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PREPARATION, CHARACTERIZATION, AND STUDY OF THE BIOLOGICAL ACTIVITY OF 5-CHLORO-8-QUINOLINOL DERIVATIVES AND ITS COORDINATION WITH THE NICKEL (II) AND DIPHOSPHINES

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Abstract: Nickel complexes are effective catalysts for cross-coupling reactions between alkyl Grignard reagents and alkenyl-S and alkenyl-Se compounds. In this study, various organic compounds were prepared. Ethylanyl 2-((5-chloro-8-quinolinol)oxy) acetate compound (designated as A1) was synthesized by adding potassium carbonate to quinoline and ethyl chloroacetate. The second compound 2-((5-chloro-8-quinolinol)oxy) acetohydrazide (A2) were obtained by reacting A1 with hydrazide. Complex compound A3 were obtained by reacting equimolar amounts of compound A2 with the nickel salt solution, using ethanol as the solvent. Phosphinate complexes were prepared by reacting equal moles of the A3 with the various phosphines and using ethanol as a solvent. The synthesized compounds and complexes were characterized using various spectroscopic techniques, including (FT-IR) spectroscopy, (^{31}P - $\{^1\text{H}\}$ - ^{13}C -NMR) spectroscopy, and C.H.N. analysis. Additionally, their melting points, purity, molar conductivity, and magnetic susceptibility were determined. The impact of some prepared compounds and complexes on the growth of two antibiotic-resistant bacterial strains, namely the Gram-negative *Staphylococcus* and the Gram-positive *Escherichia coli*, was studied. Amoxicillin was used as control antibiotics. Some of the synthesized compounds exhibited significant inhibitory activity against the tested bacterial strains. The A2 compound readily forms complexes, especially with nickel, manganese, and copper salts. The prepared compounds and complexes exhibited high stability and strength, maintaining their structure, color, and melting point even under varying laboratory temperatures between winter and summer.

Keywords: 5-chloro-8-quinolinol, Ester, Nickel, Diphosphine, Complexes, Biological Activity.

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1. Introduction

Nickel complexes play a pivotal role in the realm of coordination chemistry, exhibiting diverse structural and electronic properties that contribute to their significance in various biological processes [1]. Nickel, with its unique electronic configuration and versatile coordination preferences, forms complexes that serve as essential cofactors in numerous enzymes crucial for biological functions [2]. Nickel's role in metalloenzymes, where it acts as a catalytic center to support essential physiological processes and facilitate redox reactions, is a well-known illustration of its biological significance [3].

Urease is a prime example of a nickel-containing enzyme that has been well-investigated [4]. Because it catalyzes the breakdown of urea into ammonia and carbon dioxide, urease, a metalloenzyme found in many different organisms, is essential to the metabolism of nitrogen [5]. This enzyme activity has broad consequences for cellular homeostasis and is essential for the recycling of nitrogen in living systems [6]. Nickel complexes are key parts of other enzymes that participate in vital metabolic pathways like methane production and hydrogen metabolism, such as certain hydrogenases and methyl-coenzyme M

reductases [7]. Because of their varied reactivity, nickel-based catalysts have an impact on synthetic applications and may be used to create new medications and bioinspired materials [8].

Deciphering the mechanisms underlying these physiologically relevant events requires an understanding of the complicated coordination chemistry of nickel complexes [9]. This information not only broadens our

understanding of basic biochemical processes, but also serves as inspiration for the development of artificial catalysts and medicinal substances [10]. Therefore, the study of nickel complexes and their biological significance lies at the nexus of biology and chemistry, providing a rich field of inquiry with significant implications for both fundamental and applied research [11].

2. Experimental part

2.1. Materials Employed: All chemicals utilized were procured from Fluka, Aldrich Companies.

2.2 Used Instruments

Infrared spectra for the produced compounds were recorded using an FTIR-8400S instrument from SHIMADZU at the Chemistry Department of the University of Tikrit's College of Sciences. KBr pellets were used to record the spectra, which fell between 400 and 4000 cm^{-1} (^1H , ^{13}C -NMR) at the University of Tehran in Iran, nuclear magnetic resonance spectra were recorded using a Bruker Spectrometer (500 MHz), in d_6 -DMSO solvent. The melting points of synthetic substances were ascertained using an Automatic Melting Point device (model SMP10) from the British business STUART. Magnetic susceptibility measurements of specific solid metal complexes were performed at laboratory temperature using a Sherwood Scientific device. These measurements were made using the Faraday method.

2.3. Synthesis of ethyl 2-((5-chloro-8-quinolinol)oxy)acetate (1:1) A1⁽¹¹⁾

The 5-chloro-8-quinolinol (0.01 mole, 1.79g) was dissolved in 15 mL of dimethylformamide. Potassium carbonate (0.01 mole, 1.38g) was added, and the mixture was agitated for 15 minutes. The mixture was then mixed with 0.01 mole of ethyl chloroacetate (1.07mL). The reaction mixture was agitated between 40 and 50°C for four hours. Following the solution's cooling, 50 mL of distilled water was added while stirring constantly. After filtering the resultant precipitate, 100% ethanol was used to recrystallize the crystals. With an 89% yield, the resultant product was a white

solid with a melting point between 90 and 96°C.

2.4. Synthesis of Compound 2-((5-chloro-8-quinolinol)oxy)acetohydrazide A2⁽¹²⁾

Following the dissolution of compound A1 (0.01 mol, 2.65 g) in ethanol (20 mL), hydrazine (0.02 mol, 1 mL) was added dropwise while stirring for 15 minutes. After being permitted to stand for 24 hours, the reaction mixture was allowed to react for 4 hours at a temperature between 70 and 80 °C. After cooling the mixture, the precipitate was filtered, dried, and crystallized again using pure ethanol. Thin-Layer Chromatography was used to measure and validate the completion of the reaction. With an 82% yield, a white solid with a melting point between 212 and 214 °C was the result.

2.5. Synthesis of di aqua 2-((5-chloro-8-quinolinol)oxy)acetohydrazide Nickel (II) A3 Compound A2 (0.002 mol, 1.24g) was dissolved in methanol (15 mL), and an aqueous solution of the Nickel (II) chloride hydrate (0.002 mol) was added. The mixture was stirred for 2 hours at a temperature range of 70-80 °C. After cooling, the precipitate was filtered, dried, and recrystallized using absolute ethanol. The progress of the reaction was confirmed by monitoring physical properties such as color and melting point. Table (1) displays the physical properties of the prepared complexes.

2.6. Synthesis of Ni(II) complexes with diphosphines (A4-A6)

After dissolving compound A3 (0.002 mole) in 15 mL of methanol, the DiPhosphines (dppm, dppe, dppp) (0.002 mole) were added to an aqueous solution. At a temperature of between 70 and 80 °C, the mixture was agitated for two hours. Following cooling, 100% ethanol

was used to filter, dry, and recrystallize the precipitate. Monitoring physical characteristics like color and melting point allowed for the confirmation of the reaction's progress. The prepared compounds' physicochemical characteristics are shown in Table (1).

Table 1. Some physical properties, connectivity and magnetic measurements of complexes (A3-A6).

NO.	Complex	Yield	M.P C°	Color	Cond.Am ($\Omega^{-1} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$)	μ_{eff} (B.M)	C.H.N.		
							Calculated\ (Found)		
							C	H	N
A3	[Ni(A2).2H ₂ O]	79%	230-232	Light blue	1.56	1.851	---	---	---
A4	[Ni(A3)dppm]	82%	226-228	Blue	5.66	1.93	43.40 (44.29)	3.74 (4.03)	12.36 (12.91)
A5	[Ni(A3)dppe]	79%	286-288	Yellow	5.44	1.89	44.12 (44.29)	3.90 (4.03)	12.88 (12.91)
A6	[Ni(A3)dppp]	75%	285-287	Green	6.75	1.851	43.97 (44.29)	3.78 (4.03)	12.38 (12.91)

2.7. Measurement of Biological Activity

Evaluation of biological activity was done using the Agar-well diffusion method. This required using a cotton swab to inoculate the bacterial cultures throughout the whole growing medium. Next, using a sterile 6 mm diameter piercing tool, wells were made in the agar medium. Then, 100 microliters of each drug were added to these wells on different culture plates, each of which contained a different strain of bacteria. This procedure was repeated for

each of the created solutions, taking into account the targeted bacterial strains and concentrations that were being examined. Two different types of bacteria were used to evaluate the antibacterial activity: gram-positive *Staphylococcus* and gram-negative *Escherichia coli*. Both bacterial species were first re-cultivated and then incubated for 18–24 hours at 37°C in a controlled laboratory setting to guarantee the test's efficacy.

3. Results and Discussion

Scheme (1) below shows compounds and complexes that were prepared in this study.

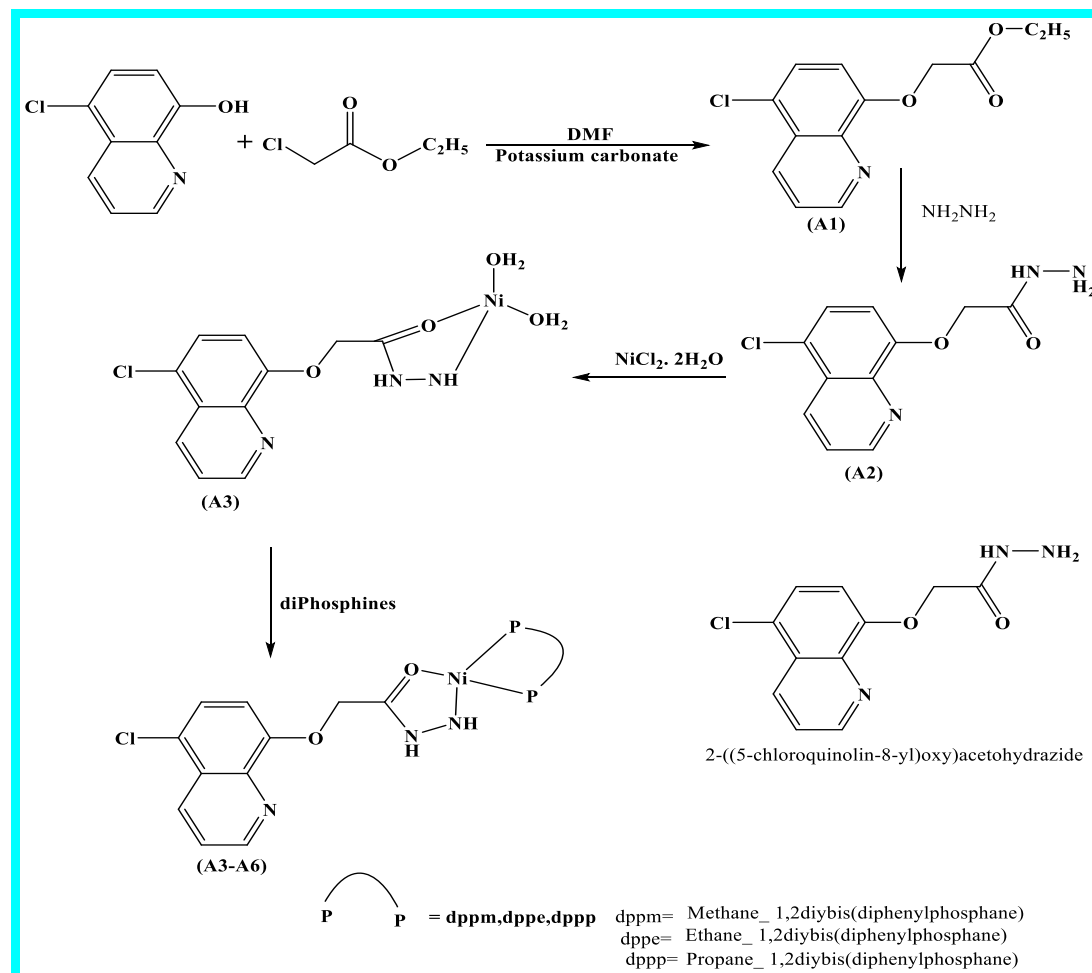
3.1. Characterization of Compounds A1-A6 by FT-IR Spectroscopy

The infrared spectra of various compounds and complexes are presented in this study, shedding light on their structural characteristics and interactions. Infrared spectroscopy, a powerful analytical technique, provides valuable insights into the chemical composition of these substances by identifying the specific vibrational modes of their constituent functional groups. Compound A1 (Figure 1) exhibits distinctive absorption bands in its infrared spectrum. A strong band at 1766 cm^{-1} corresponds to the carbonyl group (C=O), while two bands at 1589 and 1504 cm^{-1} are attributed to the aromatic C=C bonds. Another band at 1369 cm^{-1} is associated with the C-O group, and

a band at 786 cm^{-1} arises from the C-Cl group. Compound A2 (Figure 1) reveals distinct bands in its infrared spectrum. Bands at 3321 and 3232 cm^{-1} correspond to the NH₂ group, while a band at 3205 cm^{-1} indicates the presence of the NH group. The amide carbonyl group (C=O) is marked by a band at 1674 cm^{-1} , and two bands at 1620 and 1500 cm^{-1} relate to aromatic C=C bonds. Additionally, a band at 1365 cm^{-1} signifies the C-O-C group, and a band at 821 cm^{-1} suggests the presence of C-Cl bonds. Complex A3 displays changes in its infrared spectrum, reflecting interactions between the metal and specific functional groups. A decrease in frequency at (3305, 3220) cm^{-1} is indicative of NH₂ group-metal bonding, while a reduction in frequency at 1658 cm^{-1} points to metal-C=O amide bonding. Notably, a band at 3460 cm^{-1} corresponds to water molecules coordinated

with the metal, while other bands remain unchanged. When studying the infrared spectrum of phosphinate complexes, new bands were observed. Notably, a band appeared at a frequency of (408-416) cm^{-1} attributed to the

(Ni-P) bond, another band at a frequency of (1406-1409) cm^{-1} assigned to the (P-C) bond, and two bands at frequencies (3401-3409 and 3315-3325) cm^{-1} associated with the (NH_2) group.



Scheme 1. Route of prepared compounds A1, A2 and complexes (A3-A6).

Additionally, a band at (3160-3166) cm^{-1} was identified as originating from the (NH) group, a band at (1661-1667) cm^{-1} was linked to the (C=O) bond, and a band at (1591-1595) cm^{-1} was attributed to the (C=C) bond. Furthermore, a band at (1291-1300) cm^{-1} corresponded to the (C-O-C) bond, and the strong bands at 1431-1436 cm^{-1} , assigned to the $\nu(\text{C}_6\text{H}_5\text{-P})$ grouping. It is thought that this vibration arises by the deformation of the planarity of the phenyl ring bonded to a heavy atom (phosphorus) and a

band at (944-954) cm^{-1} was associated with the (Ni-O) bond. Additionally, a band at (314-320) cm^{-1} was indicative of the (Ni-N) bond, while a band at (715-739) cm^{-1} corresponded to the (C-Cl) bond. These bands closely aligned with the literature findings. These infrared spectra provide essential information about the composition and interactions of the compounds and complexes under investigation, contributing to a deeper understanding of their chemical properties and potential applications (Table 2).

Table 2. IR absorption results (cm^{-1}) for compounds and complexes (A1-A6).

No.	νNH_2	νNH	$\nu\text{C=O}$	$\nu\text{C=C}$ Arom.	$\nu(\text{P-Ph})$	$\nu(\text{COC})$	$\nu\text{Ni-O}$	$\nu\text{Ni-P}$	$\nu\text{Ni-N}$	$\nu\text{C-Cl}$
A1	---	---	1766	1589		1369	---	---	---	786
A2	3321; 3232	3205	1674	1620		1365	---	---	---	821
A3	3400; 3294	3170	1678	1589		1303	941	---	312	713

A4	3402; 3315	3160	1665	1591	1431	1291	944	416	314	739
A5	3409; 3319	3166	1661	1592	1438	1299	950	408	318	715
A6	3401; 3325	3160	1667	1595	1431	1300	954	411	320	724

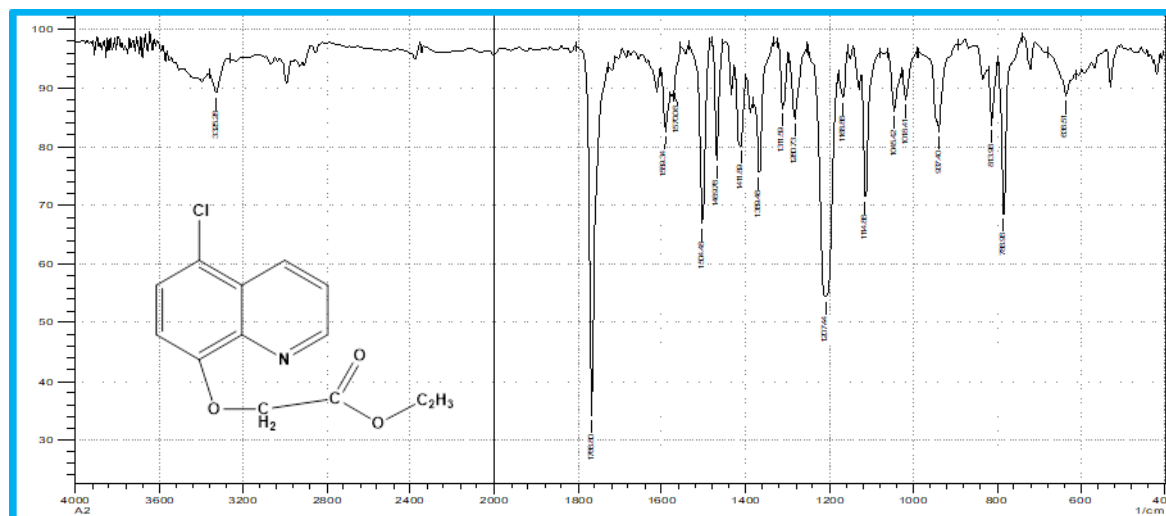


Figure 1. FT-IR spectrum of compound A1

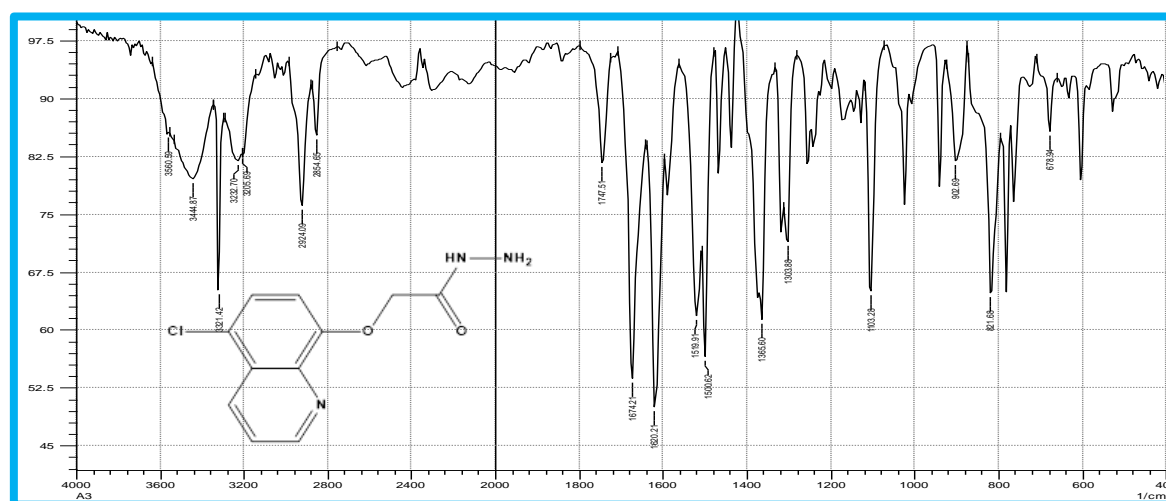


Figure 2. FT-IR spectrum of compound A2

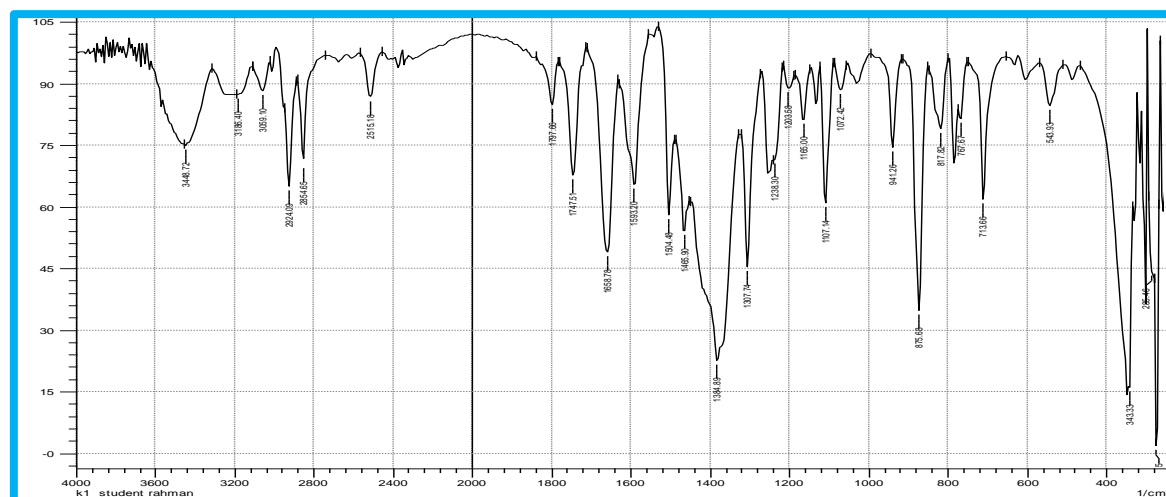


Figure 3. FT-IR spectrum of compound A3

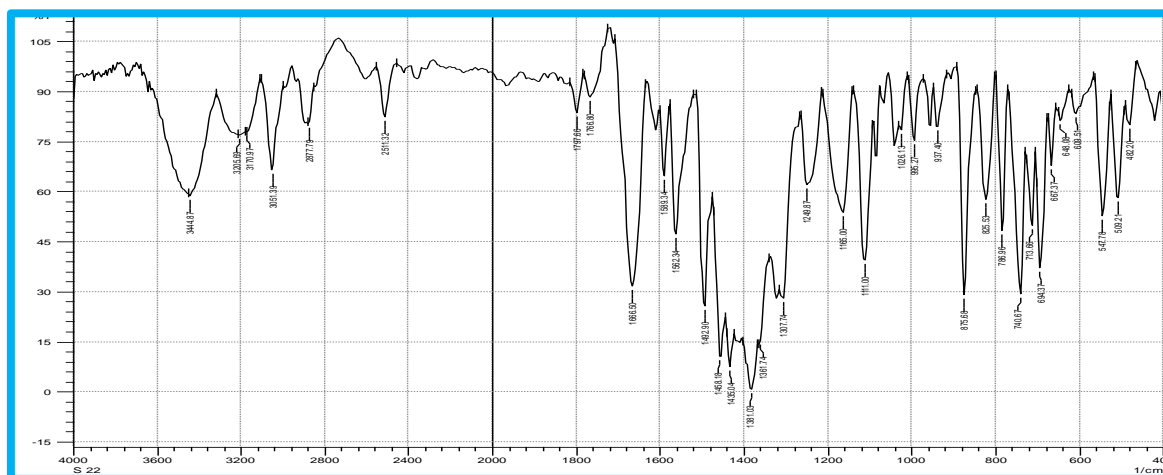


Figure 4. FT-IR spectrum of compound A5

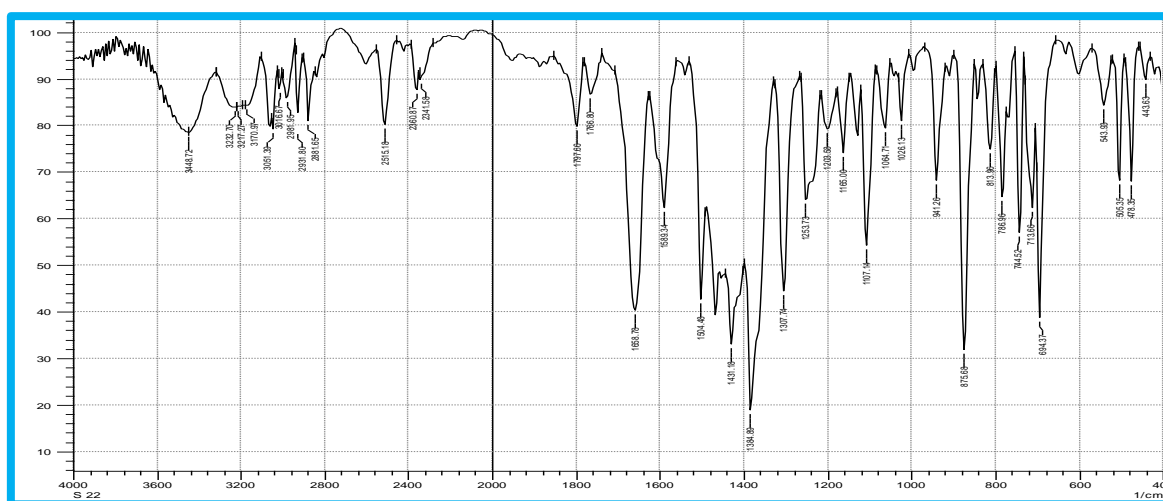
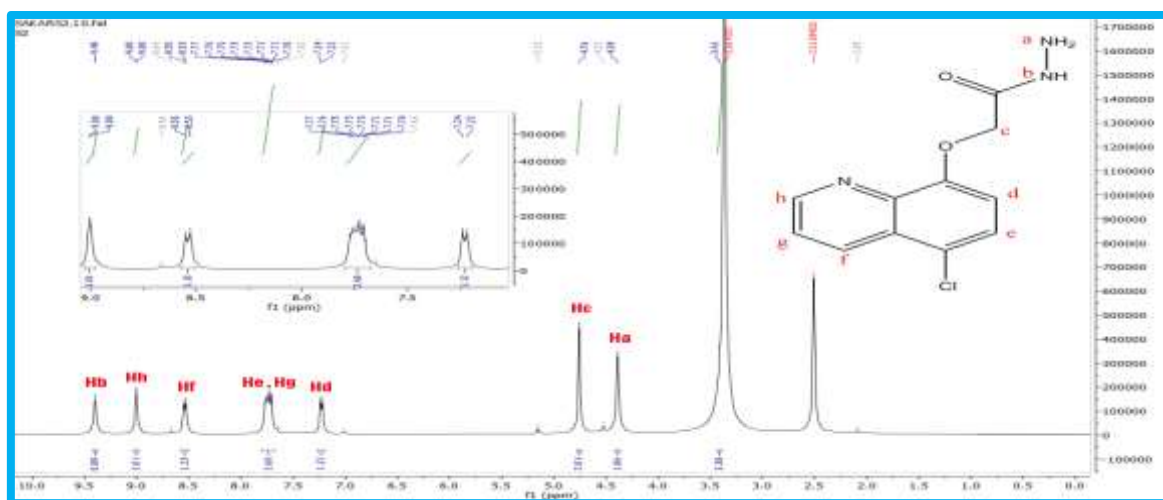


Figure 5. FT-IR spectrum of compound A6

Figure 6. The ^1H -NMR spectrum of A2

3.2. Characterization of Compound A2-A6 by ^1H , ^{13}C , ^{31}P -NMR Spectroscopy

Displays a singlet for two protons at $\delta=4.39$ ppm assigned to the NH_2 group labeled (Ha) (s, 2H). Another singlet showed at $\delta=4.39$ ppm assigned to the methylene protons, which

were labeled as Hc (s, 2H). Moreover, the protons of the aromatic rings display a doublet at $\delta=7.23$ ppm attributed to Hd with a coupling constant of ($^3J_{\text{H-H}}=8.40$ Hz). A multiple at δH (7.71) ppm with an integration value of two protons assigned to Hg and He respectively. The

Hf proton showed as a doublet at $\delta=8.54$ ppm with a coupling constant of ($^3J_{\text{HH}}=8.90\text{Hz}$). The integration value of Hf is one proton. Another doublet showed at $\delta=9.00$ ppm which represents Hh proton. Finally, the amide proton, which is labeled as (Hc) displays a singlet at $\delta=9.40$ ppm with an integration value of one proton (Figure 6).

The ^{13}C -NMR spectrum of A2 confirms the suggested structure. The spectrum showed the methylene carbon labeled Cb at $\delta=68.43$ ppm. The carbons of the aromatic rings appear

at $\delta= (111.78- 153.84)$ ppm which attributed to Cd, Cf, Cj, Cg, Ce, Ch, Ck, Ci and Cc. Finally, the carbonyl carbon which is labeled as Ca display at $\delta=166.98$ ppm (Figure 7).

When studying the ^{31}P -NMR spectrum for complex (A6), it was observed that a single signal appeared for phosphorus group at $\delta=29.96$ ppm returning to 1,3-Bis(diphenylphosphino)propane, and this confirms the validity of the prepared structures of the complexes (Figure 8) [12].

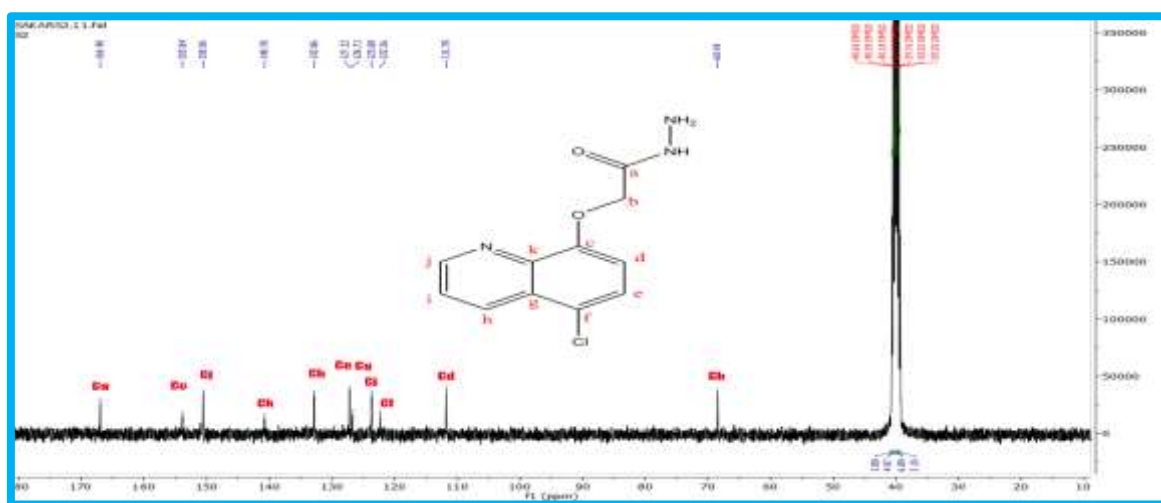


Figure 7. The ^{13}C -NMR spectrum of A2

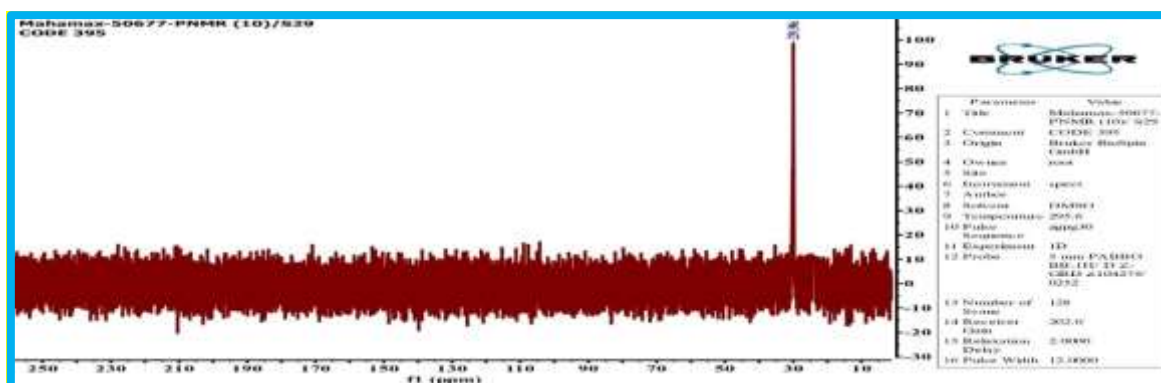


Figure 8. The ^{31}P -NMR spectrum of A6

Magnetic Measurements of Prepared Complexes (A3-A6)

The magnetic susceptibility of the prepared Nickel complexes was calculated at a temperature of 25°C . The diamagnetic corrections (D) for the atoms in the organic molecules, metal ions, and non-organic radicals were applied using Pascal's constants for the constituent atoms of the prepared complexes. D (in $\text{cm}^3\cdot\text{molecule}^{-1}$) is equal to the sum of the number of ions or atoms of the element

multiplied by Pascal's constant. The calculated values of the effective magnetic moment (μ_{eff}) for the prepared complexes were determined through magnetic measurements. The magnetic measurements of the prepared Nickel (II) complexes (A3-A6) revealed values ranging from (1.851-2.452) B.M. These results suggest that the metallic (II) ion exhibits tetra coordination with a high-spin tetrahedral configuration (Table 1).

Evaluation of the Biological Activity of Some Prepared Compounds A2-A6

Compounds with nonhomogeneous rings and their complexes exhibit varying biological activities against both Gram-positive and Gram-negative bacteria. In this study, the biological activity of some prepared compounds and complexes was assessed against two types of bacteria: *Staphylococcus* (Gram-positive) and *Escherichia coli* (Gram-negative). Complex (A5) showed a high inhibition of 28 mm against *Staphylococcus* bacteria, while complex (A4) showed a very high inhibition of 35 mm against

Staphylococcus bacteria. A4 and A5 showed more efficacy against Gram-positive bacteria compared to Gram-negative bacteria. This might happen due to Gram-negative bacteria having highly lipophilic cell walls. Therefore, chemicals having a stronger hydrophobic effect are needed to increase absorption at the site of action and exhibit a higher activity. The results indicate that these compounds and complexes possess the ability to inhibit the growth of both Gram-positive and Gram-negative bacteria to varying extents (Table 3, Figure 9).

Table 3. Biological effectiveness of prepared compounds, complexes and control parameters (in mm).

Comp. No.	<i>Escherichia coli</i>			<i>Staphylococcus</i>		
	25	50	100	25	50	100
A2	11	12	13	11	13	15
A3	13	16	17	24	26	29
A4	12	14	16	12	17	35
A5	10	16	22	14	21	28
A6	14	15	17	14	18	25
<i>Amoxicillin</i>	21	24	34	20	25	31
Blank disk	0	0	0	0	0	0

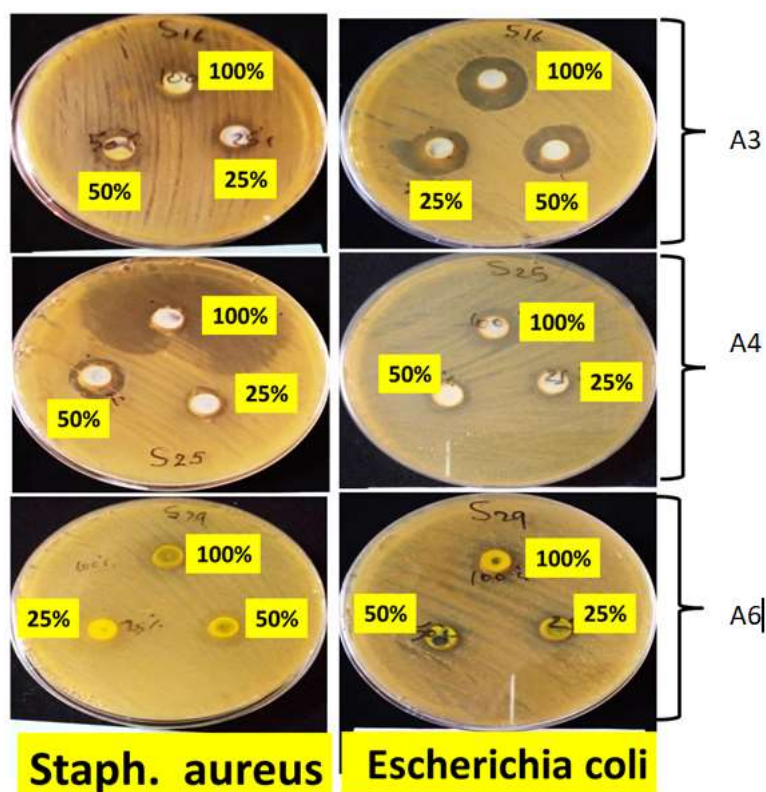


Figure 9. Inhibitory effectiveness of compounds A3, A4, A6 against both types of bacteria.

Conclusion

Compound A2 readily forms complexes, especially with nickel salts. The prepared compounds and complexes exhibited high stability and strength, maintaining their structure, color, and melting point even under varying laboratory temperatures between winter and summer. The biological study revealed that

most of the prepared compounds and complexes possess antibacterial activity and the ability to inhibit bacterial growth. These compounds exhibited higher biological efficacy compared to their parent compounds, which is of significant importance since the parent compounds are used as pharmaceuticals in the medical field.

Competing Interest: The authors declare that there is no conflict of interest.

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5-XLORO-8-XINOLINOL TÖRƏMƏLƏRİNİN ALINMASI, XARAKTERİSTİKASI, BİOLOJİ AKTİVLİYİNİN TƏDQIQI VƏ ONUN NİKEL (II) VƏ DİFOSFİNLƏRLƏ KOORDİNASİYASI

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Xülasə: Nikel kompleksləri alkil Qriqnard reagentlərinin alkenil-S və alkenil-Se birləşmələri ilə çarpaz birləşmə reaksiyaları üçün effektiv katalizatorlardır. Bu tədqiqatda müxtəlif üzvi birləşmələr əldə edilmişdir. Etilnil 2-((5-xloro-8-xinolinol)oksi)asetat birləşməsi (A1) xinolin və etilxloroasetata kalium karbonat əlavə edilməklə sintez edilmişdir. İkinci birləşmə 2-((5-xloro-8-xinolinol)oksi)asetohidrazid (A2) A1-in hidrazidlə reaksiyası nəticəsində əldə edilmişdir. A3 kompleks birləşməsi həlledici kimi etanoldan istifadə etməklə ekvimolyar miqdarda A2 birləşməsinin nikel duzunun məhlulu ilə reaksiya aparmaqla hazırlanmışdır. Fosfinat kompleksləri ekvimolyar miqdarda A3-ün müxtəlif fosfinlərlə reaksiyaya girməsi və həlledici kimi etanoldan istifadə etməklə alınmışdır. Sintez edilmiş birləşmələr və komplekslər müxtəlif spektroskopik üsullardan istifadə etməklə xarakterizə edilmişdir, o cümlədən FT-İnfra qırmızı spektroskopiya, (^{31}P - $\{^1\text{H}\}$ - ^{13}C -NMR) spektroskopiyası və C.H.N. analizi. Bundan əlavə, onların ərimə nöqtələri, təmizlik dərəcəsi, molyar keçiriciliyi və maqnit həssaslığı müəyyən edilmişdir. Bəzi hazırlanmış birləşmələrin və komplekslərin iki antibiotikə davamlı bakteriya ştamlarının: qram-mənfi stafilocokk və qram-müsbət Escherichia coli-nin böyüməsinə təsiri öyrənilmişdir. Nəzarət antibiotik kimi amoksisillindən istifadə edilmişdir. Sintez edilmiş birləşmələrin bəziləri sınaqdan keçirilmiş bakteriya ştammlarına qarşı əhəmiyyətli inhibitor xassə göstərdiyi müəyyən edilmişdir. A2 birləşməsi xüsusilə nikel, manqan və mis duzları ilə asanlıqla komplekslər əmələ gətirir. Yaranan birləşmələr və komplekslər qış və yay aylarında laboratoriya temperaturu dəyişdikdə belə öz strukturunu, rəngini və ərimə temperaturunu saxlayaraq yüksək dayanıqlıq və möhkəmlik nümayiş etdirmişdir.

Açar sözləri: 5-xloro-8-xinolinol, efir, nikel, difosfinat, komplekslər, bioloji aktivlik.

ПОЛУЧЕНИЕ, ХАРАКТЕРИСТИКА И ИССЛЕДОВАНИЕ БИОЛОГИЧЕСКОЙ АКТИВНОСТИ ПРОИЗВОДНЫХ 5-ХЛОРО-8-ХИНОЛИНОЛА И ЕГО КООРДИНАЦИИ С НИКЕЛЕМ (II) И ДИФОСФИНАМИ

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Резюме: Комплексы никеля являются эффективными катализаторами реакций перекрестного сочетания алкильных реагентов Гриньяра с соединениями алкенил-S и алкенил-Se. В ходе данного исследования были получены различные органические соединения. Этилнил-2-((5-хлоро-8-хинолинол)окси)ацетатное соединение (обозначенное как А1) синтезировали добавлением карбоната калия к хинолину и этилхлорацетату. Второе соединение 2-((5-хлоро-8-хинолинол)окси)ацетогидразид (А2) получали взаимодействием А1 с гидразином. Комплексное соединение А3 получали взаимодействием эквимольных количеств

соединения А2 с раствором соли никеля, используя в качестве растворителя этанол. Фосфинатные комплексы были получены путем взаимодействия равных молей А3 с различными фосфинами и использованием этанола в качестве растворителя. Синтезированные соединения и комплексы были охарактеризованы с использованием различных спектроскопических методов, включая Фурье-ИК спектроскопию, (^{31}P - $\{^1\text{H}\}$ - ^{13}C -ЯМР) спектроскопию и С.Н.Н. анализ. Дополнительно были определены их температуры плавления, чистота, молярная проводимость и магнитная восприимчивость. Изучено влияние некоторых приготовленных соединений и комплексов на рост двух антибиотикорезистентных штаммов бактерий: грамотрицательного стафилококка и грамположительной кишечной палочки. В качестве контрольного антибиотика использовали амоксициллин. Некоторые из синтезированных соединений проявили значительную ингибирующую активность в отношении тестируемых штаммов бактерий. Соединение А2 легко образует комплексы, особенно с солями никеля, марганца и меди. Полученные соединения и комплексы продемонстрировали высокую стабильность и прочность, сохраняя свою структуру, цвет и температуру плавления даже при изменении лабораторных температур зимой и летом.

Ключевые слова: 5-хлор-8-хинолинол, сложный эфир, никель, дифосфинат, комплексы, биологическая активность.