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Synthesis, Characterization, and Antimalarial Evaluation of Cu(II) and Zn(II) Complexes of Isoniazid-Quinoline Schiff Bases

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Abstract

Schiff bases are versatile ligands capable of forming stable coordination compounds with transition metals, often enhancing biological activity through chelation. In this study, two quinoline-based Schiff base ligands derived from isoniazid were synthesized and used to prepare their Cu(II) and Zn(II) complexes. The ligands and their complexes were characterized using infrared, ultraviolet-visible, and nuclear magnetic resonance spectroscopy. Spectroscopic data confirmed bidentate coordination through azomethine nitrogen and carbonyl oxygen atoms, with Cu(II) complexes exhibiting distorted octahedral and square planar geometries, while Zn(II) complexes showed octahedral geometry. The compounds were evaluated for *in vitro* antimalarial activity against *Plasmodium falciparum* (3D7 strain) using the SYBR Green I fluorescence assay. All compounds demonstrated more than 50% inhibition at 10 μM, with the Cu(II) complex of ligand L1 showing the highest activity (69.0%), close to the values of chloroquine (74.3%) and artemisinin (74.4%) under the same screening conditions. These findings highlight the potential of isoniazid-derived Schiff base metal complexes as promising candidates for the development of new antimalarial agents.

Keywords

Schiff Bases, Isoniazid, Quinoline, Cu(II) Complexes, Antimalarial Activity

1. Introduction

Schiff bases, characterized by the azomethine (–C=N–) functional group, represent one of the most versatile classes of ligands in coordination chemistry [1] [2]. Formed via condensation of primary amines with carbonyl compounds, these ligands possess nitrogen and oxygen donor atoms that readily coordinate to transition metal ions, yielding stable chelate complexes with diverse structural motifs [3] [4]. The ability to fine-tune their electronic and steric properties by varying the amine and aldehyde precursors makes them valuable for designing metal complexes with specific coordination geometries [5] [6].

Transition metal complexes of Schiff bases have attracted considerable attention due to their intriguing structural diversity, ranging from tetrahedral and square planar to octahedral and distorted geometries [7] [8]. Copper(II) and zinc(II) are of special interest in coordination chemistry. Copper(II), with its d^9 configuration, exhibits Jahn-Teller distortion, leading to elongated octahedral or square planar geometries [9] [10]. Zinc(II) is d^{10} and forms stable complexes with different coordination numbers, often serving as structural models for biologically relevant metalloenzymes [11] [12].

The ligand backbone significantly influences the coordination behavior and properties of the resulting metal complexes. Isoniazid (isonicotinic acid hydrazide), a good pharmacophore widely known for its role in tuberculosis therapy, serves as an excellent precursor for Schiff base formation due to its hydrazine moiety, capable of undergoing condensation with aldehydes to yield hydrazone-type Schiff bases [13] [14]. The resulting ligands contain multiple potential donor sites, including the azomethine nitrogen, carbonyl oxygen, and pyridine nitrogen, offering flexible coordination modes [15]. Incorporating quinoline moieties into the ligand framework introduces additional aromatic character and electron delocalization, which can influence the electronic environment around the metal center [16] [17].

Beyond their fundamental coordination chemistry interest, Schiff base metal complexes have demonstrated a wide range of applications, including catalytic, magnetic, and biological properties [18] [19]. Often, the metal complex is more active biologically than the free ligand. This is due to chelation, which increases lipophilicity, changes electron distribution and facilitates interaction with biomolecular targets [20] [21].

Malaria remains a major global health challenge, with an estimated 249 million cases and 608,000 deaths reported in 2022, predominantly in sub-Saharan Africa [22]. Resistance to artemisinin-based drugs and other frontline antimalarials is increasing, so there is an urgent need for novel chemotherapeutic agents with new mechanisms of action [23] [24]. Metal-based compounds offer promising alternatives due to their ability to interact with multiple biological targets and overcome resistance mechanisms [25] [26].

The 2- and the 4-quinoline isomers were selected to investigate the influence of ligand isomerism on coordination behavior. Understanding how these structural variations translate into differences in biological activity provides valuable insight into structure activity relationships in coordination compounds.

In the present work, we report the synthesis of two isomeric Schiff base ligands derived from isoniazid and quinoline-2-carboxaldehyde (L1) and quinoline-4-

carboxaldehyde (L2), along with their Cu(II) and Zn(II) complexes. The compounds were characterized by standard spectroscopic methods, and their antimalarial activity was evaluated. The antimalarial screening serves to demonstrate the potential biological relevance of these coordination compounds, highlighting the role of metal complexation in modulating biological activity.

2. Experimental

2.1. Materials

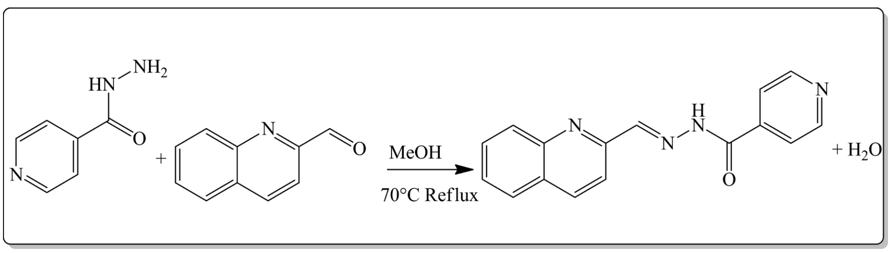
All reagents used were of analytical grade and purchased from recognized suppliers. Isoniazid, quinoline-2-carboxaldehyde, and quinoline-4-carboxaldehyde were sourced from Sigma-Aldrich. $CuCl_2 \cdot 2H_2O$, $ZnCl_2$, methanol, ethanol, and dimethyl sulfoxide (DMSO) were purchased from Fisher Scientific. These chemicals were used as received without further purification. Solubility and melting point measurements were conducted using standard techniques.

2.2. Instrumentation

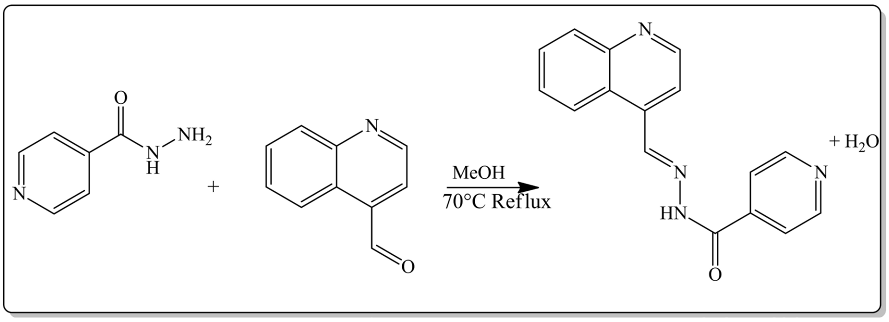
Melting points were determined using a digital melting point apparatus and are uncorrected. Infrared (FT-IR) spectra were recorded on Alpha-P Bruker spectrometer on diamond plate in the range; 450 - 4000 cm^{-1}. UV-Visible spectra were obtained using a UV (200 - 400 nm) and visible (400 - 800 nm) regions. The UV/Vis spectroscopic analysis of the ligands and the metal complexes was carried out in DMSO at room temperature. ^1H and ^{13}C spectra were recorded on an NMR V500 spectrophotometer in DMSO and values were reported relative to TMS as internal standard. All spectra and characterization data were compared with literature where appropriate.

2.3. Synthesis of the Schiff Base Ligands

The ligands were synthesized by a condensation reaction between equimolar quantities of isoniazid (0.549 g, 4.0 mmol) and the appropriate quinoline aldehyde (0.629 g 4.0 mmol). For ligand L1, isoniazid was reacted with quinoline-2-carboxaldehyde, Scheme 1, while for L2, the reaction involved quinoline-4-carboxaldehyde Scheme 2. The reactions were carried out under reflux in methanol for 4 hours at 70˚C. Thin Layer Chromatography (TLC) was used to monitor the reaction progress. The solid products were filtered, washed with cold methanol, and dried in a desiccator.



Scheme 1. Synthesis of N-(quinolin-2-ylmethylene)isonicotinohydrazide L1.



Scheme 2. Synthesis of N-(quinolin-4-ylmethylene)isonicotinohydrazide L2.

2.4. Synthesis of Metal Complexes

To synthesize Cu(II) and Zn(II) complexes, the Schiff base ligands (1.0 mmol) were dissolved in hot ethanol (10 mL) and mixed with a stoichiometric amount of the metal chloride salt (0.5 mmol) in ethanol. The reaction mixtures were refluxed at 80˚C for 2 hours and then left to stand at room temperature overnight. The resulting precipitates were filtered, washed several times with ethanol, and dried between filter papers. The metal-to-ligand molar ratio used was 1:2 (M:L), consistent with bidentate coordination [15].

2.5. Antimalarial Activity Assay

Antimalarial activity was evaluated against chloroquine-sensitive *P. falciparum* (3D7 strain) using a SYBR Green I-based fluorescence assay at Centre Pasteur, Cameroon [27]. Parasites were cultured in RPMI-1640 medium supplemented with human O^+ erythrocytes at 1% parasitemia and 1.5% hematocrit. Test compounds and controls (chloroquine, artemisinin) were prepared in DMSO and tested in duplicate at 10 μM. The experiment was performed twice independently, and variability is presented as individual assay values in Table 4. After 72 h incubation, parasite growth inhibition was quantified fluorometrically. Percentage inhibition was calculated relative to solvent control (0.1% DMSO).

3. Results and Discussions

3.1. Physical Properties

The principal physical properties are presented in Table 1. The condensation of isoniazid with the respective quinoline aldehydes yielded the Schiff base ligands L1 and L2. The Schiff bases (L1 and L2) exhibited pale yellow and white colors respectively, while the Cu(II) and Zn(II) complexes exhibited distinct color changes, suggesting complex formation. The melting points of the ligands were sharp (L1: 190.4˚C, L2: 237.1˚C), indicating purity, while all complexes showed decomposition temperatures above 300˚C, indicating high thermal stability [28]. All compounds were insoluble in water and alcohols but soluble in DMSO.

Elemental analysis (CNH) data for the ligands and their metal complexes are presented in Table 2. The experimentally determined values of the elements are in good agreement with the calculated values for the proposed 1:2 metal-to-ligand stoichiometry

Table 1. Some physical properties of the synthesized compounds.

Compound	Physical nature	Colour	%Yield	Melting point/˚C
L1	Powder	Pale yellow	84.81	190.40
L2	Powder	White	97.74	237.05
CuL1	Powder	Blue green	70.24	>300.00
CuL2	Powder	Forest green	70.64	>300.00
ZnL1	Powder	Yellow	67.21	>300.00
ZnL2	Powder	Pale yellow	39.76	>300.00

Table 2. Elemental analysis.

Compound code	Carbon		Hydrogen		Nitrogen	
	Expected	Found	Expected	Found	Expected	Found
L1	65.30	65.78	4.79	4.93	19.04	18.45
L2	59.27	58.25	5.87	5.31	16.27	16.92
CuL1	58.93	58.29	4.33	4.00	17.18	17.68
CuL2	62.38	62.93	3.93	3.87	18.19	18.01
ZnL1	58.77	58.51	4.32	4.18	17.17	17.99
ZnL2	58.77	58.86	4.32	4.69	17.17	17.61

3.2. Spectral Characterization

3.2.1. IR Spectroscopy

The IR spectra of L1 and L2 showed characteristic bands at ~1553 cm^{-1} ν (C=N), ~1659 cm^{-1} ν (C=O), and ~3165 cm^{-1} ν (N-H) [7]. Upon complexation, the ν (C=N) band shifted to lower frequencies (1541 - 1556 cm^{-1}), indicating coordination via the azomethine nitrogen. An upward shift in ν (C=O) suggested participation of the carbonyl oxygen in coordination [29]. New bands in the 420 - 520 cm^{-1} region were attributed to ν (M-N) and ν (M-O) [30]. Broad bands around 3400 cm^{-1} in some complexes indicated the presence of coordinated or lattice water, this band was absent in the CuL2 complex [16]. The combined spectral shifts and the appearance of new metal-ligand bands strongly suggested that the ligands act as neutral bidentate donors, coordinating through the azomethine nitrogen and carbonyl oxygen atoms [7] [31]. The major IR absorption bands of the compounds are summarized in **Table 3**.

Table 3. The IR absorption bands of the ligands and the complexes.

Compound	(C=N)	(C=O)	(N-H)	(C=C)	(H_2O)	(C=N)
L1	1553	1656	3184	1596	3425	1553
L2	1552	1662	3146	1559	3321	1552

Continued

CuL1	1556	1658	3169	1598	3407	1556
CuL2	1546	1698	3104	1616	-	1546
ZnL1	1554	1662	3176	1596	3438	1554
ZnL2	1541	1693	3120	1633	3262	1541

3.2.2. UV-Visible Spectral Analysis

The ligands displayed strong absorption bands at: L1: 354 nm (n→π) and L2: ~357 nm (n→π), characteristic of azomethine (–C=N) chromophores, confirming successful Schiff base formation [32]. The Cu(II) complex of L1 (CuL1) exhibited a broad d-d transition at 658 nm attributed to the $^2Eg \rightarrow {}^2T2g$ transition typical of distorted octahedral geometry arising from Jahn-Teller distortion [9] [33]. In contrast, CuL2 showed a broad d-d band at 661 nm consistent with square planar geometry, which is supported by the absence of an OH band due to water in the IR spectrum [11] [34]. Additional bands in the near-UV region (~357 nm) correspond to intra-ligand transitions, confirming retention of ligand chromophores in the metal-bound form [12]. Zinc(II) complexes, being d^{10}, showed no d-d transitions, as expected. Their spectra are dominated by charge-transfer bands (M→L CT) at 398 - 406 nm and intra-ligand bands around 357 nm [35] [36].

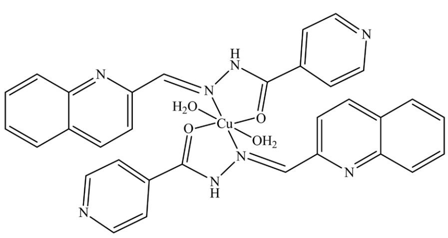
3.2.3. NMR Spectroscopy

The ^1H NMR spectra of the ligands displayed diagnostic singlets for the azomethine proton (δ 8.62 - 9.09 ppm) and the hydrazide NH proton (δ 12.39 - 12.42 ppm) [37]. The ^{13}C NMR spectra showed signals for the azomethine carbon (δ 146 - 147 ppm) and carbonyl carbon (δ 162 ppm), confirming Schiff base formation [38] [39]. No major impurities were detected, confirming the purity and integrity of the ligands.

3.3. Proposed Structures of Metal Complexes

Based on spectroscopic data and literature [11] [12], the proposed structures are as follows:

- CuL1 and ZnL1: Six-coordinate octahedral geometry (**Figure 1**).
- CuL2: Four-coordinate square planar geometry. (**Figure 2(a)**)
- ZnL2: Six-coordinate octahedral geometry. (**Figure 2(b)**)



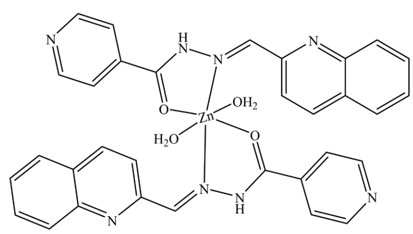


Figure 1. (a) The Cu(II) complex of ligand one (L1); (b) The Zn(II) complex of L1.

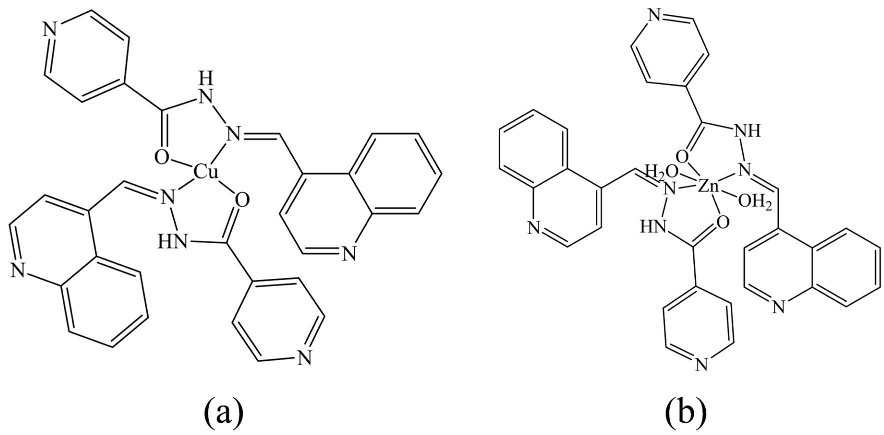


Figure 2. (a) The Cu(II) complex of L2; (b) The Zn(II) complex of L2.

The proposed geometries are based on IR, UV-Vis, and NMR spectroscopic data, along with elemental analysis; single crystal X-ray diffraction will be required for unambiguous confirmation.

3.4. Antimalarial Activity

The parasite growth inhibition for the ligands and complexes was evaluated, and the results are presented in **Table 4**. All compounds demonstrated > 50% inhibition of *P. falciparum* growth at 10 μM. The two isomeric ligands showed different activities, with L1 (57.4%) exhibiting greater inhibition than L2 (54.2%). Metal complexation generally enhanced activity compared to the free ligands, except for ZnL1 [20]. The copper complex of L1 (CuL1) was the most active among the synthesized compounds, with 69.0% inhibition at 10 μM. Under the same assay conditions, the reference drugs, chloroquine and artemisinin gave 74.3% and 74.4% inhibition respectively [17]. The enhanced activity upon chelation can be attributed to increased lipophilicity, modified electron distribution, and potential redox activity of the metal center [14] [21].

Table 4. SYBR Green I-based parasite growth inhibition.

	Inhibition percentage (%)		
Compound	**Assay 01**	**Assay 02**	**Mean**
L1	55.7	59.2	57.4
L2	54.7	53.7	54.2
CuL1	66.0	72.1	69.0
CuL2	61.6	61.5	61.6
ZnL1	53.2	58.7	55.9
ZnL2	60.9	63.2	62.1
Chloroquine	73.3	75.3	74.3
Artemisinin	73.4	75.4	74.4

4. Conclusion

Novel Schiff bases derived from isoniazid and quinoline aldehydes and their Cu(II) and Zn(II) complexes were successfully synthesized and characterized by various methods of analysis with yields ranging from 39% to 97%. Spectroscopic analysis (NMR, IR, UV-Visible) confirmed bidentate coordination through the azomethine nitrogen and carbonyl oxygen. Based on the spectral data and in accordance with literature, a six-coordinate octahedral structure was proposed for CuL1 and both zinc(II) complexes, while a four-coordinate square planar structure was proposed for CuL2. All compounds were screened for antimalarial activity against chloroquine-sensitive *P. falciparum* at 10 μM, with all showing >50% inhibition. The complexes generally exhibited higher activity than the free ligands, demonstrating that metal coordination enhances biological properties. CuL1 showed the highest inhibition (69.0%), close to the values of chloroquine (74.3%) and artemisinin (74.4%) under the same screening conditions. These findings identify CuL1 as a promising candidate for further antimalarial development. Future work should include IC_{50} determination and testing against drug-resistant strains to fully establish its potency profile.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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