

Synthesis, characterization and biological activity of a new Schiff base oxime-imine ligand and its Zn (II) and Ni (II) complexes

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Abstract

In this study, a new ligand derived from Schiff base oxime-imine, namely (3Z)-3-((2-aminophenyl) imino) butan-2-one oxime (L) was prepared by the reaction of o-phenylenediamine with diacetyl monooxime at refluxing methanol. The structure of the ligand was confirmed by elemental analysis, and spectroscopic instruments (IR, UV-Vis, ¹H, ¹³CNMR and Mass spectrometry). Complexes of Zn (II) and Ni (II) metal ions with the specified ligand have been synthesized by reacting their chloride salts with the ligand in refluxing methanol, using a 1:1 mole ratio of (ligand: metal). Elemental analysis, spectroscopic techniques, magnetic moments and molar conductivity measurements confirmed the structure of the prepared complexes. The geometry of complexes was tetrahedral and square planar, for Zn (II) and Ni (II) respectively.

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1. INTRODUCTION

Schiff base compounds contain an azomethine group (-HC=N-) and were first reported by Hugo Schiff in 1864. These compounds are also known as anils, imines, or azomethines. Typically, they are formed by the condensation of a ketone or an aldehyde with a primary amine[1]. Schiff bases are among the most widely used organic compounds. its play a vital role in coordination chemistry, due to their ability to react with a range of metal ions forming stable complexes which have applications in different fields[2]. Schiff base-oxime ligands and Their complexes which contain imine and oxime groups play a very important role in medicine as chelating therapeutics, as pharmaceutical drugs, as inhibitors of specified enzymes and used in the biosynthesis of some nitrogen oxide compounds [3, 4]. Di oximes are amphoteric ligands because of slightly basic nitrogen atoms and moderately acidic hydroxyl groups forming different structural geometries with most transition metal ions[5, 6]. In addition, oxime metal ion complexes have an excellent ability to make good coordination sites[7, 8]. Vic-Dioxime complexes are used as model compounds for examining the structure of some biological compounds such as B₁₂ and coenzyme B₁₂, which have important uses in biological mechanisms [9]. Moreover, they are important in medical, industrial and analytical applications, these compounds exhibit various noteworthy attributes, such as high thermal stability, flame retardancy, and degradability. As a result, they have been widely employed in catalyst development, gas separation, and drug release industries[9, 10].

Although a large number of oximes and their transition metal ion complexes have been studied, there have been little amine-imine-oxime complexes reported [11]. Several examples of nickel Schiff base complexes have been reported, highlighting their significant functions, including their important role as catalysts in olefin epoxidation and oxidation reactions[12].

Some oxime Schiff bases and their metal complexes exhibit antibiotic, antiviral, and antitumor activities. It has been suggested that the azomethine linkage is responsible for the biological activities of Schiff bases, which include antitumor, antibacterial, antifungal, and herbicidal effects[13]. Oxime ligands can form stable biological and toxicological metal complexes with a wide range of metals. There is a growing interest in the synthesis and characterization of biologically active transition metal complexes, both within our group and in the broader research community[14].

2. METHOD

All reagents and solvents used in preparation were of reagent quality. Diacetylmonooxime, o-phenylene diamine, as well as $ZnCl_2$, $NiCl_2 \cdot 6H_2O$, and CCl_4 , were utilized as received without additional purification. Elemental analyses (C.H.N) were conducted using the Eager 300 for EA112. Infrared spectra were acquired using a SHIMADZU 8400S Spectrophotometer with KBr and CsI pellets. Proton and carbon-13 nuclear magnetic resonance (NMR) spectra were recorded using a Bruker 400 MHz instrument.

UV-Vis spectra were obtained using a spectrophotometer with a quartz cell of 1.0 cm path length. Magnetic susceptibilities for complexes were measured at room temperature using Sherwood Scientific equipment. Molar conductivity data for complexes were determined utilizing an A Starter 3000A apparatus.

Synthesis of (3Z)-3-((2-aminophenyl) imino) butan-2-one oxime (L). (1.05g, 8.9 mmole) of o-phenylene diamine were dissolved in 10 mL of methanol with continuous stirring. A solution of (1g, 8.9 mmole) of diacetylmonooxime was added dropwise to the solution in a round bottom flask. The mixture was refluxed for 3 hours. Then the mixture had a clear crystalline brown, which later transformed into a crystalline brown precipitate. The precipitate was filtered off, washed with 5ml of methanol, then with 3ml of cold ethanol and dried. Color: crystalline brown (Yield 0.5g, 29%), (m.p; 180-182 °C).

Synthesis of complexes

Synthesis of [LZnCl]Cl (C₁). (0.2g, 1 mmole) of (L) were dissolved in 10 ml of methanol, followed by the dissolution of (0.13g 1 mmole) of Zinc Chloride in 5 ml of methanol. The first mixture was then added to the second one under continuous stirring.

The resulting mixture was heated to (82 °C) and refluxed for 3hr. Subsequently, the mixture was filtered, washed with a few drops of methanol, and the residue was collected and dried. The weight of the residue was determined to be (0.09g). Color: Reddish brown; (Yield 0.18g, 56%), (m.p; 112-118 °C).

Synthesis of LNiCl₂ (C₂). The compound (C₂) was prepared by the same procedure as compound (C₁) The substance (0.24g, 1 mmole) ($NiCl_2 \cdot 6H_2O$) was used instead of, (0.13g, 1mmole) $ZnCl_2$.

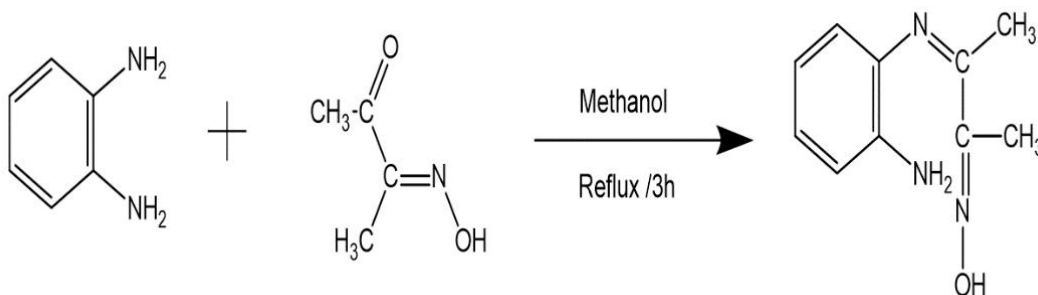
Color: Red; (Yield 0.2g, 71%), (m.p; 313-316 °C).

Study of the biological activity. The synthesized compounds were subjected to biological activity testing against two bacterial species, namely *E. coli* and *S. aureus*, utilizing the diffusion method[15]. To evaluate their biological activities, three distinct concentrations of these compounds were prepared. The starting concentration was established at 100 µg/mL, and a secondary concentration was attained by dissolving a portion of the compounds in DMSO to reach 50 µg/mL. Furthermore, a third concentration of 25 µg/mL was incorporated into the testing procedure.

Preparation of Mueller Hinton Agar. For preparing this medium, 38 grams were dissolved in 1 liter of distilled water, then sterilized using an autoclave at 121°C and 15 psi for 15 minutes. Once cooled, the medium was transferred into sterile dishes and refrigerated until needed. Using a micropipette, 100 µl of the substance was added to each well separately. Sterile distilled water was used as the representation for positive and negative controls. Subsequently, the dishes were incubated at 37°C for 24 hours[15, 16].

3. RESULTS AND DISCUSSION

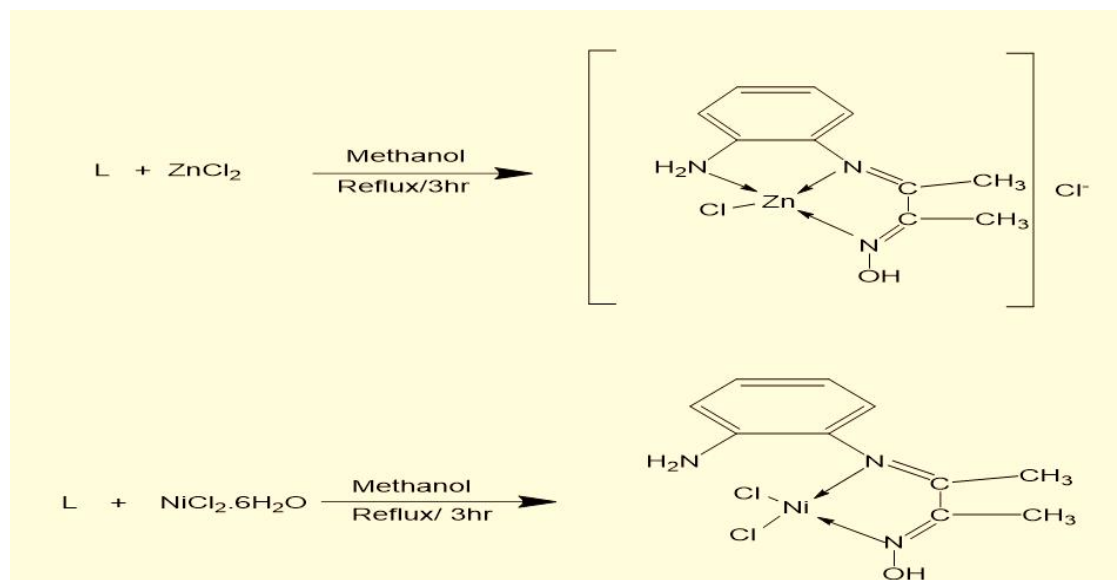
A New oxime-Imene ligand was Synthesis from the reaction of (o-phenylene diamine) according to the following procedure shown in Scheme 1. The structure of the synthesized ligand is validated through elemental analyses, showing good agreement with the calculated values (refer to Table 1), as depicted in Scheme 2.



Scheme 1. Synthesis route of the ligand

Table 1. Elemental analysis data and physical properties for ligand and its complexes

No	Molecular Formula	M.wt g/mol	Yield%	Colour	m.p°C
L	C ₁₀ H ₁₃ N ₃ O	191.23	29%	crystalline brown	180-182
C1	C ₁₀ H ₁₃ Cl ₂ ZnN ₃ O	326.48	56%	Reddish brown	112-118
C2	C ₁₀ H ₁₃ Cl ₂ NiN ₃ O	319.6	71%	Red	313-316



Scheme 2. Preparation route of the Complexes

3.1. Synthesis and characterization for L and its complexes

All complexes were synthesized using identical procedures. Microanalysis of the elements and metal analysis closely matched the calculated values, as shown in Table 1. Structural characterization of the complexes was achieved through spectroscopic analyses, magnetic moment measurements, and molar conductivity.

3.1.1. EI mass spectrum for ligand

EI mass spectrum of the ligand showed a parent ion peak at $m/z = 191.23$ corresponds to (M^+) confirming the molecular weight of the ligand, Figure (1).

3.1.2. IR spectra for the ligand L and its complexes

The infrared spectrum of ligand L displayed distinctive peaks. The peak at 3309 is belong to symmetric and asymmetric (N-H) stretching frequencies. The medium peak at 3417 cm^{-1} is assigned to $\nu(\text{O-H})$ stretching frequency of the oxime group. The Peaks at 1627 and 1496 cm^{-1} are due to $\nu(\text{C=N})$ stretching frequency of azomethine imine and oxime respectively. However, these peaks are shifted to lower frequencies in complexes and showed at the (1573-1404) cm^{-1} range. This is attributed to the participation of the nitrogen atoms of the imine and oxime group in complexation with metal ions. The peak of the ligand at 1134 cm^{-1} is belong to $\nu(\text{N-O})$ stretching frequency.

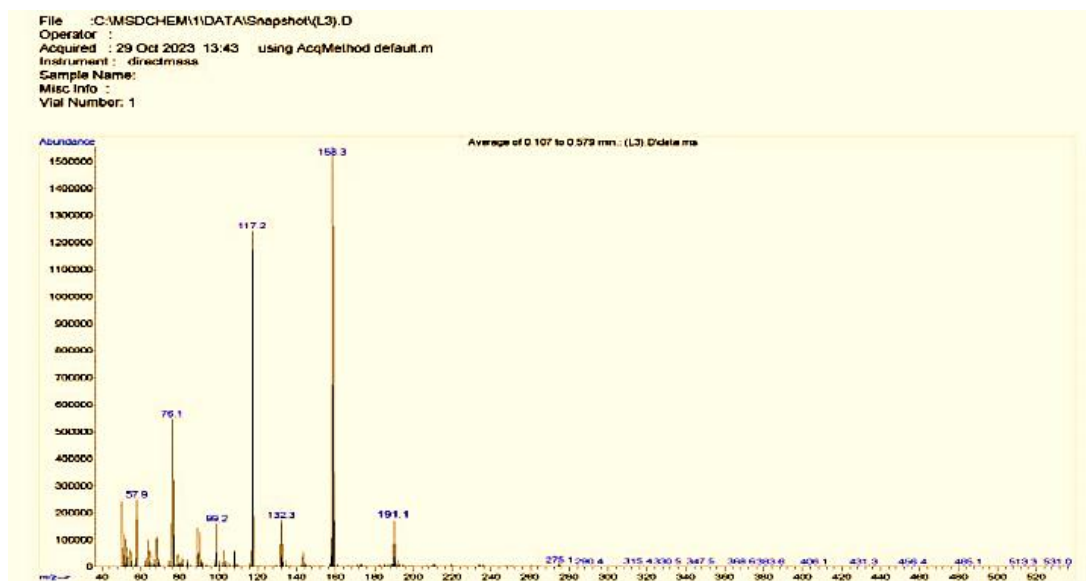


Figure 1. EI mass spectrum for the Ligand

Moreover, this peak shifted to higher and lower frequencies in complexes and appeared at the (1141-1103) cm^{-1} elucidate a complexation with the ligand through the nitrogen atom of the oxime group. In addition, all the spectra of the complexes appeared further peaks at lower frequencies showed at (470-424) cm^{-1} and (223-262) cm^{-1} range belong to $\nu(\text{M-N})$ and $\nu(\text{M-Cl})$ stretching frequencies respectively [17, 18]. These peaks are absent in the ligand spectra see Table (2) and Figures (2,3,4).

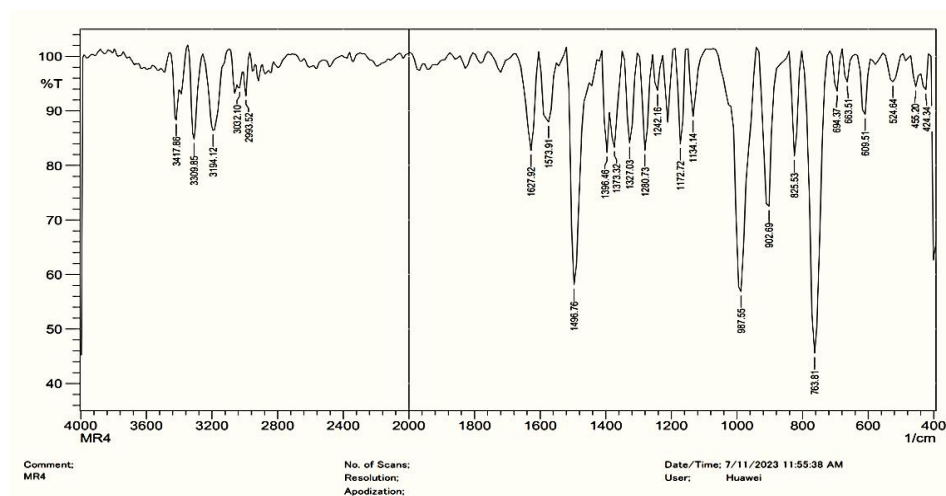


Figure 2. The infrared spectrum of the Ligand

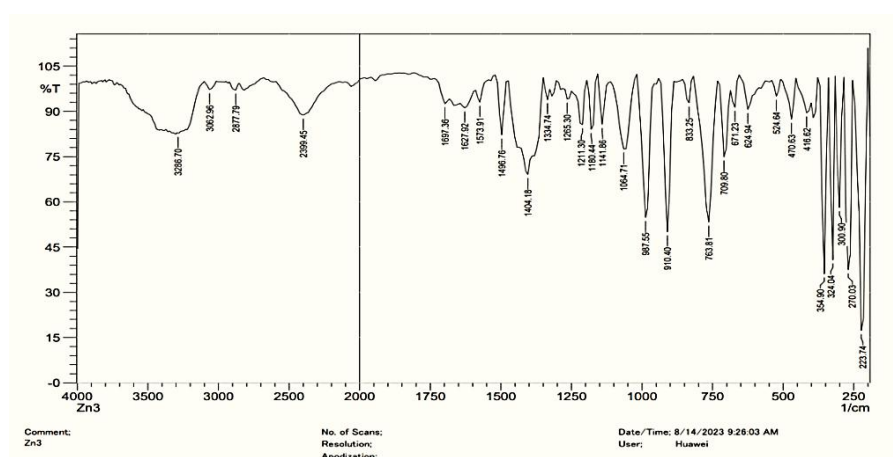


Figure 3. The infrared spectrum of [LZnCl]Cl complex

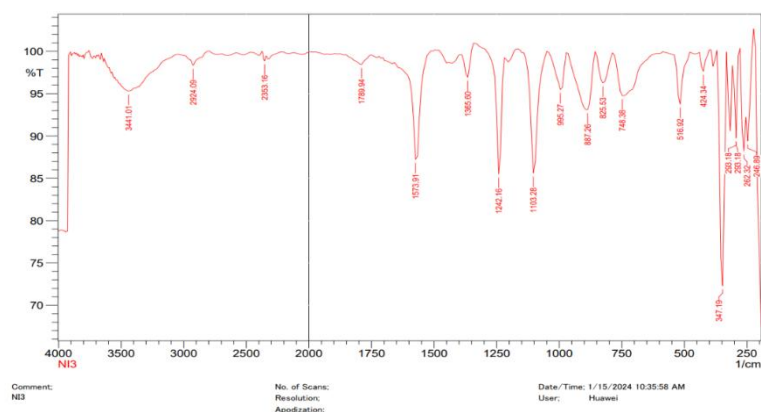
Figure 4. The infrared spectrum of LNiCl_2 complex

Table 2. Infrared spectra for ligand and its complexes

No	V(N-H) Assm. Ssm.	V(O-H) Oxime H_2O	V(C-H) Arom. Aliph.	V(C=N) Imine	V(C=N) Oxime	$\nu(\text{N-O})$	V(C-H) o.o.p Arom.	V(M- N)	$\nu(\text{M-Cl})$
L	3309m	3417w	3032w 2993w	1627s	1496m	1134m	987m	-----	-----
$[\text{LZnCl}] \text{Cl}$	3286w	-----	3062w 2877w	1496m	1404m	1141m	763w	470w	223m
LNiCl_2	3170w	3441w	3010w 2924w	1573w	1365w	1103m	995w	424w	262w

3.1.3. U.V-Vis Spectra of ligand and its complexes

U.V - Vis. The spectra of the ligand, Figure (5), appeared an intense peak at (258 nm) (38760 cm^{-1}) ($E_{\text{Max}}=1839 \text{ molar}^{-1} \cdot \text{cm}^{-1}$) and (316 nm) (31646 cm^{-1}) indicated ligand field ($\pi \rightarrow \pi^*$), ($n \rightarrow \pi^*$).

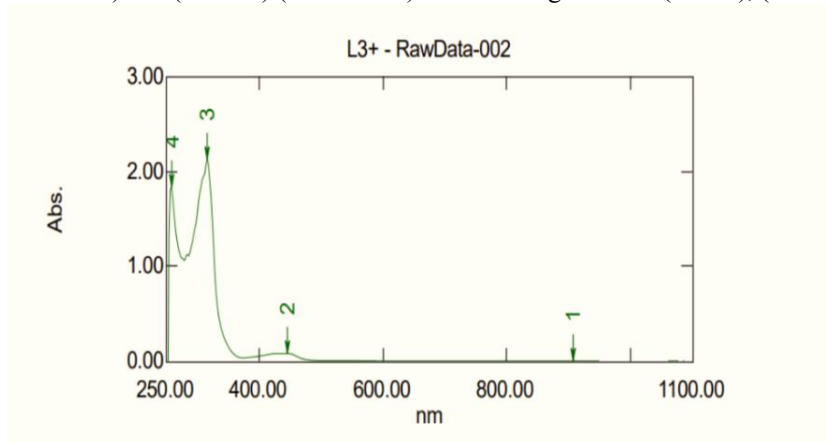
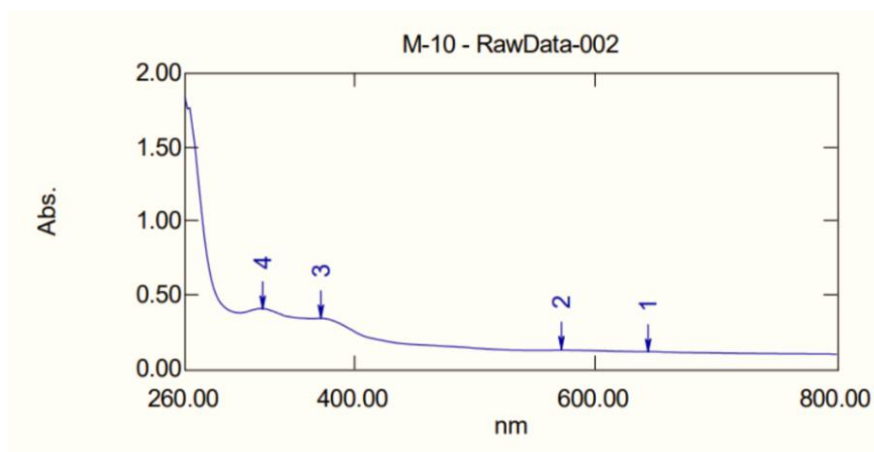


Figure 5. UV-VIS spectrum for the ligand

Figure 6. UV-VIS spectrum for LNiCl_2 complex

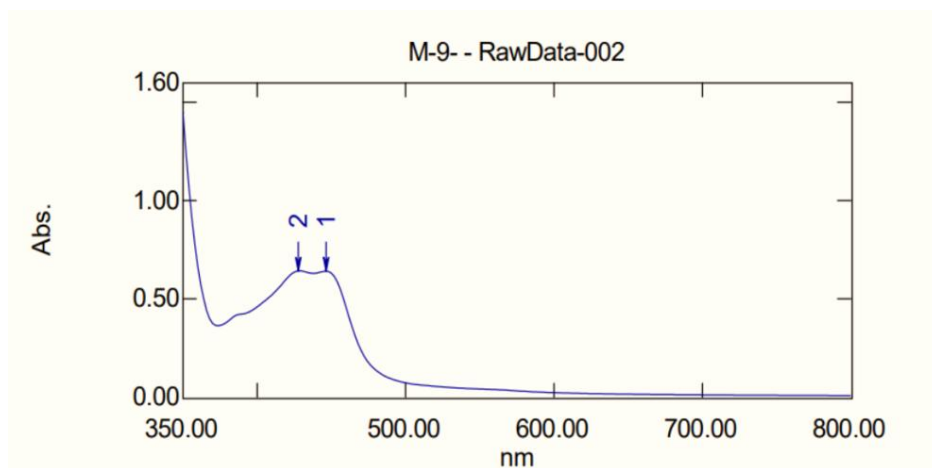


Figure 7. UV-VIS spectrum for [LZnCl]Cl complex

3.1.4. Molar conductivity

Zn (II) complex exhibited molar conductance within the $(136 \text{ S}\cdot\text{cm}^2\cdot\text{mole}^{-1})$, suggesting ionic behavior with a 1:1 ratio. Conversely, the Ni (II) complex displayed characteristics of a non-electrolyte [21], Table 4.

Table 4. The molar conductance of the complexes

COMP.	COMPLEX	Molar conductivity ($\text{S}\cdot\text{cm}^2\cdot\text{mole}^{-1}$)
C ₁	[LZnCl]Cl	136
C ₂	LNiCl ₂	0

3.1.5. Magnetic Moments

The effective magnetic moment values for the LNiCl₂ complex (0.7), indicating an square planar and tetrahedral geometry around Ni(II) and Zn(II) metal ion.[19, 22]. Table (5).

Table 5. Magnetic Moment ($\mu_{\text{eff}} = \text{B.M}$) of L complexes at 298K and suggested stereochemical structures.

Compound	μ_{eff} . B.M	Proposed Geometry
[LZnCl] Cl	diamagnetic	tetrahedral
LNiCl ₂	0.7	Square planar

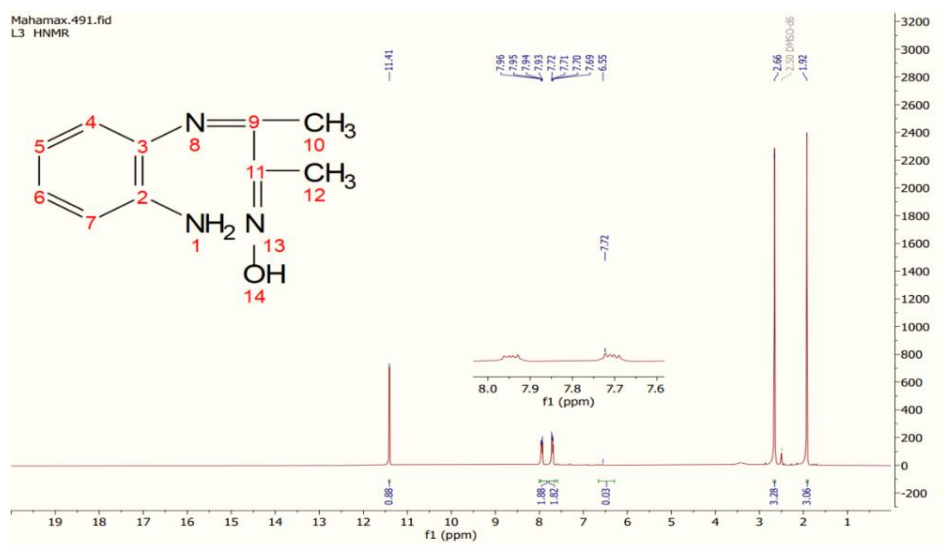
3.1.6. ¹HNMR spectrum for L ligand and [LZnCl] Cl complex

The spectra of the ligand in DMSO-d₆ solvent is presented in Figure (8) appeared a signal at (11.41 ppm, 1H, singlet) belong to (O-H) of the oxime group. though, this signal vanished when D₂O was introduced into the solution. The aromatic protons gave a signal at (7.96-7.7 ppm, 4H, multiplet). It's noteworthy, that the singlet peak at (6.55 ppm, 2H, singlet) equivalent to proton is due to NH₂. However, this signal disappeared after the addition of D₂O to the solution [23].

The signals for CH₃ protons adjacent to (-C=N-OH) showed a signal at (2.66 ppm, 3H, singlet), While the chemical shift for the CH₃ adjacent to C₉ emerged at (1.92 ppm, 3H, singlet)[24]. furthermore, the spectra for the LZnCl] Cl complex appeared a signal as foreseeable chemical shift (11.40 ppm, 1H, singlet). The signal of aromatic protons appeared at chemical shift (7.95-7.70 ppm, 2H, doublet). The signal of NH₂ was shifted to a higher field and showed at (5.87 ppm, 2H, singlet) attributed to complex formation. The CH₃ signal adjacent to (-C=NOH) showed at a chemical shift (2.66 ppm, 3H, singlet)[25]. However, this signal was shifted to a higher field upon complexation. The signal of CH₃ adjacent to (-C=N-) appeared at (1.92 ppm, 3H, singlet)[26]. All data are listed in Table (6).

Table 6. ¹HNMR data for the ligand and [LZnCl] Cl complex

Funct. Group	δ(ppm) (Ligand)	δ(ppm) (C ₁)
-C=N-OH	11.41 (s, 1H)	11.40 (s, 1H)
C-H (aromatic)	(7.7-7.96) (m, 4H)	(7.70-7.95) (m, 4H)
CH ₃ -C=N-OH	2.66 (s, 3H)	2.67 (s, 3H)
CH ₃ -C=N-	1.92 (s, 3H)	1.93 (s, 3H)
NH ₂	6.55 (s, 2H)	5.87 (s, 2H)

Figure 8. ¹HNMR spectrum of the ligand

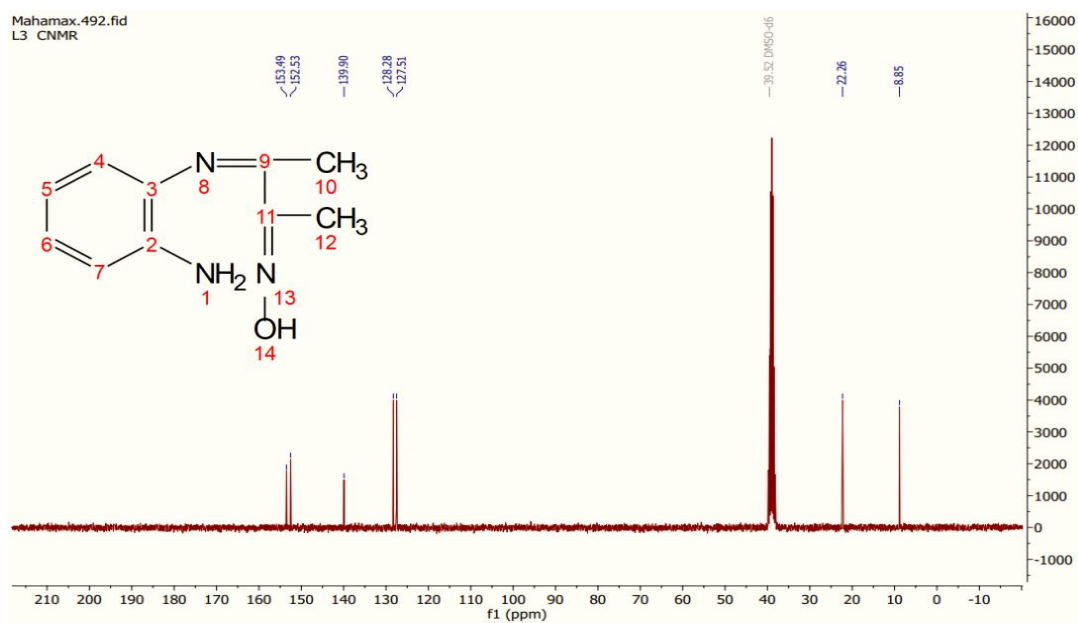
3.1.6. ¹³CNMR spectrum for ligand and [LZnCl] Cl complex

The spectra of the ligand, Figure (9) in DMSO-*d*₆ appeared a signal at 153.49 ppm belong to (-C=NOH). The signal at 152.53 ppm is due to (-C=N-). The signals at 127.51-139.90 ppm range attributed to (C₂,C₃,C₄,C₅,C₆,C₇ (aromatic)). The signal of (CH₃-C=N-) showed at 22.26 ppm. In addition, the signal at 8.80 ppm is belong to (CH₃-C=NOH)[27]. The ¹³CNMR spectra of the [LZnCl] Cl complex revealed a signal at 154.46 ppm belong for (-C=NOH). However, this signal is shifted to downfield upon complexation. The signal at 153.51 ppm is assigned to (-C=N-).

The signals at (128.45-140.84 ppm) range belong to (C₂,C₃,C₄,C₅,C₆,C₇ (aromatic)). The signals at 23.23 ppm and 9.80 ppm are due to (CH₃-C=N-) and (CH₃-C=NOH) respectively[28]. All data are reported in Table (7).

Table (7). ¹³CNMR data for the ligand and [LZnCl] Cl complex

Funct. Group	δ(ppm) Ligand	δ(ppm) (C ₁) Complex
-C=N-OH	153.49	154.46
C=N-	152.53	153.51
C ₂ ,C ₃ ,C ₄ ,C ₅ ,C ₆ ,C ₇ (aromatic)	(127.51,128.28,139.90)	(128.45,129.25,140.84)
CH ₃ -C=N-	22.26	23.23
CH ₃ -C=N-OH	8.80	9.80

Figure 9. ^{13}C NMR spectrum of the ligand

3.2. Biological Activity

The ligand and its complexes were assessed for their biological effects on two distinct bacteria strains, namely *Staphylococcus aureus* (G+) and *Escherichia coli* (G-), using DMSO concentrations of 100, 50, and 25 $\mu\text{g}/\text{ml}$. The results are presented in (Table 8) and illustrated in Figure (10). Analysis of the table indicates that at a concentration of 25 $\mu\text{g}/\text{ml}$, both the ligand and the LNiCl_2 complex exhibited no discernible biological activity against (*Staphylococcus aureus*) (G+). Similarly, at this concentration, the LNiCl_2 complex demonstrated no biological activity against (*Escherichia coli*) (G-). Conversely, all compounds exhibited biological activity at concentrations of 50 and 100 $\mu\text{g}/\text{ml}$ against both bacterial strains, except for the LNiCl_2 complex, which showed no biological activity against (*Staphylococcus aureus*) at any concentration, Because it is neutral or non-ionic [29, 30].

Table 8. Biological activity for prepared ligand and its complexes

Compound	Concentration ($\mu\text{g}/\text{ml}$)	<i>Staphylococcus Aureus</i> (G+)	<i>Escherichia Coli</i> (G-)
<i>L</i>	100	13	16
	50	11	15
	25	R	11
$[\text{LZnCl}] \text{Cl}$	100	23	25
	50	14	18
	25	12	16
LNiCl_2	100	R	16
	50	R	12
	25	R	R

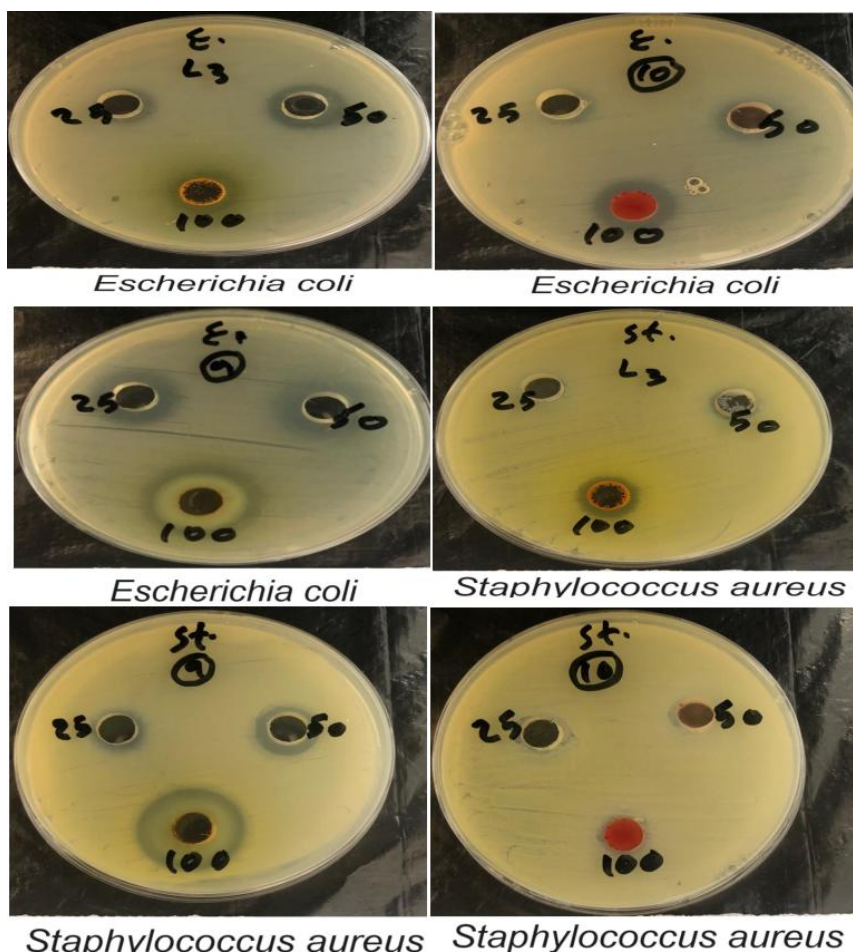


Figure 10. Assessing the Antiviral Effectiveness for the Ligand and Its Complexes [C1, C2] Against *Staphylococcus aureus* and *Escherichia coli*

4. CONCLUSION

In this study, a newly synthesized Schiff base-oxime ligand, along with its zinc (Zn^{2+}) and nickel (Ni^{2+}) complexes, were examined at appropriate ratios. Various analytical techniques, including magnetic susceptibility, molar conductivity, IR spectroscopy, nuclear magnetic resonance (NMR) with ^{13}C and 1H , ultraviolet-visible spectroscopy, and mass spectroscopy, were employed to analyze the complexes. The results obtained validated several structural characteristics. The amine-oxime ligand and its metal complexes exhibited activity ranging from moderate to good against two distinct bacterial species investigated in this study. Proposed geometric configurations revealed the Zn^{2+} complex to have a tetrahedral structure and the Ni^{2+} complex to possess a square planar geometry.



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