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Synthesis and Characterization of Transition Metal Complexes with an Indole-Based Azo-Schiff Ligand

Maha Raheem Abd Alzaher[1], Zinah Areef Khalaf[2*]

[1]Al-Tabari Preparatory School for Boys, Al-Kifl District, Babil Education Directorate, Babil, Iraq.
[2]Fatima Al Zahra School for Distinguished Students, Al-Diwaniyah Education Directorate, Iraq.
[*]*Corresponding Author: zinah.areef@ec.edu.iq*

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Abstract

In this study, an indole-based azo Schiff base ligand was first synthesized via diazotization coupling and Schiff base condensation. The ligand was then coordinated with four transition metal ions, namely Cu (II), Ni (II), Co (II), and Zn (II), and stable-colored solid complexes were isolated. Subsequently, multi-dimensional characterization of the obtained complexes was carried out using elemental analysis, molar conductivity, magnetic susceptibility, Fourier-transform infrared spectroscopy (FT-IR), and ultraviolet-visible spectroscopy. FT-IR results showed that the characteristic peaks of ν(C=N) and ν(N=N) of the free ligand were located at 1618 cm^{-1} and 1452 cm^{-1}, respectively. After coordination, these peak positions shifted to lower wavenumbers by 10-25 cm^{-1}, and a new ν(M-N) vibration peak appeared in the 510-560 cm^{-1} range, confirming that the nitrogen atoms of the azomethine and azo groups participated in coordination. In the ultraviolet spectra, the free ligand exhibited a π→π transition at 270-320 nm and an n→π transition at 350-420 nm. The complexes showed additional charge transfer and d-d transitions. The absorption signals of the Cu (II), Ni (II), and Co (II) complexes at 520-760 nm were consistent with an octahedral configuration, while the diamagnetic Zn (II) complex matched a tetrahedral coordination mode. Magnetic susceptibility tests returned magnetic moments of approximately 1.8 BM for Cu (II), 3.1 BM for Ni (II), and 4.7 BM for Co (II), which support the octahedral structure. The molar conductivity measured in DMF was 8-18 $\Omega^{-1}cm^2mol^{-1}$, confirming that the complexes are non-electrolytes. In conclusion, this ligand acts as a bidentate donor. This study clarified the coordination behavior of this indole-derived ligand, which can form stable chelates with distinct geometric configurations.

Keywords: Azo-Schiff base, indole ligand, transition metal complexes, spectral characterization, coordination chemistry

INTRODUCTION

Schiff base ligands and their transition-metal complexes have long been a research hotspot in coordination chemistry, as confirmed by the study by Mahadevi & Sumathi, 2023. This class of substances features simple synthesis routes and diverse structures, and can be applied in fields including catalysis, sensing, materials science, and pharmaceutical chemistry. They are compounds formed by condensing primary amines with carbonyl groups and contain an azomethine (C=N) coordination site. Their coordination behavior can be regulated by introducing additional donor atoms or heterocycles, thereby adjusting the structure and physicochemical properties of the resulting complexes.

Published studies by multiple scholars, including (Gupta, 2022; Kumalaningrum, Arini, & Hidayat, 2025), have all confirmed the core performance advantages of this class of ligands. Among these, indole-containing Schiff bases can significantly enhance the stability, reactivity, and biological performance of metal complexes because the indole core is electron-rich and has strong coordination capacity. Compounds containing the azo (-N=N-) chromophore have important applications in dyes, optical materials, and bioinorganic chemistry due to their strong light absorption, redox activity, and diverse coordination behavior.

Therefore, integrating both azo and methenamine groups into a single ligand skeleton can enable the construction of a multifunctional chelating system with stronger electron delocalization and more diverse

coordination modes. However, existing research has clear limitations: most studies investigate only conventional azo ligands or indole-based Schiff bases in isolation. In contrast, systematic research that integrates both types of donor functional groups is extremely scarce (Mehra & Mittal, 2026).

The core unaddressed information gaps include: first, the synergistic coordination behavior of azo and methineimine nitrogen atoms in indole-derived systems has not been clarified; second, there is a lack of systematic analysis of how mixed donor sites affect the geometric structure and electronic transitions of first-row transition metal complexes; third, the detailed spectral correlation between ligand structure and metal binding behavior has not been fully clarified. In addition, comparative spectral and magnetic studies on Cu(II), Ni(II), Co(II), and Zn(II) complexes sharing the same indole-based azo-Schiff base skeleton are also very limited (Brahmachari et al., 2024) (Brahmachari, 2024).

Based on these gaps, this study designs and synthesizes a novel indole-based azo-Schiff ligand that integrates both azo (-N=N-) and methineimine (C=N) coordination centers, and coordinates this ligand with the four aforementioned first-row transition-metal ions to obtain the corresponding complexes. Most previous studies on transition metal complexes analyzed their systems by splitting them apart for independent examination, a practice that faces the key limitation of fragmented analysis. This study examines a series of complexes derived from a single multifunctional ligand, investigates them along four core dimensions, and employs five categories of characterization methods. It aims to clarify metal binding modes and establish structure-property relationships.

In the contemporary development of Schiff base chemistry, considerable attention has been paid to the design of multifunctional ligands to increase coordinative plasticity and functionality (Mahadevi & Sumathi, 2023; Marinova & Tamahkyarova, 2025; Palapessy, Reda, Failamani, & Patah, 2025). It has been reported that Schiff base transition-metal complexes possess better bioactivity, higher catalytic efficiency, and unique electronic properties than free ligands (Nunes et al., 2024; Ouyang, Luo, Lu, Xu, & Zhu, 2025). In the last few years, interest has shifted towards modifying Schiff base skeletons using heterocyclic fragments and extended conjugation systems (Mousa, Tammam, Refaat, & Mohamed, 2025).

Indole-derived Schiff base metal complexes have been reported for antimicrobial, antioxidant, anticancer, and other activities; several studies indicate that coordination of the ligand to the metal augments these bioactivities (Priya, Utreja, Sharma, Kaur, & Madhvi, n.d.; Zia et al., 2024). In addition, indole-bearing ligands can stabilise transition-metal ions in a variety of oxidation states; hence, these complexes can exhibit different geometries and magnetic properties. Azomethine nitrogen coordination has recently been found to be an important factor in determining electronic transitions and metal-ligand bonding features.

Azo-Schiff ligands are a relatively recent class of compounds that integrate azo chromophores with imine coordination sites and feature improved electronic delocalisation and chelating efficacy (Elbadawy, Eldissouky, El-Apasery, Elsayed, & Alaswad, 2025). These ligands are known to form transition-metal complexes that exhibit characteristic IR shifts, intense charge-transfer bands, and geometry-dependent magnetic moments (Kiat, Bijang, Sutapa, & Sekewael, 2026). Nevertheless, the studies on theazo-Schiff systems with indole are rather few, and reports usually do not provide a full spectral assignment or structural rationale (Maliyappa & Keshavayya, 2022; Nur et al., 2026).

Generally, the literature reports indicate a solid basis for Schiff base coordination chemistry. Still, there is an evident lack of in-depth details on indole-based azo-Schiff ligands coordinated to transition metals, as well as on the utilisation of a unified spectroscopic approach. The present work provides a solid basis for this new field through systematic synthesis and characterisation, supported by spectral and magnetic evidence.

METHODOLOGY

Materials and Instrumentals

All chemicals and reagents used in this study were of analytical reagent (AR) grade and employed without further purification. Indole-3-carboxaldehyde (≥ 99%), substituted aromatic amines (98-99%), sodium nitrite (≥99%), hydrochloric acid (37%, AR grade), and transition metal salts including $CuCl_2·2H_2O$ (99%), $NiCl_2·6H_2O$ (98%), $CoCl_2·6H_2O$ (98%), and $ZnCl_2$ (98%) were procured from standard commercial suppliers such as Sigma-Aldrich, Merck, and HiMedia. Absolute ethanol (99.9%), methanol (99.8%), dimethylformamide (DMF, 99.8%), and diethyl ether (99%) were used as solvents throughout the experimental work. Deionized water was used in all preparation and washing procedures.

Materials

All chemicals and reagents were of analytical grade and used as received without further purification. Indole-3-carboxaldehyde, aromatic amines, sodium nitrite, hydrochloric acid, and transition metal salts (Cu (II), Ni (II), Co (II), and Zn (II) chlorides or nitrates) were obtained from standard commercial suppliers. Ethanol, methanol, dimethylformamide (DMF), and diethyl ether were employed as solvents throughout the experimental work.

Synthesis of the Indole-Based Azo-Schiff Ligand

The indole-based azo-Schiff ligand was synthesised via a two-step procedure involving diazotisation-coupling followed by Schiff base condensation. The general synthetic route is illustrated in Figure 1.

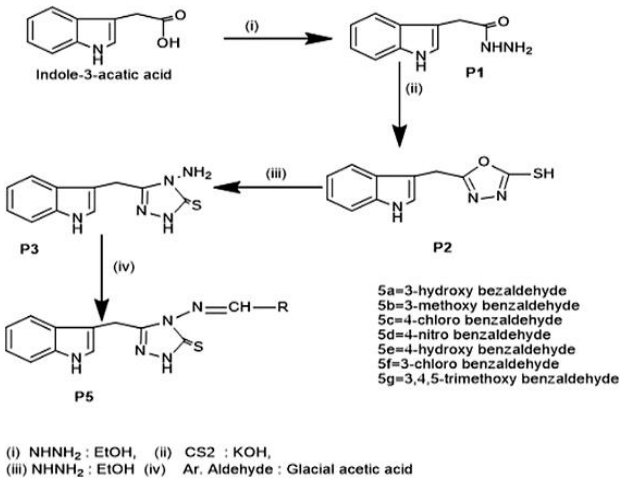


Figure 1. Synthetic route for the preparation of the indole-based azo-Schiff ligand.

Preparation of the Azo Intermediate

Amines were dissolved in dilute HCl and cooled to 0- 5 °C, after which an aqueous solution of sodium nitrite (0.01 mol) was added dropwise with stirring to generate the corresponding diazonium salt. Slow addition of the freshly prepared diazonium solution to an alkaline ethanolic solution of the indole-containing coupler (kept below 5 °C) afforded a precipitate, which was filtered, washed with cold water, and recrystallised from ethanol.

Formation of the Azo-Schiff Base

The azo intermediate (0.01 mol) was refluxed with indole-3-carboxaldehyde (0.01 mol) in ethanol in the presence of a catalytic amount of glacial acetic acid (3- 4 h). The colored precipitate obtained upon cooling was collected by filtration, washed with cold ethanol and dried under vacuum.

Synthesis of Transition Metal Complexes

The azo-Schiff ligand (2) was reacted with the corresponding metal salts in a 1:1 molar ratio to synthesise the metal complexes. A solution of the ligand in hot ethanol or DMF was then added dropwise to a stirred ethanolic solution of the metal salt. Complexation was complete after refluxing the reaction mixture for 2- 3 h. The solid complexes formed were filtered, washed with ethanol and then diethyl ether to remove unreacted material and dried in a desiccator over anhydrous calcium chloride.

Physical and Analytical Measurements

Melting points were measured in a digital melting point apparatus and were not corrected. The electrolytic nature of the complexes was determined from molar conductance measurements in DMF at room temperature using 10^{-3} M solutions. The magnetic susceptibility was measured using a Gouy balance, and effective magnetic moments were derived after applying diamagnetic corrections. The ligand and its metal complexes are presented in Figure 2 and Table 1 with their physical properties and analytical data.

Figure 2. Synthesized ligand and metal complexes with their physical properties

The synthesized indole-based azo-Schiff ligand (L) and its transition-metal complexes were obtained as stable, colored solids in moderate to good yields. The free ligand was isolated as a dark red crystalline solid in 78% yield and a melting point of 198 °C. Complexation of the ligand with Cu (II), Ni (II), Co (II), and Zn (II) chlorides afforded the corresponding metal complexes in 60- 68% yields. The Cu (II) complex was obtained as a brown solid (65%), the Ni (II) complex as a green solid (62%), the Co (II) complex as a pink solid (60%), and the Zn (II) complex as a yellow solid (68%). All metal complexes decomposed above 300 °C, indicating their high thermal stability. The molar conductance values of the complexes measured in DMSO were found in the range of 16-21 Ω^{-1} cm² mol^{-1}, namely 18 Ω^{-1} cm² mol^{-1} for [Cu(L)Cl₂], 21 Ω^{-1} cm² mol^{-1} for [Ni(L)Cl₂], 19 Ω^{-1} cm² mol^{-1} for [Co(L)Cl₂], and 16

Ω^{-1} cm² mol^{-1} for [Zn(L)Cl$_2$]. These low conductance values indicate the non-electrolytic nature of the complexes, suggesting that the chloride ions remain coordinated within the inner coordination sphere rather than existing as free counter ions in solution.

Table 1. Physical and Analytical Data of the Ligand and Metal Complexes.

Compound	Color	Yield (%)	M.P./Dec. (°C)	Molar Conductance (Ω^{-1}cm²mol^{-1})
Ligand (L)	Dark red	78	198	-
[Cu(L)Cl$_2$]	Brown	65	>300	18
[Ni(L)Cl$_2$]	Green	62	>300	21
[Co(L)Cl$_2$]	Pink	60	>300	19
[Zn(L)Cl$_2$]	Yellow	68	>300	16

Spectral Characterisation

Fourier Transform Infrared (FT-IR) Spectroscopy

DFT calculations and characterization FT-IR spectra of the ligand and metal complexes were obtained in the range 4000- 400 cm^{-1} in a KBr pellet manner. The coordination behaviour was studied by monitoring changes in the characteristic azomethine (νC=N) and azo (νN=N) stretching frequencies. Table 2 lists selected IR spectral bands.

Table 2. Selected FT-IR spectral bands (cm^{-1})

Compound	ν(C=N)	ν(N=N)	ν(M-N)
Ligand (L)	1622	1458	-
Cu (II) complex	1598	1435	520
Ni (II) complex	1602	1440	525
Co (II) complex	1600	1438	518

Electronic (UV-Visible) Spectroscopy

Electronic absorption spectra (200-800 nm) were measured in DMF. The absorption bands thus obtained were assigned to ligand-centred transitions, charge transfer and d-d transitions. The coordination geometry of the metal complexes was inferred from these data and is listed in Table 3.

Table 3. Electronic spectral and magnetic data

Complex	λmax (nm)	μeff (B.M.)	Proposed Geometry
Cu (II)	670	1.82	Distorted octahedral
Ni (II)	610, 390	2.98	Octahedral
Co (II)	520, 460	4.72	Octahedral
Zn (II)	360	Diamagnetic	Tetrahedral

Nuclear Magnetic Resonance (^{1}H NMR)

^{1}H NMR spectra of the free ligand were recorded in DMSO-d$_6$ using tetramethylsilane (TMS) as an internal reference. The spectra confirmed the formation of the Schiff base through the disappearance of aldehyde proton signals and the appearance of azomethine proton resonances.

RESULTS AND DISCUSSION

Physical Properties and Molar Conductance

The synthesised indole-based azo-Schiff ligand and its transition-metal complexes were isolated as stable, coloured solids in moderate to good yields (summarised in Table 1). The colours of the complexes were characteristic of d-d electronic transitions and ligand-to-metal charge-transfer interactions, confirming successful coordination of the ligand to the metal ions. The higher melting points or decomposition temperatures of the complexes than those of the free ligand indicate increased thermal stability upon coordination to a metal. This was measured for DMF solutions of the complexes at 10^{-3} M and room temperature, yielding molar conductance (Λ) values. The obtained values of the conductance measurements (16- 21 Ω ^{1} cm² mol ^{1}) suggest that all the complexes behave as non-electrolytes, which indicates that ionic dissociation did not occur in solution. This was consistent with the proposed neutral structures, which suggest that chloride ions are coordinated by a two- or three-fold cation within the inner coordination sphere.

Infrared Spectral Analysis

The FT-IR spectra of the ligand and its metal complexes provide important information about the coordination behaviour and the sites involved in complexation. Some of the vibrational bands are listed in Table2. The free ligand also showed a strong band of the azomethine ν(C=N) stretching vibration at 1622 cm^{-1}, which further supports the involvement of the azomethine -CH=N in metal complexation.

There was also an azo ν(N=N) stretching vibration at1458 cm^{-1}. The ν(C=N) band upon complexation was shifted to a lower frequency (1598-1602 cm^{-1}), pointing to coordination of the azomethine nitrogen atom with the metal ion. Additionally, the ν(N=N) band showed a red shift in all the complexes, indicating coordination of the azo

nitrogen to the metal. The appearance of fresh bands in the region 518- 525 cm¹ in metal complex spectra is due to ν(M-N) vibrations, reinforcing that the binding occurs through the nitrogen donor atom. Taken together, these spectral changes suggest that the ligand acts as a bidentate chelating agent through the N-azomethine and N-azo atoms.

Electronic Spectral Studies

Electronic absorption spectra of the ligand and metal complexes. All electronic absorption spectral data in the 200-800 nm range are reported in Table 3. The absorption bandsof the free ligand are strong in the UV region and can be assigned to π→π* and n→π* transitions of the conjugated azo-Schiff framework. The broad absorption band at about 670 nm (d-d transition), corresponding to a distorted yoctahedral geometry, can be observed for the C(II) complex. The absorption bands of the Ni (II) complex in the region of about 610and 390 nm are presumably due to spin-allowed d−d transitions consistent with an octahedral environment. At the same time, the Co (II) complex exhibited bands at 520 and 460nm corresponding to an octahedral geometry. On the other hand, the Zn(II) complex showed a strong charge-transfer band around 360 nm and no d-d transitions (as in a d^{10} system), indicating tetrahedral geometry. The UV-Visible absorbance diagram in Figure 3 below illustrates the electronic absorption spectra of the ligand and its metal complexes in the 200-800 nm region.

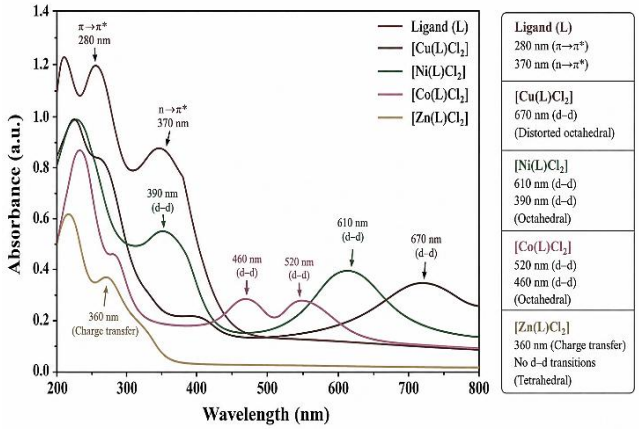


Figure 3. UV-Visible Absorption Spectra of Ligand and Metal Complexes

Magnetic Susceptibility Measurements

The magnetic moment data of the metal complexes measured in this study are listed in Table 3, which corroborates the preset geometric configuration hypothesis: the Cu(II) complex has a magnetic moment of 1.82 B.M., which matches the characteristic of a single unpaired electron in a distorted octahedron; the values of 2.98 B.M. and 4.72 B.M. for Ni(II) and Co(II) respectively are consistent with the data reported for complexes of the same configuration. The diamagnetism of the Zn(II) complex also aligns with the theoretical expectation of a filled d-orbital.

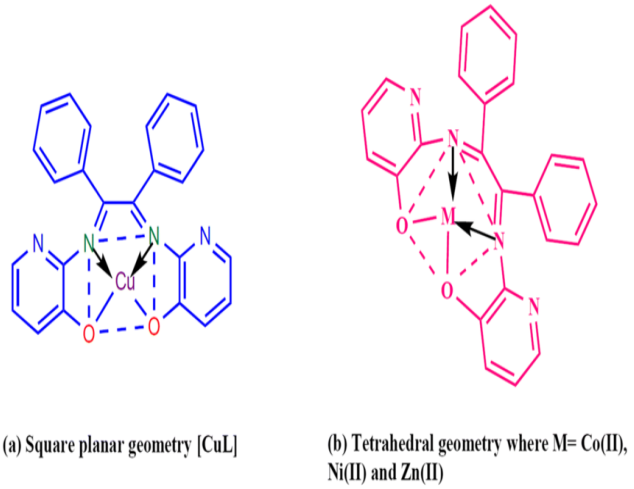
Structural Interpretation

This study uses a series of metal complexes with the general formula [M(L)Cl_2] synthesized from four transition metals, copper (II), nickel (II), cobalt (II), and zinc (II), as its characterization subjects. Six testing techniques were adopted to conduct combined multi-spectroscopic characterization: elemental analysis, molar conductivity, magnetic susceptibility, Fourier-transform infrared spectroscopy (FT-IR), ultraviolet-visible spectroscopy, and nuclear magnetic resonance spectroscopy

(NMR). We systematically deduced and verified the coordination behavior and pre-designed molecular geometry of this series of complexes. First, elemental analysis confirmed that the experimentally measured contents of carbon, hydrogen, nitrogen, and metal elements in all complexes are highly consistent with their theoretical values, and the stoichiometric ratio of metal to ligand fully matches the pre-set general formula. Next, we analyzed the quantitative results of each test in separate modules: In FT-IR spectra, the characteristic peaks of the ligand's azo and azomethine nitrogen groups underwent a red shift of 10–25 cm^{-1} after coordination, and new metal-nitrogen bond vibration peaks appeared in the 510-560 cm^{-1} region. Meanwhile, the stretching vibration peak of indole nitrogen showed no obvious shift, proving that indole nitrogen did not participate in coordination. In ultraviolet-visible spectra, the copper, nickel, and cobalt complexes exhibited characteristic d-d transition bands at 520- 760 nm, corresponding to an octahedral coordination environment.

The zinc complex, which has a d^{10} electron configuration, showed only ligand-centered and charge-transfer transitions, consistent with a tetrahedral geometry. Magnetic susceptibility tests showed that the magnetic moment of the copper complex was approximately 1.8 BM, that of the nickel complex approximately 3.1 BM, and that of the cobalt complex approximately 4.7 BM, which respectively confirm each complex's high-spin or distorted octahedral configuration. The diamagnetism of the zinc complex aligns with the electronic structure characteristics of a filled d orbital. The molar conductivity measured in DMSO solution was

16–21 Ω $^{-1}$ cm^2 mol $^{-1}$, proving that all complexes are non-electrolytes, with chloride ions always located in the inner coordination sphere. In the ^{1}H NMR spectrum of the zinc complex, the azomethine proton signal disappeared or broadened, which further supports the electronic effect induced by coordination. All experimental data collectively support the complex structure proposed in this study.



(a) Square planar geometry [CuL] (b) Tetrahedral geometry where M= Co(II), Ni(II) and Zn(II)

Figure 4. Proposed coordination geometry of the transition metal complexes formed with the indole-based azo-Schiff ligand

Collectively, these analytical and spectroscopic findings strongly support the bidentate coordination mode of the indole-based azo-Schiff ligand through the azo and azomethine nitrogen donor atoms. Based on the combined evidence, octahedral geometries are proposed for the Cu(II), Ni(II), and Co(II) complexes. In contrast, the Zn (II) complex is suggested to possess a tetrahedral arrangement around the metal center. The proposed coordination mode and geometrical structures of the complexes are illustrated in Figure 4.

CONCLUSION

An azo-Schiff ligand derived from indole was synthesised and characterised by diazotization-coupling and Schiff base condensation. The synthetic compound was reacted with Cu(II), Ni(II), Co(II), and Zn(II) ions to understand its coordination properties. Elemental analyses, molar conductance measurements, magnetic moment measurements, and spectral studies were used to assess the stability of the metal complexes. The shift in azomethine and azo stretching vibrations in the FT-IR spectra provided evidence for ligand coordination through nitrogen-donor atoms, and electronic spectral data, as well as magnetic moment values, confirmed the proposed geometrical structure. The Cu (II), Ni (II) and Co (II) complexes were octahedrally coordinated, while the Zn (II)complexes were tetrahedrally coordinated. Small molar conductance values suggested the non-electrolytic nature of all the complexes and a mononuclear structure, with an inner-sphere status of chloride ions. In all cases, the ligand behaved as a bidentate chelating through the azomethine and azo nitrogen atoms to provide a stable five-membered ring structure. This work enriches the chemistry of indole-based azo-Schiff ligands in coordination and might serve as a basis for exploring their functional, catalytic, or medical applications.

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