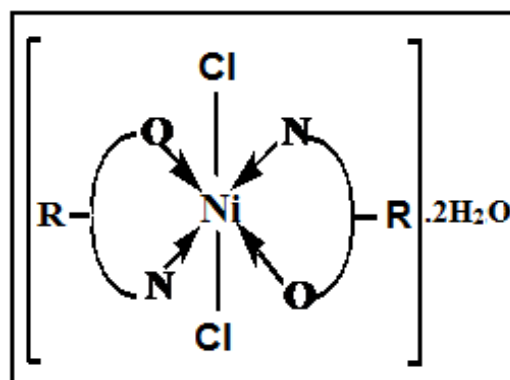
The dark green cobalt(II) complex with the ligand showed a magnetic moment of (4.73)BM, which indicates a high-spin type tetrahedral [17,18]. Electronic spectrum in DMSO solution exhibited splitted bands in the 16,393 and 14,771 cm^{-1} , the splitting band is typical tetrahedral type and can be assigned to ${}^4A_2 \rightarrow {}^4T_{1(p)}$ (ν_3), in addition there is band at (3394) cm^{-1} in the infrared spectrum can be assigned to ${}^4A_2 \rightarrow {}^4T_{2(F)}$ (ν_1) transition . The (ν_2) and various ligand field parameter were calculated, table(3). According to these results and the previous data, the tetrahedral structure can be proposed. By referencing to Tanaba-Sugano diagram for d^7 configuration [19] and the spin-orbit coupling constant (λ') was calculated as :

$$\mu_{\text{obs}} = \mu_{\text{s.o}} - 15.59\lambda' / 10Dq$$

Where: μ_{obs} = The observed effective magnetic moment ; $\mu_{\text{s.o}}$ = the electronic spin only magnetic moment .The resulting value ($\lambda' = -192.3$) showed that the present complex to be distorted tetrahedral [20,21].The factor β was calculated and found to be (0.61) indicating high degree of covalence in bonding of ligand-donor atoms with cobalt(II) ion .The molar conductance showed that the complex is non ionic .

NiL complex :

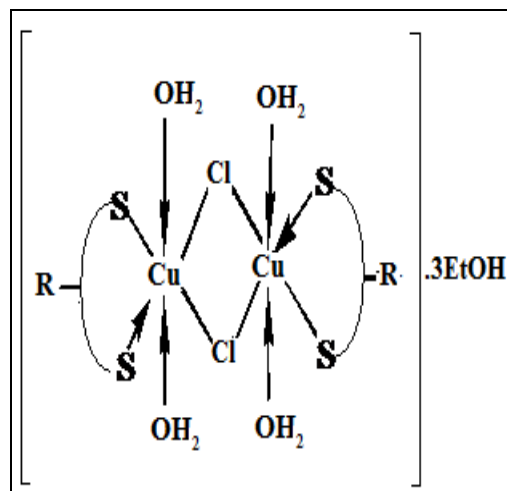
The UV-Vis spectrum of the present green Ni(II) complex ,showed two bands at (14,750 cm^{-1}) and (24,096 cm^{-1}) can be assigned to ν_2 and ν_3 respectively [19,22] and weak band at (10,080 cm^{-1}) can be assigned to (ν_1) transition [19,23,24].



The Racah parameter B', β and the value of $10Dq(\nu_1)$ also calculated according to Tanaba-Sugano diagram for d^8 configuration . The value of the calculated μ_{eff} was (2.7BM), this value is in the range of an octahedral geometry [19,25-27], table(3) .The conductivity measurement showed that the complex is a non ionic, from the spectroscopic study and metal analysis, the octahedral structure can be postulated.

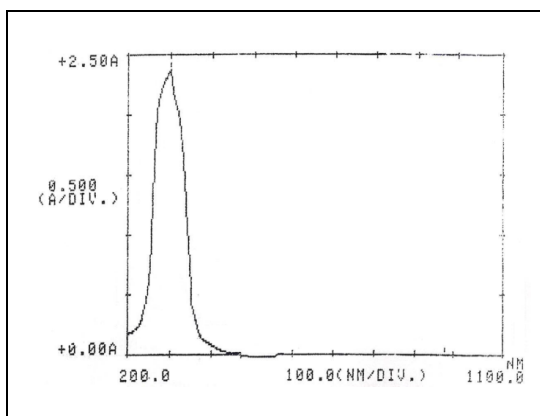
CuL complex :

The electronic spectrum of green complex showed one broad absorption band in the region around $(12,901)\text{cm}^{-1}$ which can be attributed to ${}^2B_{1g} \rightarrow {}^2B_{2g}$ and ${}^2B_{1g} \rightarrow {}^2E_g$ transition . The electronic spectra coupled with magnetic moment $(1.02)\text{B.M}$, this value in the range of dimeric octahedral geometry [28,29].The conductivity measurement showed that the complex was non ionic, table(3) .The result from above in addition to IR spectrum , flame atomic absorption analysis which gave a good support, the structure of this complex can be suggested :

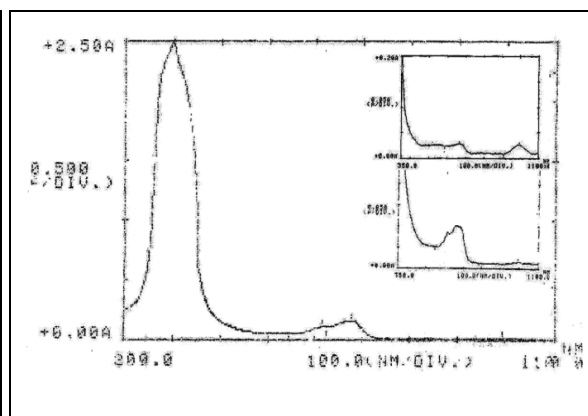


Table(3): Electronic spectral data, magnetic moment, conductivity measurements and suggested structure for the complexes

Comp	Bands cm^{-1}	assignment	B	B'	β	10Dq	15B'	μ_{eff} B.M	$\mu_s \text{ cm}^{-1}$	Suggested structure
CoL	$\nu_1=3394$ $\nu_2=4993\text{cal}$ $\nu_3=15.582$	${}^4A_2 \rightarrow {}^4T_{2(F)}$ ${}^4A_2 \rightarrow {}^4T_{1(F)}$ ${}^4A_2 \rightarrow {}^4T_{1(P)}$	1120	693	0.61	3467	4995	4.73	20	Tetrahedral
NiL	$\nu_1=10.080$ 10.098cal $\nu_2=14.750$ $\nu_3=24.096$	${}^3A_{1g} \rightarrow {}^3T_{2g(F)}$ ${}^3A_{2g} \rightarrow {}^3T_{1g(F)}$ ${}^3A_{1g} \rightarrow {}^3T_{1g(P)}$	1035	570	0.55	10262	10098	2.70	23	Octahedral
CuL	$\nu_1=12.901$	${}^2B_{2g} \rightarrow {}^2B_{1g}$ ${}^2B_{2g} \rightarrow {}^2E_g$	-	-	-	-	-	1.02	21	Dimeric octahedral



Electronic spectrum of L



Electronic spectrum of CoL

4-Biological activity :

As a result from the study of antimicrobial for ligand and its complexes, table (4), the following points have been concluded :

1- The results of antibacterial activity study for the trithiocarbonate derivative (L) showed that the new compound is not active against the bacteria under test at low and high concentrations .

2- Generally, the results showed that the copper complex has a highly significant activity toward *Pseudomonas aeruginosa* but no inhibition toward *Staphylococcus aureus*, in both concentrations.

3- The cobalt complex showed a high antibacterial activity toward G⁺ve and G⁻ve bacteria in case of high concentration more than in low concentration, while the nickel complex showed a high activity toward G⁻ve bacteria in low concentration.

The biological activity of the metal complexes was governed by the following factors: (i) the chelate effect of the ligand, (ii) the nature of the donor atoms, (iii) the total charge on the complex ion, (iv) the nature of the metal ion, and (v) the geometrical structure of the complex. Furthermore, chelation reduces the polarity of the metal ion because of partial sharing of its positive charge with the donor groups and possibly the π -electron delocalization within the whole chelate ring system that is formed during coordination [30] . Accordingly, the antimicrobial activity of the complexes can be referred to the increase of their lipophilic character. Also it is proposed that the action of the toxicant is the denaturation of one or more proteins of the cell and this impairs normal cellular process.

Table(4): Antibacterial activity of the ligand and its metal complexes .

Compounds	<i>Pseudomonas aeruginosa</i>		<i>Staphylococcus aureus</i>	
	5mM	10mM	5mM	10mM
L	-	-	-	-
CoL	+	++	+	++
NiL	-	++	+	-
CuL	++	++	-	-

(-) inactive ; (+) moderate active ; (++) active

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تحضير وتشخيص ودراسة الفعالية البيولوجية لبعض معقدات الليكاند (بوتاسيوم (2-كاربوميثوكسي امينو- 5 ثلاثي ثايوكاربونيت- 4,3,1 ثايديازول)

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**وزارة العلوم والتكنولوجيا/بغداد

الخلاصة :

تم في هذا البحث تحضير الليكاند الجديد (بوتاسيوم-2-كاربوميثوكسي امينو- 5 ثلاثي ثايوكاربونيت-4,3,1 ثايديازول) (L) من مفاعلة (2-كاربوميثوكسي امينو-5-مركبتو-4,3,1 ثايديازول) مع ثنائي كبريتيد الكربون بوجود هيدروكسيد البوتاسيوم . تم عزل الليكاند المحضر وتشخيصه باستخدام الطرائق الفيزيائية المناسبة، الطيفية والالكترونية بالإضافة الى درجة الانصهار استخدم الليكاند لتحضير عدد من المعقدات الجديدة مع بعض ايونات العناصر الانتقالية (Co(II), Cu(II) و Ni(II) ، اذ تم تشخيص ودراسة تراكيب المعقدات الجديدة في الحالة الصلبة باستخدام الاشعة تحت الحمراء وفوق البنفسجية-المرئية وقياسات التوصيلية النوعية فضلاً عن الحساسية المغناطيسية وتقنية التحليل الكمي للعناصر وذلك باستخدام تقنية الامتصاص الذري اللهب . أعطى تفاعل الليكاند(L) مع ايونات النحاس(II) والنيكل(II) معقدات شكلها الهندسي ثماني السطوح ، كما أعطى معقد الكوبلت الناتج من تفاعل الليكاند مع ايون الكوبلت(II) معقداً شكله هندسي رباعي السطوح . فسرت طبيعة التآصر بين الفلز والذرات المانحة في الليكاند ودرجة التشوه كما تم احتساب معامل راکاح والمجال الليكاندي باستخدام مخططات تاناها-سوكانو المناسبة تم تقييم الفعالية البيولوجية لجميع المركبات المحضرة ضد نوعين من البكتريا *Staphylococcus aureus* و *Pseudomonas earuginsa* باستخدام تركيزين (10 و 5 ملي مولاري) في وسط زرع متعادل وقد اظهرت النتائج زيادة في فعالية بعض المعقدات المحضرة بالمقارنة مع الليكاند وسبب الفعالية المضادة للبكتريا هو حصول التأثير التآزري بين الفلز والليكاند فضلاً عن الاختلاف في تركيب المركبات المحضرة .