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Synthesis and Characterization of Transition Metal Complexes Derived from Benzil Ethylenediamine and Isonitrosopropiophenone: A Spectroscopic Investigation

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Abstract: *A new Schiff base was prepared and used as a coordinating agent to form a series of transition-metal complexes. The ability of the Schiff base to bind metal ions was investigated through spectroscopic and magnetic studies. Infrared and UV–visible spectroscopy were used to confirm coordination between the Schiff base and the metal ions, while magnetic moment measurements provided information about the electronic environment around the metal centers. The combined results demonstrate that the Schiff base forms stable metal complexes with well-defined electronic properties. This work highlights the usefulness of Schiff base ligands in coordination chemistry and their potential for developing new metal-based materials with interesting physicochemical characteristics*

Keywords: Schiff base, transition metal complexes, coordination chemistry, spectroscopic characterization, magnetic properties, metal–ligand interactions

I. INTRODUCTION

Schiff base complexes occupy a significant position in coordination chemistry due to their versatile applications in organic synthesis, catalysis, and medicinal chemistry[1,2]. These compounds, characterized by the azomethine group (-C=N-), are capable of stabilizing various metal ions in different oxidation states, making them valuable for mimicking biological systems and developing new materials .

The chemistry of oxime-based ligands has gained particular attention in recent years. Isonitrosopropiophenone (NPP-H), an oxime derivative, has been studied for its ability to coordinate with metal ions and participate in dioxygen-dependent oxidation reactions [3,4]. Research by Bhattacharya and colleagues demonstrated that iron(II) complexes containing isonitrosopropiophenone can activate dioxygen and facilitate the oxidative transformation of oximes to nitric oxide or nitroxyl, highlighting the biological relevance of such systems [4].

Benzil ethylenediamine derivatives have been widely employed as precursors in macrocyclic chemistry. Reddy and co-workers utilized bis(benzil)ethylenediamine in template synthesis to prepare cobalt(II) complexes with varying ring sizes, establishing the coordination behavior of such ligand systems[5] . Similarly, Bansal and colleagues reported the synthesis of lead(II) macrocyclic complexes using bis(benzil)ethylenediamine as a precursor, confirming the involvement of amido nitrogen in coordination to the central metal ion [6].

In the present work, we report the synthesis of a Schiff base ligand derived from benzil ethylenediamine and isonitrosopropiophenone, followed by the preparation of its transition metal complexes with Ni(II), Cu(II), Co(II), and Fe(II). The characterization of these complexes through spectroscopic techniques and magnetic measurements forms the core of this investigation.

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II. EXPERIMENTAL

2.1 Materials and Methods

All chemicals used were of analytical reagent grade. Benzil, ethylene diamine, and isonitrosopropiophenone were procured from commercial sources and used without further purification. Metal salts (nickel chloride, copper chloride, cobalt chloride, and ferrous ammonium sulfate) were obtained from standard suppliers.

2.2 Synthesis of Ligand

Step 1: Synthesis of Benzil Ethylenediamine Intermediate

Benzil was reacted with ethylene diamine in appropriate stoichiometric ratio. The reaction mixture yielded an orange compound, which was isolated and purified. This intermediate (bis(benzil)ethylenediamine) serves as a precursor for further condensation, consistent with previously reported methods for macrocyclic ligand synthesis[1,4,5] .

Step 2: Synthesis of Final Ligand

The orange intermediate was reacted with isonitrosopropiophenone and refluxed for one hour. Upon cooling, yellow-colored crystals were obtained. The product was recrystallized using toluene to obtain pure ligand. The role of isonitrosopropiophenone in such systems has been documented in bioinorganic chemistry, particularly in iron-mediated oxidation reactions .

2.3 Synthesis of Metal Complexes

The metal complexes were prepared by reacting the ligand with metal salts in 2:1 ligand-to-metal stoichiometry (ML_2). The following complexes were synthesized:

· Nickel(II) complex: Brick red color
· Copper(II) complex: Green-brown color
· Cobalt(II) complex: Green color
· Iron(II) complex: Blue color

The reaction mixtures were refluxed appropriately, and the resulting complexes were filtered, washed, and dried.

2.4 Physical Measurements

Infrared spectra were recorded in the range 4000-400 cm^{-1} using KBr pellets. Electronic spectra were recorded in the UV-Visible region (200-800 nm) using appropriate solvents. Magnetic susceptibility measurements were carried out at room temperature using a Gouy balance, and effective magnetic moments (μ_eff) were calculated.

III. RESULTS AND DISCUSSION

3.1 Synthesis and General Properties

The ligand synthesis proceeded through two distinct steps, yielding an orange intermediate followed by yellow crystalline product. The color transitions observed during complexation are characteristic of d-d transitions and charge transfer phenomena in transition metal complexes[3,4]. Similar color variations have been reported for Schiff base complexes with various metal ions [1].

The ML_2 stoichiometry (2:1 ligand-to-metal ratio) is consistent with the coordination behavior of Schiff base ligands derived from diamines, where the ligand typically acts as a tetradentate or bidentate donor depending on the reaction conditions[5,6].

3.2 Infrared Spectral Studies

Table 1: Characteristic IR Absorption Bands (cm^{-1}) of Ligand and Metal Complexes

Compound	ν(C=N)	ν(C-O)	ν(N-O)	ν(M-N)	ν(M-O)
Ligand	1640	1350	1250	----	---
Ni(II) complex	1615	1330	1235	510	595

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Cu(II) complex	1610	1325	1240	505	590
Co(II) complex	1620	1335	1232	515	600
Fe(II) complex	1605	1320	1225	500	585

The IR spectrum of the free ligand showed a strong band at 1640 cm^{-1} attributable to the azomethine (C=N) stretching vibration. Upon complexation with metal ions, this band shifted to lower frequencies (1605-1620 cm^{-1}), indicating coordination of the azomethine nitrogen to the metal center . This shift is consistent with the reduction in electron density due to donation of the lone pair of electrons on nitrogen to the metal ion[5,6] .

Bands in the region 1350-1250 cm^{-1} correspond to C-O and N-O stretching vibrations from the isonitrosopropiophenone moiety. The shift in these bands upon complexation suggests involvement of oxygen atoms in coordination. Similar observations have been reported for Schiff base complexes where phenolic or oxime oxygen participates in metal-ligand bonding. The appearance of new bands in the regions 500-515 cm^{-1} and 585-600 cm^{-1} corresponds to ν(M-N) and ν(M-O) stretching vibrations, respectively, confirming the formation of metal-ligand coordinate bonds[7,8] .

3.3 Electronic Spectral Studies

The electronic spectrum of the free ligand exhibited absorption bands at approximately 280 nm and 340 nm, assigned to π→π* transitions (involving molecular orbitals localized at the azomethine linkage) and n→π* transitions, respectively[3] . These bands are characteristic of Schiff base ligands containing conjugated systems.

In the metal complexes, the π→π* and n→π* transitions showed slight shifts, indicating coordination of the ligand to metal ions. Additionally, new absorption bands appeared in the visible region (500-610 nm), corresponding to d-d transitions. These bands are responsible for the characteristic colors observed for each complex [7].

The nickel(II) complex showed a d-d transition band around 520 nm, suggesting an octahedral geometry. Copper(II) complex exhibited a broad band around 580 nm, characteristic of distorted octahedral geometry due to Jahn-Teller distortion[3]. Cobalt(II) complex displayed multiple bands in the 550-610 nm region, consistent with octahedral geometry[8]. The iron(II) complex showed a band around 570 nm, also indicative of octahedral coordination[4] .

3.4 Magnetic Moment Measurements

Table 2: Magnetic Moment Data and Geometry Assignment

Complex	μ_eff (B.M.)	Number of Unpaired Electrons	Geometry
Ni(II)complex	3.12	2	Octahedral
Cu(II) complex	1.86	1	Distorted octahedral
Co(II) complex	4.98	3	Octahedral (high-spin)
Fe(II) complex	5.42	4	Octahedral (high-spin)

The magnetic moment values provide crucial information about the geometry and electronic configuration of the metal complexes. The nickel(II) complex showed a magnetic moment of 3.12 B.M., which is within the range expected for octahedral nickel(II) complexes with two unpaired electrons. Octahedral Ni(II) complexes typically exhibit μ_eff values in the range 2.9-3.3 B.M. due to orbital contribution [2,6].

The copper(II) complex exhibited a magnetic moment of 1.86 B.M., close to the spin-only value of 1.73 B.M. for one unpaired electron. This value is typical for mononuclear copper(II) complexes with d^9 configuration and suggests distorted octahedral geometry [3,4].

The cobalt(II) complex showed a magnetic moment of 4.98 B.M., which is characteristic of high-spin octahedral cobalt(II) complexes. High-spin octahedral Co(II) (d^7) typically exhibits μ_eff in the range 4.7-5.2 B.M. due to significant orbital contribution [5,6].

The iron(II) complex displayed a magnetic moment of 5.42 B.M., indicating high-spin octahedral geometry. High-spin octahedral Fe(II) (d^6) typically shows μ_eff values around 5.1-5.7 B.M., consistent with four unpaired electrons [4,6].

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3.5 Structural Interpretation

Based on the combined spectroscopic and magnetic data, an octahedral geometry is proposed for all the complexes, with the copper(II) complex exhibiting distortion due to Jahn-Teller effects. The ligand coordinates through azomethine nitrogen and oxygen atoms (from the isonitrosopropiophenone moiety), forming stable five- or six-membered chelate rings[2,8].

The ML_2 stoichiometry suggests that two ligand molecules satisfy the coordination requirements of the metal ion, with additional sites possibly occupied by solvent molecules or anions, as commonly observed in such systems[1,5].

The formation of these complexes can be understood in terms of the hard-soft acid-base concept, where the borderline Ni(II), Cu(II), Co(II), and Fe(II) ions interact effectively with nitrogen and oxygen donor atoms of the ligand[3,7].

IV. CONCLUSION

A novel Schiff base ligand derived from benzil ethylenediamine and isonitrosopropiophenone was successfully synthesized and characterized through its transition metal complexes with Ni(II), Cu(II), Co(II), and Fe(II). The complexes exhibited distinct colors ranging from brick red to blue, reflecting their unique electronic structures.

IR spectral studies confirmed the coordination of azomethine nitrogen and oxygen atoms to the metal center, with characteristic shifts in ν(C=N) and the appearance of new ν(M-N) and ν(M-O) bands[2,3,6]. UV-Visible spectroscopy revealed $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions in the ligand, while d-d transitions in the visible region accounted for the complex colors. Magnetic moment measurements established high-spin octahedral geometries for Ni(II), Co(II), and Fe(II) complexes, while the Cu(II) complex showed distorted octahedral geometry[4,8].

The present work contributes to the understanding of Schiff base complexes derived from benzil ethylenediamine and oxime-based ligands. Future studies may explore the biological activities, catalytic properties, and redox behavior of these complexes, given the growing interest in such systems for bioinorganic and materials chemistry applications[1,4,5] .

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