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Preparation and characterization and biological activity of new complexes for some transition elements with Schiff bases derived from one antipyrine substitute

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RESEARCH ARTICLE

Preparation and Characterization and Biological Activity of New Complexes for Some Transition Elements with Schiff Bases Derived from one Antipyrine Substitute

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ABSTRACT

A group of divalent complexes was prepared in the formula $[M(L)Cl_2]$ when M = Co^{+2}, Ni^{+2}, Cu^{+2}, Zn^{+2}, and Cd^{+2} L = (Z)-4-((1-(H-indol-3-yl)ethylidene)amino)1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one. The Schiff base ligand is prepared by reacting 4-aminoantipyrine with 3-acetylindol. The prepared ligand and complexes were identified by elemental analysis, an infrared spectrometer, the nuclear magnetic resonance spectrum, molar electrical conductivity, electronic spectra, magnetic susceptibility, atomic absorption of the elements, and biological efficiencies. The measurements showed that the synthesized ligand interacts with the central metal ion through the oxygen carbonyl group and nitrogen azomethine atoms, that the coordination of the prepared complexes has a square planar nickel complex and the tetrahedral geometry of other complexes, and that these complexes are non-electrolytic.The synthesized compounds have good antibacterial activity against Gram-negative bacteria, represented by *Pseudomonas savastanoi*, and Gram-positive bacteria, represented by *Bacillus subtilis*.

Keywords: 4-aminoantipyrin substitute, Biological activity, Schiff base ligand, Square planar complexes, Transition metals

Introduction

Schiff bases are chemical substances that are typically created by the condensation of an aldehyde or ketone with a primary amine and typically occur under acid, base, or heat catalysis. [1,2] 4-Aminoantipyrine (AAP), a pyrazolone derivative, has drawn a lot of interest from medicinal chemists since it has so many potential uses. pharmacological effects such as analgesic, anti-inflammatory, antiviral, and antibacterial. Two nitrogen atoms, the carbonyl and amine groups, which are present in the cyclic structure of AAP, have a significant impact on its biological action. As donor ligands, the carbonyl and amine groups of AAP help to create different coordination assemblies and chelate structures. The study of AAP derivative complexes is fascinating since the development of the organic-metal chelate results in significant changes to the compound's biological characteristics. [3–6] The novel complexes with L ligands were synthesized in this research and investigated using a variety of spectroscopic methods. These complexes have been prepared by the reaction of many transition metal salts with Schiff bases. Mononuclear complexes with different stoichiometry and structure were characterized. This type of produced complex was found depending on the reaction medium. Moreover, the complexes antibacterial effectiveness was investigated using Hinton Mueller agar medium. [7,8] In this work, we aim to prepare

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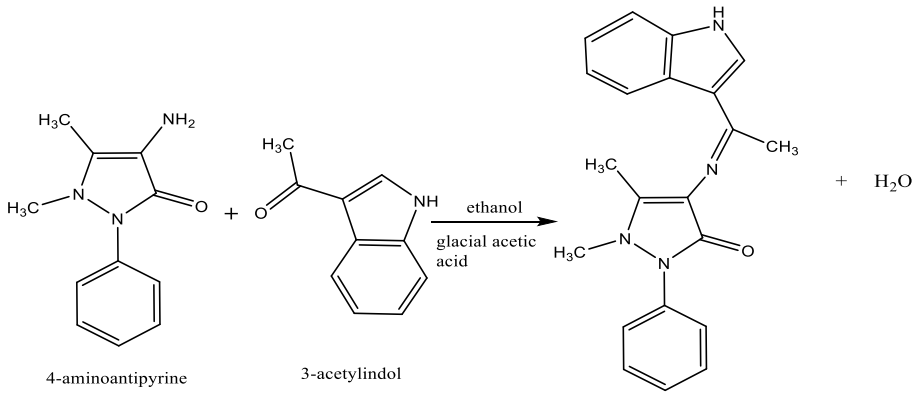

Fig. 1. Synthesis of Schiff base ligand (L).

some transition metal complexes (II) with Schiff base ligands and characterize them to show probable applications for these complexes in the future.

Materials and methods

The Fluka or Merck companies supplied all of the reagent-grade chemicals utilized in this experiment. Without any further purification, they were used totally as they were. Using the Stuart-SMP30 melting point apparatus, the melting point temperature was calculated. The atomic absorption spectrophotometer of type Shimadzu used spectral methods to determine the elemental contents. Lovibond Con200 was used to test the molar conductance in DMF (10^{-3} M) at 25 °C. Using KBr pellets at 400 to 4000 cm^{-1}, Shimadzu FTIR was used to record the IR spectra of the ligand and its complexes. UV/visible spectra measurements were made by the T80 UV/VIS Spectrometer. Magnetic susceptibility measurements were made by the magnetic susceptibility balance of Sherwood Scientific, Cambridge, UK. Using deuterated DMSO as a solvent, the ^1H NMR was captured at 400 MHz on the BioSpin Gmbh shield. The agar and bacteria were supplied by the department of biology at the College of Science at the University of Mosul.

Synthesis of ligand (L)

In 30 mL of hot ethanol, dissolve 4-aminoantipyrine (6.09 g, 0.03 mol) and a hot solution of 3-acetylindol (4.77 g, 0.03 mol) and mix. Glacial acetic acid was then added in small amounts to the mixture and put under reflux for 3 hours. The resulting solid was purified by recrystallization, filtered, washed with ether, and then dried.[9,10] The orange Schiff base product yield: 85%, m.p. 68–70 °C, Anal. Calc. for $C_{21}H_{20}N_4O$: C; 73.25; H; 5.81; N; 16.27; found: C; 73.02; H; 5.68; N; 16.07%. The synthesis of Schiff base ligand (L) is shown in Fig. 1.

Complexes' synthesis

The following general process[11,12] was used to prepare each complex: Schiff base ligand (L) (1.72 g, 0.005 mol), dissolved in ethanol, was added to a heated solution of metal(II)chloride ($CoCl_2.6H_2O$ (1.19 g, 0.005 mol), $NiCl_2.6H_2O$ (1.18 g, 0.005 mol), $CuCl_2.2H_2O$ (0.85, 0.005 mol), $ZnCl_2$) 0.68, 0.005 mol) and $CdCl_2$ (0.91, 0.005 mol)) in a mole ratio of 1:1 L:M. For six hours, the mixture was refluxed, and as the complexes cooled, they were filtered out, washed with ethanol and ether, and then dried.

Results and discussion

The metal (II) complexes in this investigation were soluble in DMF and other solvents but insoluble in water and stable in air. Various spectroscopic techniques revealed the structure of the produced compound. Elemental analysis (C.H.N.), as well as melting points, supported the predicted molecular composition of the compound. The molar conductance of the prepared complexes was measured at a concentration of 10^{-3} M using DMF solvent, which gave a conductivity ranging between 12–22 $Ohm.cm^2.mol^{-1}$ which is consistent with non-electrolyte complexes.[13] Characterization, analytical, and molar conductance data of the complexes listed in Table 1.

Infrared spectra

The spectra of the ligand showed the recognizable bands at 1620 cm^{-1}, 1730 cm^{-1}, 3163 cm^{-1}, 2927 cm^{-1}, and 3116 cm^{-1}, and the bands were referred to as υ C=N azomethine, υ C=O carbonyl,

Table 1. Data on the characteristics, analysis, and molar conductance of complexes.

no.	(Formula)	(Color)	Yield%	(m.p) (°C)	(Λ^M Ohm^{-1}. cm^2. mol^{-1})	(calculate (found)%)			
						(C%)	(H%)	(N%)	(M%)
1	$[Co(L)Cl_2]$	Gray	67	179–181	12	53.18 (53.07)	4.22 (4.12)	11.81 (11.54)	12.43 (12.19)
2	$[Ni(L)Cl_2]$	Brownish Yellow	94	159–160	17	53.14 (52.68)	4.22 (4.10)	11.82 (11.54)	12.39 (12.12)
3	$[Cu(L)Cl_2]$	Brown	86	112–113	22	52.67 (52.43)	4.18 (3.89)	11.70 (11.57)	13.28 (13.12)
4	$[Zn(L)Cl_2]$	Red	64	117–119	18	52.47 (52.26)	4.16 (3.86)	11.66 (11.31)	13.61 (13.38)
5	$[Cd(L)Cl_2]$	Gray	90	166–168	30	47.79 (47.26)	3.79 (3.41)	10.62 (10.30)	21.31 (21.11)

Table 2. Ligand and their complexes' selected IR bands cm^{-1}.

Compound no.	υ(N-H)	δ(N-H)	υ(C = N)	υ(C = O)	υ(N-N)	υ(M-N)	υ(M-O)
L	3163	1238	1620	1730	1016	—	—
1	3166	1240	1571	1614	1020	509	556
2	3168	1240	1596	1616	1020	510	565
3	3165	1240	1523	1618	1022	459	555
4	3161	1241	1521	1616	1021	459	509
5	3167	1240	1520	1616	1020	455	557

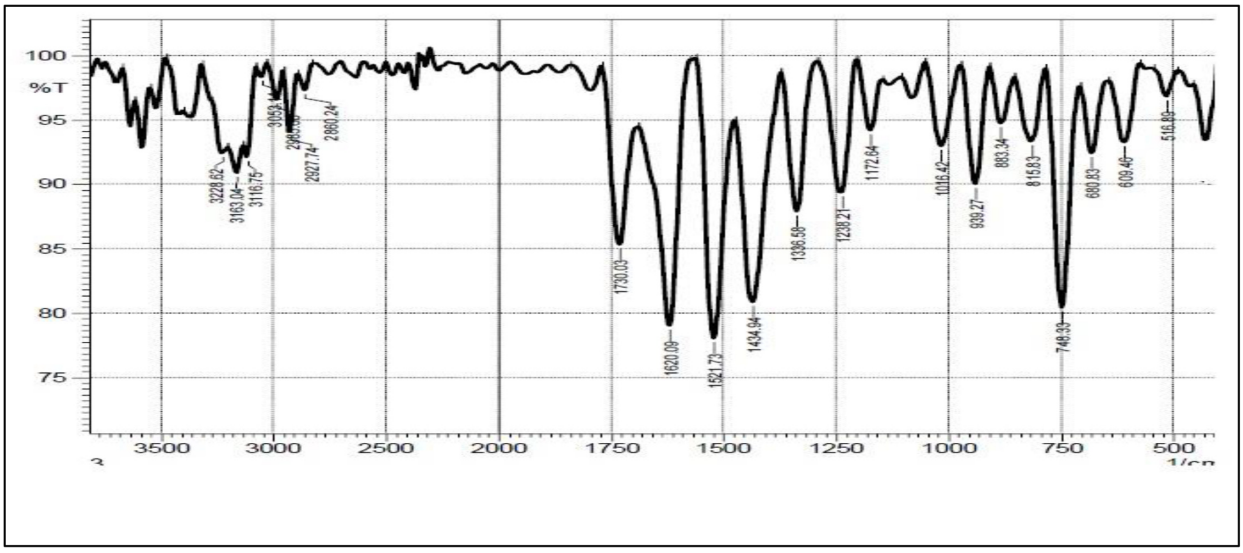

Fig. 2. FTIR spectra of the ligand (L).

υ N-H, υ C-H aliphatic, and υ C-H aromatic, respectively.[14,15] In all the metal complexes, the stretching frequencies of the azomethine and carbonyl groups shifted downward, which indicated that the N and O atoms were involved in coordination. The spectra of IR of the complexes referred to new bands in 510–455 cm^{-1}, 565–509 cm^{-1} regions, which can be assigned to υ (M–N) and υ (M–O) vibrations.[16–19] Ligand and their complexes' selected IR bands cm^{-1} listed in Table 2 and Fig. 2 show the FTIR spectra of the ligand (L).

Electronic spectra and magnetic moment measurement

The transitions $\pi \rightarrow \pi^*$ and n $\rightarrow \pi^*$ are attributed to the bands in the 37750, 33444 cm^{-1} range of the ligand's electronic spectra.[20] The Co(II) d^7 complex's electronic spectra exhibit absorption bands at 17182 cm^{-1} due to (υ_3), which is attributed to the transition $^4A_2$ (F) $\rightarrow$ $^4T_1$(P) and 4.46 B.M. practical magnetic moment values of the cobalt complex, which is higher than the theoretical value of 3.82 B.M. due to the orbital contribution.[21] The nickel (II) d^8 complex has electronic spectrum bands that are ascribed to transitions $^1A_1g \rightarrow {}^1A_2g$ (υ_1) and $^1A_1g \rightarrow {}^1B_1g$ (υ_2), 20618 cm^{-1} and 24213 cm^{-1}, respectively, and a dia magnetic moment that was measured.[22] In Cu(II) d^9 complexes, the band at 15290 cm^{-1} is due to $^2T_2$ (D) $\rightarrow$ ^2E (P), as well as the practical magnetic moment of the Cu(II) complex of 2.12 B.M., which is higher than the theoretical value of 1.73 B.M. due to the second-order orbital contribution.[23] These results were compatible with

Table 3. Electronic transition and magnetic moment for metal complexes and ligand.

Number	Compounds	Λmax (nm)	Band (cm^{-1})	Assignments	magnetic moment	Structures
	ligand (L)	265	37750	$\pi \rightarrow \pi^*$	–	–
		299	33444	$n \rightarrow \pi^*$		
1	$[Co(L)Cl_2]$	582	17182	$^4A_2$ (F) $\rightarrow$ $^4T_4$(P)	4.46	Tetrahedral
		339	29498	C.T		
2	$[Ni(L)Cl^2]$	485	20618	$^1A_1g \rightarrow {}^1A_2g$	Dia	Squer planar
		413	24213	$^1A_1g \rightarrow {}^1B_1g$		
		340	29411	C.T		
3	$[Cu(L)Cl_2]$	654	15290	$^2T_2 \rightarrow {}^2E$	2.12	Tetrahedral
		329	30395	C.T		
4	$[Zn(L)Cl_2]$	305	32786	C.T	–	Tetrahedral
5	$[Cd(L)Cl_2]$	303	33033	C.T	–	Tetrahedral

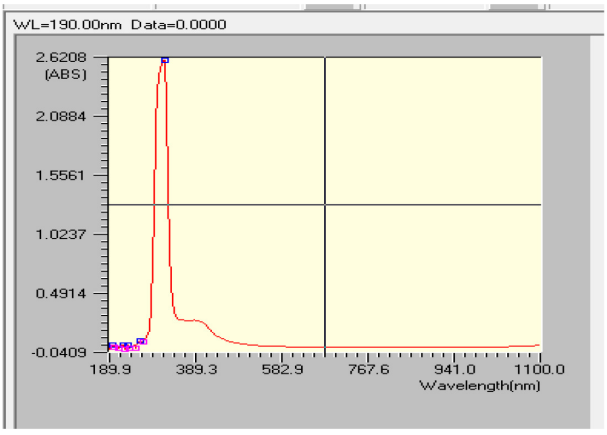

Fig. 3. Electronic spectrum of ligand (L).

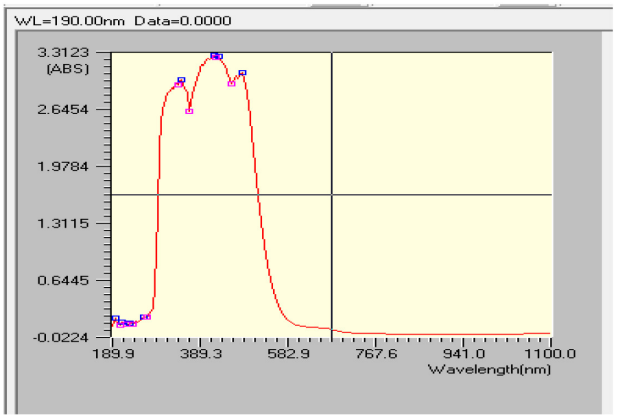

Fig. 4. Electronic spectrum of [Ni(L) Cl_2].

the tetrahedral structure of the Co(II) and Cu(II) ions, while the Ni(II) complex supports the square-planar structure. The magnetic moment measurements of the zinc (II) d^{10} and cadmium (II) d^{10} complexes showed that they are diamagnetic, and the peaks in the electronic spectra are attributed to charge transfer transitions and suggest that the metal ions are surrounded by a tetrahedral structure.[24,25] Table 3 shows the electronic transition and magnetic data of the ligand and complexes, and Figs. 3 and 4 show the UV spectra of the ligand and complex $[Ni(L)Cl_2]$, respectively.

^{1}H-NMR spectra

The ^{1}H-NMR spectrum of the prepared ligand (L) Fig. 6 gave the following signals: -NH at 11.9 ppm, -N = C-CH_3 at 2.13 ppm, =C-CH_3 at 2.74 ppm, -N-N-CH_3 at 3.84 ppm, 7.1–8.3 ppm due to the aromatic proton, These signs did not change in the prepared complexes, which confirms their steric structure for the prepared compound.[26–28] ^{1}H-NMR spectrum of the ligand (L) is shown in Fig. 5.

Biological activity of the prepared compounds

The bacteriological activity of the prepared ligand and its metal complexes was conducted on two types of bacteria that are pathogenic to humans. The first type was Gram-negative bacteria, represented by *Pseudomonas savastanoi*, and the second type was Gram-positive bacteria, represented by *Bacillus subtilis*. The solutions in our study were prepared at a concentration of 10 mg/ml dissolved in the solvent (DMSO), and 100 microliters were placed inside the holes in the culture medium. The dishes were closed with a special tape, and the dishes were placed inside the incubator for 24 hours at a temperature of 37 °C, after which a ruler was used to measure the diameter of inhibition to show that the sensitivity of the studied compounds depending on the diameter of the inhibition. One type of antibiotic (Ciprodar) was used. The prepared ligand showed higher effectiveness against both types of bacteria than the prepared complexes. The reason for this is the presence of alkyl groups and donor atoms within the structure of the active ligand, which led to the formation of complexes with the elements present inside the cell that the bacterial cell needs, such as cobalt and copper, and the loss of these elements leads to cell death. The prepared complexes also showed good biological activity, and the reason for this may be attributed to the coordination of surrounding the cell wall that contains a high percentage of fats the ligand with the central metal ion through the nitrogen and oxygen donor atoms. Bacterial cells

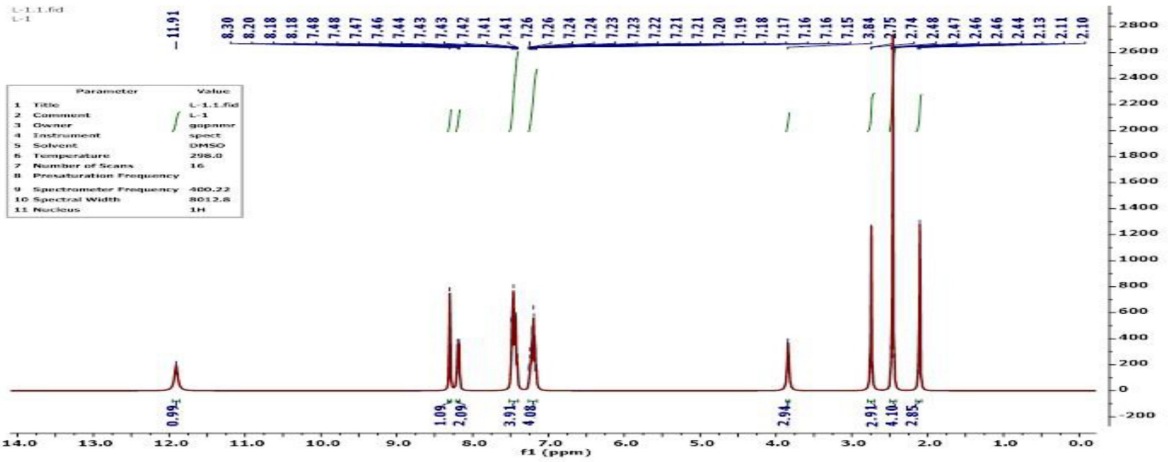

Fig. 5. ^{1}H-NMR Spectrum of the ligand (L).

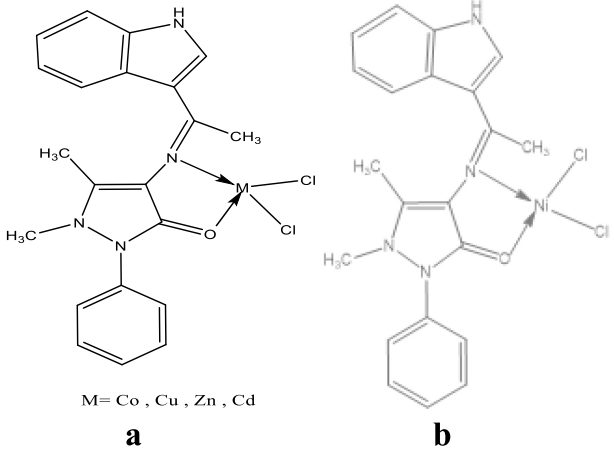

Fig. 6. The structure of the prepared complexes a $[M(L)Cl_2]$ b $[Ni(L)Cl_2]$.

Table 4. Antibacterial activity of the ligands and their complexes (10 μg/1 ml).

prepared compound	*Bacillus subtilis*	*Pseudomonas savastanoi*
Control	20	12
L	14	15
$[Co(L)Cl_2]$	10	11
$[Ni(L)Cl_2]$	8	10
$[Cu(L)Cl_2]$	13	14
$[Zn(L)Cl_2]$	10	12
$[Cd(L)Cl_2]$	9	10

are resistant to some chemical compounds because of the presence of a thick membrane.[29–31] The antibacterial activity of the ligands and their complexes (10 μg/1 ml) is shown in Table 4 and Figs. 7 and 8.

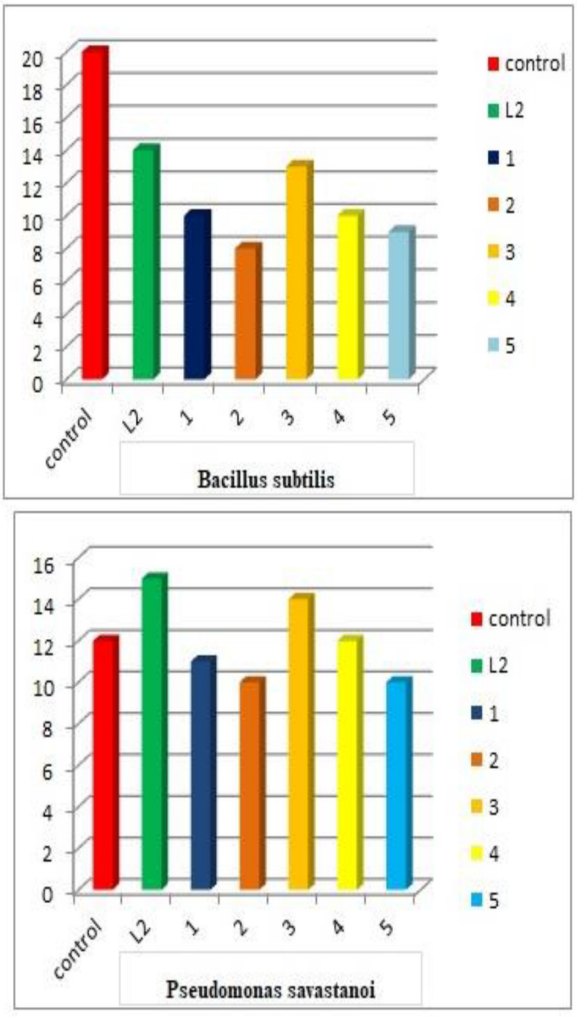

Fig. 7. Differences in biological activity against bacteria for ligand and complexes.

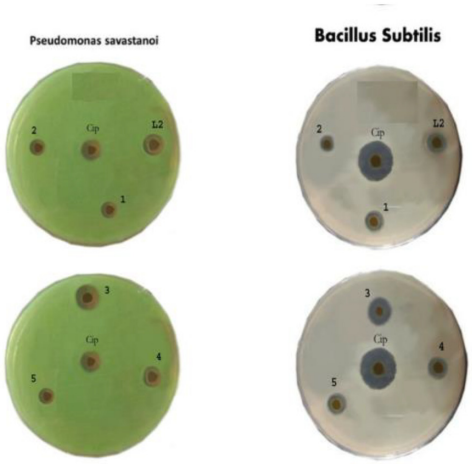

Fig. 8. Inhibitory region of ligands and complexes against bacteria.

Conclusion

Through several physicochemical, Electronic Spectra, Infrared Spectra, ^{1}H-NMR spectra, and magnetic susceptibilities measurements, it was found that the ligand is bidentate and it is linked through the nitrogen and oxygen donor atoms to the central metal ion and completes the coordination number with a chlorine ion to become the tetrahedral geometry of the cobalt(II), copper(II), zinc(II) and cadmium (II) complexes, while the nickel(II) complex is a square planar geometry. The prepared ligand showed higher activity than the complexes against the two types of bacteria used in this research, which may be due to its ligand structure containing azomethine and carbonyl groups and its ability to coordinate with metal ions.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Mosul.

Authors' contribution statement

The role of first authors Z.U.J. Prepared ligands and complexes, did IR test and data analysis, The role of second authors A. M. A. (UV visible and ^{1}H NMR tests, measure molarity conductivity and study biological activity) for the complexes, the role of the third author H. A. M. writing the manuscript, drafting and designing the Ms and the forth author M.A. M. Conception, analysis, revision and proofreading, acquisition of data, data analysis.

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تحضير وتشخيص ودراسة الفعالية البايولوجية لمعقدات جديدة لبعض العناصر الانتقالية مع قواعد شيف مشتقة من احدى معوضات الأنتي بايرين

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الخلاصة

حضرت مجموعة من المعقدات ذات الصيغة $[M(L)Cl_2]$ حيث $M= Co^{+2},Ni^{+2},Cu^{+2},Zn^{+2}, Cd^{+2}$
L= (Z)-4-((1-(H-indol-3-yl)ethylidene)amino)1,5-dimethyl-2-phenyl-1,2-dihydro-3H-pyrazol-3-one.
من ليكاند قاعدة شيف مشتق من المركب 4-امينو انتبيرين مع 3-استايل اندول كما تم تشخيص الليكاند والمعقدات المحضرة بالتحليل الدقيق للعناصر واطياف الاشعة تحت الحمراء وطيف الرنين النووي المغناطيسي وقياسات الحساسية المغناطيسية وتحليل المحتوى الفلزي بطريقة الامتصاص الذري فضلا عن دراسة الفعالية البايولوجية للمركبات المحضرة. اثبتت القياسات المستخدمة ان الليكاند المحضر يتناسق مع ايون الفلز المركزي من خلال ذرة اوكسجين مجموعة الكاربونيل وذرة نتروجين مجموعة الازوميثين ويتخذ معقد النيكل المحضر الشكل الفراغي مربع مستوي اما بقية المعقدات فتتخذ الشكل الفراغي رباعي السطوح ،كما تبين ان المعقدات المحضرة غير موصلة الكتروليتيا واثبتت المركبات المحضرة فعالية عالية كمضاد بكتريا لبعض أنواع بكتريا كرام الموجبة والسالبة.

الكلمات المفتاحية: معوضات 4-امينو انتي بايرين، الفعالية البايولوجية،ليكاند قاعدة شيف، معقدات المربع المستوي، الفلزات الانتقالية.