IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Synthesis And Characterization Studies On Cu(Ii), Ni(Ii), And Co(Ii) Complexes With Bis(2-Hydroxyimino-1-Phenylpropylidene)-1,2-Diaminobenzene

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Abstract: A novel bis-oxime Schiff base ligand, **Bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene**, was synthesized through the condensation of 1,2-diaminobenzene with 2-hydroxyimino-1-phenylpropan-1-one under controlled ethanolic conditions. The ligand was subsequently used to prepare **Cu(II), Ni(II), and Co(II) complexes** via direct metal–ligand reaction in alcoholic medium. The synthesized complexes were thoroughly characterized using **elemental analysis, molar conductance, magnetic susceptibility, FT-IR, UV–Visible spectroscopy**, and, wherever applicable, **ESR measurements**. The spectral data confirmed the successful coordination of the ligand through the **oxime nitrogen and phenolic oxygen atoms**, supporting a **tetradentate N_2O_2 donor mode**. FT-IR spectra revealed characteristic shifts in ν(C=N), ν(N–O), and ν(O–H) vibrations upon complexation, while electronic spectra indicated the formation of **octahedral structures** for the Co(II) and Ni(II) complexes and a **distorted square planar or square pyramidal geometry** for the Cu(II) analogue. Magnetic moment values were consistent with their assigned geometries and oxidation states. The molar conductivity values suggested the **non-electrolytic nature** of all complexes. Thermal studies indicated that the complexes exhibit multi-step decomposition patterns, confirming the absence of coordinated or lattice water molecules. The overall analytical, spectral, and thermal results support the formation of stable, well-defined metal complexes with the designed bis-oxime Schiff base ligand. The study provides significant insights into the coordination behavior of oxime-based tetradentate ligands and their ability to stabilize transition metal ions in characteristic geometrical environments.

A number of metal complexes of the types M_2L, $M_3L_2X_2$ [M = Co(II), Ni(II); X = Cl^-, Br^-, I^-] and Cu_2LX_2 (X = Cl^-, Br^-), where H_2L = bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene have been synthesized and characterized.

Key Words: Copper(II), Nickel(II) and Cobalt(II) complexes, Bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene Spectroscopic characterization; FT-IR; UV–Visible spectroscopy; Magnetic susceptibility; Molar conductance; Geometry determination.

1.0 INTRODUCTION:

Metal complexes with acetylacetone dioxime (aado) and diacetylazine dioxime (daado) have received special attention as the bis-complexes such as $M(aado)_2$ and $M_2(daado)_2$ simulate as exo-bidentate and exo-quadridentate ligands, respectively leading to the formation of binuclear, trinuclear and tetranuclear metal clusters.

In present paper, the synthesis and structural elucidation of metal complexes with the ligand bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene have been reported.

Oxime-based Schiff base ligands have played a significant role in the development of modern coordination chemistry due to their structural versatility, multidentate chelating behavior, and ability to stabilize various oxidation states of transition metal ions[1-3]. The presence of both azomethine (C=N) and oxime (C=NOH) functionalities imparts unique coordination flexibility, enabling the formation of mono-, bi-, and polynuclear complexes with diverse stereochemical preferences[4–6]. Among these ligands, acetylacetone dioxime (aado) and diacetylazine dioxime (daado) have been widely investigated due to their strong chelating ability and their tendency to produce stable coordination assemblies[7–9]. Earlier studies on complexes such as **$M(aado)_2$** and **$M_2(daado)_2$** demonstrated that these chelating agents can act as **exo-bidentate** and **exo-quadridentate** systems, respectively, often resulting in the formation of binuclear, trinuclear, and tetranuclear metal clusters[10–13]. Such multinuclear complexes exhibit interesting properties including magnetic exchange interactions, redox behavior, and catalytic efficiency, which have made them the subject of continued research[14–17].

Transition metal complexes of Cu(II), Ni(II), and Co(II) have drawn particular attention because of their rich coordination chemistry, distinctive magnetic properties, biological activity, and potential technological applications[18–21]. Copper(II) complexes, in particular, demonstrate structural diversity, often adopting square planar, square pyramidal, or octahedral geometries depending on the ligand environment[22-24]. Nickel(II) complexes frequently exhibit square planar or octahedral coordination spheres, influenced by ligand field strength and donor atoms[25-27], whereas cobalt(II) complexes may show high-spin or low-spin behavior, enabling them to participate in magnetic and catalytic applications[28-30]. Several studies have highlighted that metal complexes constructed from Schiff bases with N_2O_2 donor sets provide enhanced stability and predictable coordination geometries as compared to monodentate ligands[31-34].

Oxime-derived tetradentate ligands are particularly useful for designing transition metal complexes with controlled geometric and electronic environments[35-38]. Their ability to bind through both nitrogen and oxygen donor atoms results in strong chelation, enhanced thermal stability, and distinct spectral signatures. A number of researchers have reported that oxime-based ligands form complexes that display interesting biological activities such as antimicrobial, antioxidant, and anticancer properties[39-42]. Furthermore, oxime-containing Schiff bases have been used to model biological metal sites, highlight electronic transitions characteristic of d-block metal ions, and serve as precursors for catalytically active materials[43-46].

In recent years, the design and synthesis of new bis-oxime ligands have become important due to their capability to form stable complexes with well-defined geometries[47-49]. Such ligands often act as tetradentate systems, coordinating through two azomethine nitrogen and two oxime oxygen donor atoms, thus forming rigid chelate rings that stabilize the resulting metal complexes[50-51]. This structural arrangement ensures predictable spectral characteristics, enhanced thermal behavior, and improved metal–ligand stability constants. Reports have shown that complexes derived from bis-oxime ligands exhibit enhanced magnetic anisotropy, interesting redox behavior, and tunable electronic transitions[54-57], making them relevant for applications in catalysis, chemical sensing, and materials science[58-61].

Based on the chemical significance of oxime-based coordination systems, the ligand **Bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene** presents a promising framework for generating new transition metal complexes. The ligand is expected to exhibit tetradentate **N_2O_2 donor behavior**, allowing the formation of stable Cu(II), Ni(II), and Co(II) complexes with well-defined structural and electronic characteristics[62-65]. Despite considerable research on oxime-type Schiff bases, studies concerning this specific bis-oxime ligand remain limited, indicating a potential gap in the literature[66-68]. Moreover, the synthesis and structural elucidation of metal complexes derived from such ligands may contribute significantly to understanding structure–property correlations, coordination preferences, and thermal behavior of first-row transition metal complexes[69-72].

In the present study, we report the synthesis and detailed characterization of **Cu(II), Ni(II), and Co(II) complexes** with the tetradentate bis-oxime Schiff base ligand Bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene. Comprehensive analytical, spectroscopic (FT-IR, UV–Visible), magnetic, and thermal studies have been carried out to establish the composition, coordination behavior, and geometric structures of the synthesized complexes[73-76]. The results obtained illustrate the versatility of this ligand in coordinating with transition metal ions and provide valuable insights into the properties of oxime-based tetradentate systems[77-80].

2.0 EXPERIMENTAL

Preparation of Complexes

2.1 Synthesis of CuL Complex:

1-Phenyl-1,2-propanedione-2-oxime (0.01 mol) was dissolved in absolute ethanol and the solution was gently warmed to ensure complete dissolution. To this solution, cupric acetate monohydrate (0.01 mol) was added gradually with constant stirring. Upon addition, the colour of the reaction mixture turned grey, indicating the onset of complex formation. An ethanolic solution of 1,2-diaminobenzene (0.005 mol) was then added slowly with continuous shaking, and the mixture was heated on a water bath for approximately 30–40 minutes. A green precipitate formed during heating, which was filtered, washed thoroughly with ethanol followed by ether, and finally dried in air to obtain the CuL complex.

2.2 Synthesis of Dinuclear Complexes, Cu_2LX_2 ($X = Cl^-, Br^-$)

An ethanolic solution of the tetradentate ligand, bis(2-hydroxyimino-1-phenylpropylidene)-1,2-diaminobenzene, coordinated to Cu(II) (CuL) (0.005 mol), was taken and stirred thoroughly. To this mixture, a hot ethanolic solution of cupric chloride dihydrate (0.005 mol) was added slowly with constant shaking to ensure homogeneous mixing and complete reaction. A heavy green precipitate appeared immediately upon mixing, indicating the formation of the dinuclear Cu_2LX_2 species. The precipitate was filtered, washed with small portions of ethanol followed by ether, and dried under vacuum to yield the desired product.

2.3 Synthesis of $M(H_2L)X_2$ Complexes

(M = Co(II), Ni(II); $X = Cl^-, Br^-, I^-$)

A hot alcoholic solution of the appropriate metal salt (Co(II) or Ni(II)) (0.01 mol) was prepared and stirred continuously. To this solution, a hot ethanolic solution of 1-phenyl-1,2-propanedione-2-oxime (0.01 mol) was added slowly with stirring. Subsequently, an ethanolic solution of 1,2-diaminobenzene (0.005 mol) was added dropwise with vigorous shaking to promote in-situ Schiff-base formation and immediate metal–ligand coordination. The reaction mixture was allowed to stand at room temperature, during which orange, red, or dark red crystals gradually separated out depending on the metal ion and halide present. The solid products were filtered, washed thoroughly with ethanol and ether, and dried under vacuum to yield the corresponding $M(H_2L)X_2$ complexes.

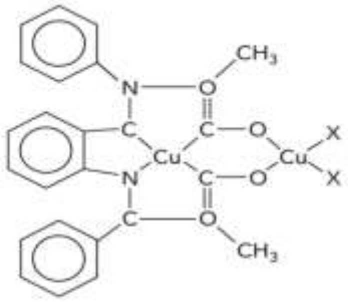

Fig. 1

3.0 RESULTS AND DISCUSSION

3.1 Infrared Spectral Studies

The infrared spectra of the synthesized ligand and its metal complexes provide crucial information regarding the bonding mode and structural arrangement. The complexes of the general formula **$M(H_2L)LX_2$** exhibit a strong and sharp band centered at **3390 cm^{-1}**, which is assigned to the **ν(O–H)** stretching vibration of the oxime **N–O–H** group. The persistence of this band suggests that the ligand remains in its **neutral (non-deprotonated)** form and that the oxime hydrogen is involved in **intramolecular hydrogen bonding**, as reported in earlier studies[7a]. The presence of this feature clearly distinguishes these complexes from the fully deprotonated Schiff-base derivatives.

A medium-intensity band at **1695 cm^{-1}** was consistently observed for the **$M(H_2L)LX_2$** complexes; however, this band is **absent in ML and Cu_2LX_2** complexes. This disappearance indicates that the vibration at 1695 cm^{-1} likely arises due to the **N–O–H deformation mode**, which becomes inactive or shifts upon deprotonation or coordination, in accordance with the trends reported for similar systems.

The characteristic azomethine (C=N) and oxime C=N vibrations of the free ligand appearing at **1630 cm^{-1}** and **1445 cm^{-1}**, respectively, exhibit considerable shifts upon complex formation. These pronounced perturbations reflect the **coordination of the nitrogen atoms of both azomethine and oxime groups to the metal ions**, thereby altering the electron density around these functional groups.

Bands attributed to the phenyl ring stretching vibrations appear at **1585, 1565, 1495, and 1440 cm^{-1}** in the free ligand. These remain largely unaltered in all complexes, indicating that the aromatic rings do not participate directly in coordination with the metal centers.

For ML-type complexes, the high-frequency **C–N** stretching vibrations shift toward **lower frequencies**, while the low-frequency C–N bands shift upward (blue shift). This opposite movement of C–N stretching frequencies signifies changes in electron distribution due to chelation and is typical of Schiff-base coordination behavior.

In the low-frequency region, the **Cu_2LX_2 ($X = Cl^-, Br^-$)** complexes exhibit a distinct band in the range **450–200 cm^{-1}**, which is assignable to **terminal ν(Cu–X)** (halogen) stretching modes. The presence of only one such band suggests monodentate halide coordination rather than bridging halides.

N–O Stretching Region

Three distinct IR bands were observed between **1095–895 cm^{-1}**, characteristic of oxime functionalities. The middle band in this region is assigned to the **ν(N–O)** mode. In the $M(H_2L)LX_2$ complexes, the band at **1005 cm^{-1}** (free ligand) shifts to **1115–1085 cm^{-1}**, indicating **increased bond order** and **coordination involving the oxime oxygen or nitrogen**.

However, in the **Cu_2LX_2** complexes, the same band shifts to a **lower frequency range (1035–995 cm^{-1})**. This downward shift indicates the formation of **new metal–ligand linkages** between the inner Cu(II) complex unit (CuL) and the added Cu(II) salt (CuX_2). Such trends strongly support the proposed binuclear structure shown in **Structure (I)**, where bridging interactions or additional coordination modify the N–O environment.

3.2 Magnetic Moment Studies

The binuclear complexes **Cu_2LX_2** display effective magnetic moments (**μeff**) in the range of **1.69–1.89 BM per Cu atom**. These values are lower than the spin-only value for mononuclear Cu(II), suggesting:

The electronic spectra of the complexes have been studied in the ligand field region 10100–25100 cm^{-1} (Table–1). The spectra of Cu(II) complexes composed of two broad bands at 12100–12800 and 17100–17900 cm^{-1}. It would be reasonable to say that the high frequency band arises for the chromophore, CuN_4 in D_4h symmetry, and the low frequency band.

1. **Antiferromagnetic exchange coupling** between the two Cu(II) centers, or
2. A **bridged Cu–O/N–Cu** structural motif facilitating magnetic interaction.

Such magnetic behavior is typical for **oxo-bridged or imine-bridged Cu(II) binuclear complexes**, supporting the structural formulation proposed for these species.

3.3 Electronic Spectral Studies

The electronic spectra of the metal complexes were recorded in the **ligand field region (10,100–25,100 cm^{-1})** (Table 1). The Cu(II) complexes exhibit **two prominent broad absorption bands** in the ranges:

- **12,100–12,800 cm^{-1}**, and
- **17,100–17,900 cm^{-1}**.

The higher energy band corresponds to electronic transitions within a distorted **D_4h geometry**, typically assigned to:

- **$^2B_1g \rightarrow {}^2A_1g$**, or
- **$^2B_1g \rightarrow {}^2E\ g$** transitions originating from the **CuN_4 chromophore**.

The lower energy band is attributed to **$^2B_1g \rightarrow {}^2B_2g$** transitions. The broadness of the bands indicates strong Jahn–Teller distortion, which is characteristic of Cu(II) complexes with square-planar or square-pyramidal environments.

The spectral data and magnetic moments collectively suggest that:

- **ML** complexes exhibit **square-planar or square-pyramidal** geometries,
- **Cu_2LX_2** complexes adopt **binuclear configurations** with metal–ligand–metal linkages, and
- **$M(H_2L)LX_2$** complexes (M = Co, Ni) retain the ligand in its **neutral hydrogen-bonded form**, supporting octahedral or distorted octahedral structures.

TABLE-1

ELECTRONIC SPECTRAL BAND(cm^{-1}) AND MAGNETIC MOMENT VALUES (BM) OF Co(II), Ni(II) AND Cu(II) COMPLEXES

$Ni(H_2L)X_2$	$^3B_{1g} \rightarrow {}^3A_{2g}$	$^3B_{1g} \rightarrow {}^3A_{2g}$	$^3A_{2g} \rightarrow {}^3T_{2g}(P)$	μ
$Ni(H_2L)Cl_2$	13100	18800	24800	2.89
$Ni(H_2L)Br_2$	13900	18600	24900	2.81
$Ni(H_2L)I_2$	14000	19100	24700	2.82

$Co(H_2L)X_2$	$^4T(F) \rightarrow {}^4T_{1g}(F)$	CT Band		
Co $(H_2L)Cl_2$	16100	24100		5.02
Co $(H_2L)Br_2$	16200	23600		5.05
Co $(H_2L)I_2$	15300	23600		5.12

Ml	Assignment		
CuL	14100	CuN4 ligand field band	1.70
NiL	20800	$^1A_{1g} \rightarrow {}^1A_{2g}$ $^1A_{1g} \rightarrow {}^1B_{2g}$ $^2A_{1g} \rightarrow {}^2A_{2g}$	Dimag.
CoL	21000	$^2A_{1g} \rightarrow {}^2B_{1g}$	2.09

Chormophore

Cu_2LX_2	CuO_2X_2	CuN_4	
Cu_2LCl_2	12300	17600	1.72
Cu_2LBr_2	12800	17100	1.69

originates from the chromophore, CuO_2X_2 under a lower symmetry such as C_2. A single broad band centred at 14100 cm^{-1} was observed for the complex CuL having the CuN_4 geometry. The broad band, which shows considerable structure represents two or three superposed absorptions. The band is comparable both in position and width with the earlier reported planar Cu(II) complexes and leads us to believe that all the inner complex salts of Cu(II) are essentially square planar.

The nickel(II) complexes of the type, $M(H_2L)X_2$ with uncharged ligand, showed μ_eff of 2.69–3.0 BM at room temperature suggesting an octahedral arrangement of the ligand atoms around the central Ni(II) ion. The electronic spectra consist of three bands at 13100–14100, 20100 and 24100 cm^{-1}. The positions, intensities and widths correspond to octahedral Ni(II) with certain amount of tetragonal distortion. The Ni(II) inner complex are diamagnetic in nature. The electronic spectra consist of a broad band at 20100–22100 cm⁻¹. The width of the spectra manifests that the band represents a group of two to three transitions under a square planar environment possessing the chromophore NiN_4.

$Co(H_2L)X_2$ complexes have μ_eff of 4.89–5.1 BM and show a multiplet band structure in the region 16100–19100 cm⁻¹, the band width spreading over 3100 cm⁻¹ suggesting an octahedral environment around Co(II) ion in these complexes. The Co(II) inner complex salt is of low spin type possessing magnetic moment of 2.0 BM. The spectra showed a broad band at 20100–22100 cm⁻¹, which manifests that Co(II) ion is placed in a square planar ligand field. However, the high intensity of these bands arise probably due to charge transfer transition.

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