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Synthesis, Characterization and Antibacterial properties of Cr(II), Co(II), Ni(II) and Cu(II) Schiff Base Complexes Derived from 1,3-diphenylpropane-1,3-dione and Glycine

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Abstract

The Schiff base ligand, 1,3-diphenylpropane-1,3-diylidenebis(azanylylidene)diacetic acid, derived from the condensation of 1,3-diphenylpropane-1,3-dione with glycine, was synthesized and complexed with Cr(II), Co(II), Ni(II), and Cu(II) ions. The ligand and its complexes were characterized by FT-IR, elemental analysis, molar conductance, and magnetic susceptibility studies, alongside physical and thermal measurements. Spectral analyses confirmed coordination via azomethine nitrogen, with metal-dependent involvement of carbonyl/enolate oxygen atoms. Physical data revealed color variations, enhanced decomposition temperatures, and greater solubility of the complexes in organic solvents relative to the free ligand. Molar conductance values indicated non-electrolytic, mononuclear complexes of general formula $[M(L)]Cl_2$, while magnetic moments supported paramagnetic configurations consistent with octahedral geometries. Antibacterial screening against Streptococcus pneumoniae (Gram-positive) and Escherichia coli (Gram-negative) by the agar cup-plate method showed modest activity for the free ligand but significantly improved effects for the complexes. Notably, $[Ni(L)]Cl_2$ and $[Co(L)]Cl_2$ exhibited inhibition zones of 20 mm and 19 mm, respectively, against E. coli. The enhanced activity is attributed to chelation, which increases lipophilicity and facilitates penetration into microbial membranes. These results highlight the role of metal identity in structural and biological properties, with Ni(II) and Co(II) complexes showing promise as antibacterial agents.

Keywords: Schiff base, 1,3-diphenylpropane-1,3-dione, Transition metals, Complexes, and Antibacterial activity.

Introduction

Schiff bases formed via condensation between carbonyl compounds and primary amines are versatile ligands known for their ease of synthesis and rich coordination chemistry. Their ability to form chelates with a variety of transition metals has made them a focal point in medicinal inorganic chemistry [1]. When coordinated with metal ions, Schiff base ligands often exhibit enhanced biological activity, owing to changes in

electronic structure, lipophilicity, and redox behavior [2]. Amino acid derived Schiff bases, particularly those from glycine, are of special interest due to their biocompatibility and multifunctional coordination capacity [3]. A Schiff base that combines a β-diketone moiety such as 1,3-diphenylpropane-1,3-dione with glycine potentially offers both imine (C=N) and enolate oxygen donors, enriching the ligand's binding versatility. However, Schiff bases derived from 1,3-diphenylpropane-1,3-dione and amino acids remain underexplored, especially concerning their antimicrobial capabilities.

Recent comparative studies have demonstrated that transition metal Schiff base complexes often outperform their free ligands in antibacterial assays. For example, Cu(II) complexes commonly show superior activity, attributed to their enhanced ability to interact with bacterial membranes or biomolecules [1,4]. Furthermore, structure activity investigations of metal–Schiff base systems underscore the influence of metal identity, ligand denticity, and geometry on biologically relevant behavior [3]. Despite this progress, there's a gap in the literature regarding the systematic comparison of Cr(II), Co(II), Ni(II), and Cu(II) complexes of a 1,3-diphenylpropane-1,3-dione and glycine Schiff base: - a ligand offering dual coordination modes (imine + enolate). Filling this gap would deepen the understanding of how metal ligand interactions influence spectroscopic signatures, geometry, thermal stability, and antibacterial efficacy. Considerations of global health further motivate this work: the rise of multidrug-resistant (MDR) bacteria highlights the urgency to identify novel antimicrobial agents with unconventional modes of action. Metal complexes of Schiff base ligands present a promising frontier, potentially disrupting bacterial function via multiple pathways including DNA binding, redox cycling, and membrane penetration [2]. Therefore, the present study aims to synthesize Co(II), Ni(II), Cu(II), and Zn(II) complexes of a 1,3-diphenylpropane-1,3-dione and glycine Schiff base then characterize (via FT-IR, solubility, molar conductance measurements and magnetic susceptibility), and evaluate the antibacterial activity of the synthesized schiff base against representative Gram-positive and Gram-negative bacteria. By systematically comparing these four metal centers with a uniquely dual-donor Schiff base, the research also aims to elucidate structure activity relationships that could guide the design of new coordination-based antibacterial agents.

Materials and Methods

All chemicals (including ethanol, glycine, 1,3-diphenylpropane-1,3-dione distilled water, P_2O_5, $CuCl_2$, $CrCl_2$, $CoCl_2$, $NiCl_2$,Ciproflaxacin, DMSO) and materials (including filter paper, petri dish, thermometer, weighing machine, electric conductivity used were of analytical grade and were used without further purification. All weightings and incubator) were carried out with B154 balance. Melting point and decomposition temperatures were carried out on Stuart SMP 10 melting point apparatus. Infrared Spectral data were recorded using Agilent Technologies FT-IR spectrophotometer carry 630. The electrical conductivity measurements were carried out using conductivity meter model DDS 307, Jenway. The magnetic susceptibility measurement was carried out using magnetic susceptibility balance Sherwood Scientific MKI at room temperature.

Synthesis of Schiff Base

A 25cm^3 solution of (25% distilled water + 75% ethanol) glycine (1.5g, 0.01mol) was mixed under magnetic stirring with 25cm^3 ethanolic solution of 1,3-diphenylpropane-1,3-dione (2.24g, 0.01 mol). The mixture was refluxed at 80 - 85°C with continuous stirring for five (5) hours. The mixture was cooled and the solution was evaporated to about half of its volume and left overnight for completion of precipitation. The precipitate was then collected by filtration, washed with 1:1 ethanol-water mixture and dried over P_2O_5 in a desiccator [3].

Synthesis of Metal complexes

The complexes were prepared by adding 30 cm^3 ethanolic solution of Cr(II), Co(II), Ni(II) and Cu(II) chloride salts (0.01mol) to an ethanolic solution of the Schiff base (0.01mol), heated at 80 - 85°C under reflux for three (3) hours as illustrated in scheme 1. On cooling, the complexes were precipitated. The precipitate was then filtered, washed with aqueous ethanol and dried over P_2O_5 in a dessicator.

Antibacterial screening

The ligand and its metal complexes were screened for antibacterial activity against *Streptoococcus pneumonia* (gram positive) and *Escherichia coli* (gram negative) using agar cup-plate method. The suspension of each microorganism was rubbed onto the surface of solidified Muller Hinton agar (Hi-Media) already poured into Petri dishes. Different concentrations (200 mg/ml, 100 mg/ml and 50 mg/ml) of the ligands and the metal complexes in DMSO were prepared

through serial dilution and placed on the culture media before incubation at 37°C for 24 hours. Ciprofloxacin in DMSO was used as reference standard for positive control whereas distilled water was used as negative control [5,6].

Scheme of the Reaction

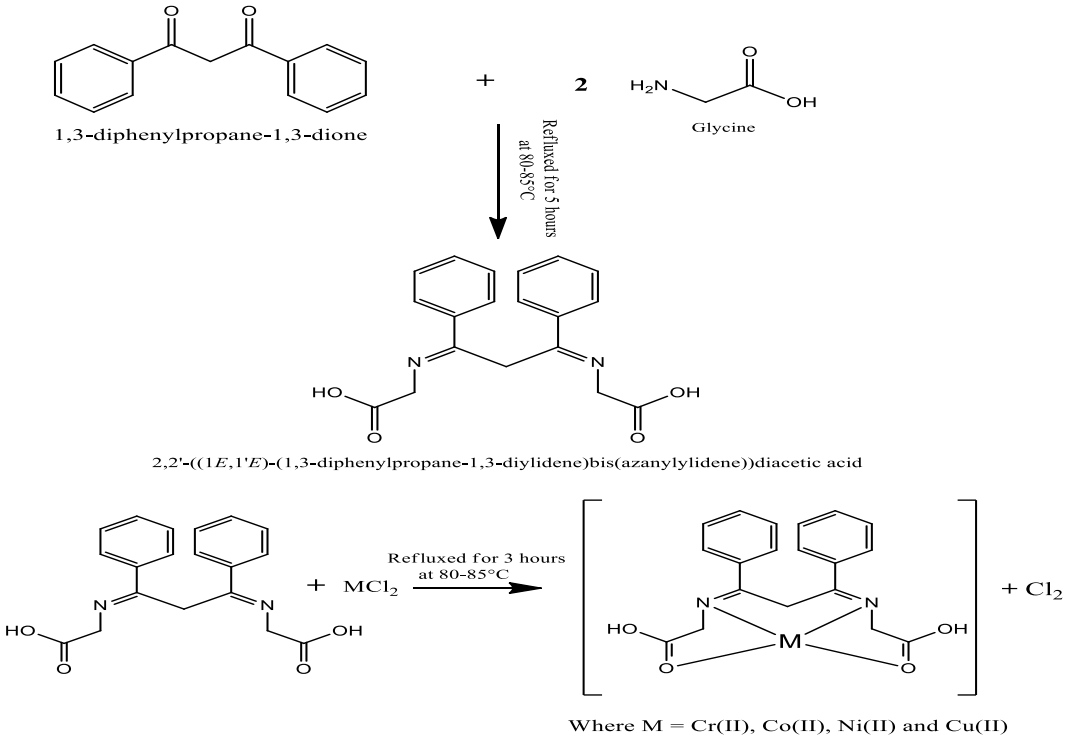

Figure 1: Synthesis of the Schiff base and its metal (II) complexes.

Results and Discussions

The physical characterization of the Schiff base ligand ($C_{19}H_{18}N_2O_4$) and its metal complexes with Cr (II), Co (II), Ni (II) and Cu (II) provides significant insights into their formation, stability, and coordination environment.

Color changes observed upon complexation are a primary indication of metal-ligand interactions. The ligand itself exhibited a milky appearance, while the complexes showed distinct colors: deep green for Cu (II), green for Ni (II), pink for Co (II), and light brown for Cr (II). Such variations are attributed to d–d electronic transitions and ligand-to-metal charge transfer (LMCT) processes that occur upon coordination, consistent with the characteristic crystal field effects of each transition metal ion [7, 8].

Percentage yields ranged between 66.0% and 84.3%, with the ligand exhibiting the highest yield (84.3%) and the Co (II) complex the lowest (66.0%). The differences may be linked to the steric and electronic factors influencing complexation efficiency. Similar observations have been reported in Schiff base transition metal syntheses, where bulky substituents and hydration states of metal salts impact reaction yields [9].

Melting/decomposition temperatures (M.P/D.T.) provide further evidence of coordination. The free Schiff base displayed a melting point of 104.6 °C, significantly lower than those of the complexes, which ranged from 220.3 °C (Ni (II)) to 326.5 °C (Cr (II)). This marked increase in decomposition temperature indicates enhanced thermal stability upon chelation. Such stability arises from the formation of strong coordinate covalent bonds between the azomethine nitrogen/oxygen donor atoms of the ligand and the central metal ions, leading to the creation of rigid chelate rings [10].

The highest decomposition temperature observed for the Cr (II) complex (326.5 °C) suggests that chromium forms the most thermally robust complex, possibly due to its stronger ligand field stabilization energy and higher charge density compared to the other studied metals. On the other hand, the Ni (II) complex displayed the lowest thermal stability among the complexes (220.3 °C), aligning with literature reports that Ni (II) Schiff base complexes often exhibit relatively lower thermal resistance due to their tendency to adopt less stable coordination geometries [11].

Table 1: Physical data of the Schiff base and its Complexes

S/N	Compound	Molecular Formula	Colour	Yield (%)	M.P/D.T (^{O}C)
1	Ligand	$C_{19}H_{18}N_2O_4$	Milky	84.3	104.6
2	$[Cr(L)]Cl_2$	$CrC_{19}H_{18}N_2O_4$	Light brown	80.0	326.5
3	$[Co(L)]Cl_2$	$CoC_{19}H_{18}N_2O_4$	Pink	66.0	236.6
4	$[Ni(L)]Cl_2$	$NiC_{19}H_{18}N_2O_4$	Green	68.5	220.3
5	$[Cu(L)]Cl_2$	$CoC_{19}H_{18}N_2O_4$	Deep green	82.3	282.4

Where L = $C_{19}H_{18}N_2O_4$

The solubility profile of the Schiff base ligand ($C_{19}H_{18}N_2O_4$) and its metal (II) complexes provides valuable information regarding polarity, intermolecular interactions, and potential applications in biological and industrial systems. The ligand displayed insolubility in water but was sparingly soluble in methanol, DMSO, and acetone, and insoluble in benzene. This behavior can be attributed to the presence of moderately polar functional groups (C=N, C=O, and aromatic moieties) that limit its solubility in highly polar (water) and nonpolar (benzene) solvents. Schiff bases are generally known to exhibit poor aqueous solubility due to their aromatic backbone and limited hydrogen-bonding sites [13].

For the metal complexes, distinct variations were observed upon coordination. All complexes showed enhanced solubility in DMSO and benzene, reflecting the increased polarity and π–π interactions introduced by the metal-ligand framework. In particular, Ni(II), Co(II), and Cr (II) complexes were soluble in all organic solvents tested except water (for Cr(II)), suggesting improved charge delocalization and molecular interactions in polar aprotic solvents [14]. This improved solubility pattern in organic solvents is consistent with previous studies, where metal chelation increases dipole moment and enhances ligand–solvent interactions [15].

The Cu(II) complex, while soluble in DMSO and benzene, remained sparingly soluble in water, methanol, and acetone. This may be explained by its stronger lattice enthalpy and reduced interaction with protic solvents compared to Ni(II) and Co(II) analogues. Furthermore, the Cr(II) complex exhibited solubility in methanol, DMSO, and benzene but insolubility in water and acetone, which could be attributed to its relatively higher coordination stability and partial hydrolysis in protic solvents [16].

Overall, the results highlight that metal complexation enhances solubility in organic solvents compared to the free ligand, which is advantageous for biological and catalytic applications, particularly since DMSO is a common medium for in vitro antimicrobial testing [17]. The ligand's poor aqueous solubility may limit its direct pharmacological use, but the complexes' improved solubility profile supports their potential as bioactive agents.

Table 2: Solubility test of the Schiff base its metal (II) complexes in some solvents

Compounds	Water	Methanol	DMSO	Benzene	Acetone
Ligand	IS	SS	SS	IS	SS
$[Cr(L)]Cl_2$	IS	S	S	S	IS
$[Co(L)]Cl_2$	SS	SS	S	S	S
$[Ni(L)]Cl_2$	SS	S	S	S	S
$[Cu(L)]Cl_2$	SS	SS	S	S	SS

KEY: S=Soluble, SS=Sparingly Soluble and IS=Insoluble, where L = $C_{19}H_{18}N_2O_4$.

The molar conductance values of the complexes $[Cu(L)]Cl_2$ (11.73), $[Ni(L)]Cl_2$ (7.44), $[Co(L)]Cl_2$ (5.30), and $[Cr(L)]Cl_2$ (8.02 $\Omega^{-1}\cdot cm^2\cdot mol^{-1}$) fall within the low range typical of non-electrolytic coordination compounds. Such values confirm that the chloride ions are coordinated directly to the metal centers rather than present as dissociated counter-ions [18, 19]. This supports neutral complex formulations of the type $[M(L)]Cl_2$.

The magnetic moment values of the synthesized complexes provide crucial information regarding the oxidation states, spin configurations, and likely geometries of the metal centers.

$[Cr(L)]Cl_2$ (μeff = 4.97 B.M.): This value is close to the spin-only magnetic moment for a d^4 system in high-spin octahedral geometry (~4.9 B.M.). The result confirms the presence of four unpaired electrons, indicating that chromium remains in the +2 oxidation state and that the ligand field provided by the Schiff base and chloride is not strong enough to induce a low-spin configuration [18]. $[Co(L)]Cl_2$ (μeff = 3.55 B.M.): High-spin Co(II) complexes typically exhibit μeff values in the range of 4.3–5.2 B.M. due to orbital contributions. The lower value here (3.55 B.M.) suggests a partial quenching of orbital contributions, possibly arising from distorted geometry, strong covalency, or antiferromagnetic interactions between paramagnetic centers [11]. This may indicate a deviation from a purely octahedral environment, perhaps toward a distorted tetrahedral or square-planar arrangement. $[Ni(L)]Cl_2$ (μeff = 2.71 B.M.): This value falls within the expected range for high-spin octahedral Ni(II) complexes (2.8–3.5 B.M.) but is slightly lower than the ideal spin-only value for two unpaired electrons (2.83 B.M.).

The small decrease may reflect ligand field effects or slight orbital overlap leading to reduced magnetic moment. This is consistent with octahedral or slightly distorted octahedral geometry [19]. $[Cu(L)]Cl_2$ (μeff = 1.83 B.M.): The observed value matches well with the typical magnetic moment of mononuclear Cu(II) complexes (~1.7–2.2 B.M.), corresponding to one unpaired electron in a d^9 configuration. This strongly supports a distorted octahedral or square-planar geometry around Cu(II), common in Schiff base complexes due to the Jahn–Teller effect [5].

Overall trend

The μeff values follow the order: Cr(II) (4.97 B.M.) > Co(II) (3.55 B.M.) > Ni(II) (2.71 B.M.) > Cu(II) (1.83 B.M.), consistent with the expected number of unpaired electrons across d^4, d^7, d^8, and d^9 systems. The data strongly support that all complexes are paramagnetic and adopt predominantly octahedral or distorted octahedral geometries, with subtle deviations caused by covalency, orbital contributions, or geometry distortions.

Table 3: Molar conductance and magnetic moment of the synthesized complexes.

S/N	COMPOUND	MOLAR CONDUCTANCE ($\Omega^{-1}cm^2mole^{-1}$)	Magnetic moment, μ_{eff} (BM)
1	$[Cr(L)]Cl_2$	8.02	4.97
2	$[Co(L)]Cl_2$	5.30	3.55
3	$[Ni(L)]Cl_2$	7.44	2.71
4	$[Cu(L)]Cl_2$	11.73	1.83

Where L = $C_{19}H_{18}N_2O_4$

Antibacterial Activity

The Schiff base ligand and its complexes were tested against *Streptococcus pneumoniae* and *Escherichia coli* using the agar cup-plate method at concentrations of 200, 100, and 50 mg/mL. The ligand exhibited modest activity, with inhibition zones ranging from 8-12 mm. Upon complexation, antibacterial activity significantly improved, especially for Ni(II) and Co(II) complexes, which showed the

largest inhibition zones: $[Ni(L)]Cl_2$ recorded 20 mm against E. coli and $[Co(L)]Cl_2$ 19 mm against E. coli, both outperforming the free ligand.

This enhancement can be explained by chelation theory, whereby coordination reduces the polarity of the metal ion through partial sharing of positive charge with donor groups (azomethine N and carbonyl/enolate O). This increases the lipophilicity of the complexes, improving their ability to penetrate microbial cell membranes [5, 6]. Furthermore, metal complexation may generate reactive oxygen species or disrupt enzyme-metal interactions essential for microbial survival, contributing to the enhanced activity [7].

The trend of activity across the series was Co(II) ≈ Ni(II) > Cr(II) > Cu(II) > ligand. The relatively lower activity of Cu(II) compared to Ni(II) and Co(II) may reflect differences in ligand field stabilization energy and geometry, influencing how efficiently the complexes interact with bacterial biomolecules. The observation that complexes are more active against Gram-negative *E. coli* than Gram-positive *S. pneumoniae* is consistent with literature reports, table 4, highlighting the greater permeability of Gram-negative bacterial membranes to lipophilic compounds [5].

Table 4: Antibacterial data of the Schiff base and its Metal Complexes

Compound	**200 Mg/ml**		**100 Mg/ml**		**50 Mg/ml**	
	S.pneumoniae	***E.coli***	***S.pneumoniae***	***E.coli***	***S.pneumoniae***	***E.coli***
Ligand	10	12	08	08	11	08
$[Cr(L)]Cl_2$	16	14	13	12	11	09
$[Co(L)]Cl_2$	18	19	16	17	12	11
$[Ni(L)]Cl_2$	16	20	15	17	11	14
$[Cu(L)]Cl_2$	14	15	12	10	10	09

Where L = $C_{19}H_{18}N_2O_4$

Integrated structural inference

Combining the low molar conductance (non-electrolytic) with typical magnetic expectations suggests the most parsimonious structural model is neutral mononuclear complexes $[M(L)]Cl_2$, with coordinated chlorides occupying sites in an approximately octahedral (or tetragonally distorted octahedral for Cu) coordination sphere. Confirmation requires the missing magnetic numerical data ($\chi g \rightarrow \chi m \rightarrow \mu\_eff$ after diamagnetic correction), plus corroborating spectroscopic (FT-IR shifts in ν(C=N) and M–Cl signatures, UV-Vis d–d bands) and elemental/crystallographic evidence (elemental analysis, single-crystal XRD or PXRD) [18, 19].

FT-IR results (schiff base L = $C_{19}H_{18}N_2O_4$ and $[M(L)Cl_2]$ complexes)

The FT-IR data show clear spectral changes on complexation that illuminate the ligand binding mode. The free Schiff base displays ν(O–H) at 3551 cm^{-1}, ν(C=O) at 1704 cm^{-1}, and ν(C=N) at 1689 cm^{-1}. All metal (II) complexes exhibit downshifts of the azomethine ν(C=N) (to 1659–1603 cm^{-1}), which is strong, classical evidence that the azomethine nitrogen coordinates to the metal centers; the magnitude of the downshift is greatest for Co and Cr, indicating stronger N→M donation or greater delocalization in those complexes [7].

The carbonyl region shows metal-dependent behavior: Ni (1707 cm^{-1}) is nearly unchanged relative to the ligand, Cu shows a slight upshift (1722 cm^{-1}), Co displays a pronounced downshift to 1666 cm^{-1}, and Cr exhibits an anomalous band at 1797 cm^{-1}. These variations suggest different tautomeric/coordination outcomes across the series. In particular, the downshift for Co (and the pronounced perturbation for Cr) is consistent with partial or full involvement of the carbonyl/enolate oxygen in coordination (N,O-bidentate binding) for these metals, whereas the small upshift in Cu and minimal change in Ni imply that the carbonyl remains largely non-coordinated or is influenced by changes in conjugation or hydrogen bonding rather than direct M–O bond formation [18, 19]. Broadening and low-frequency shifts of the O–H region on complexation (e.g., Cu at 3083 cm^{-1}, Ni/Co ~3330–3342 cm^{-1}) point to coordinated and/or lattice water and stronger hydrogen-bonding environments in the complexes, in agreement with the hydrated formulas reported for several complexes. Identification of H–O–H bending modes in the 1600–1650 cm^{-1} region (which may

overlap with other bands) would further corroborate water incorporation [7]. Finally, the features listed under "M–N" (1224–1391 cm^{-1}) and "M–O" (1004–1071 cm^{-1}) in your table fall in regions more typically assigned to organic C–N and C–O vibrations rather than true metal–ligand stretches. Authentic ν(M–N) and ν(M–O) stretches normally appear at much lower frequencies (≈ 600–300 cm^{-1}); therefore, definitive assignment of M–N and M–O bands requires inspection of the far-IR/low-frequency region. When those low-frequency bands are present and new upon complexation, they will directly confirm metal–nitrogen and metal–oxygen bonding [18, 19]. The IR spectra support azomethine nitrogen coordination for all complexes, with metal-dependent involvement of the carbonyl/enolate oxygen (likely N,O bidentate coordination for Co and Cr; predominantly N-coordination for Cu and Ni). The presence of coordinated/lattice water is indicated in several spectra. For unambiguous assignment of M–N and M–O stretches, acquisition/analysis of the low-frequency (<600 cm^{-1}) region is recommended [7, 18].

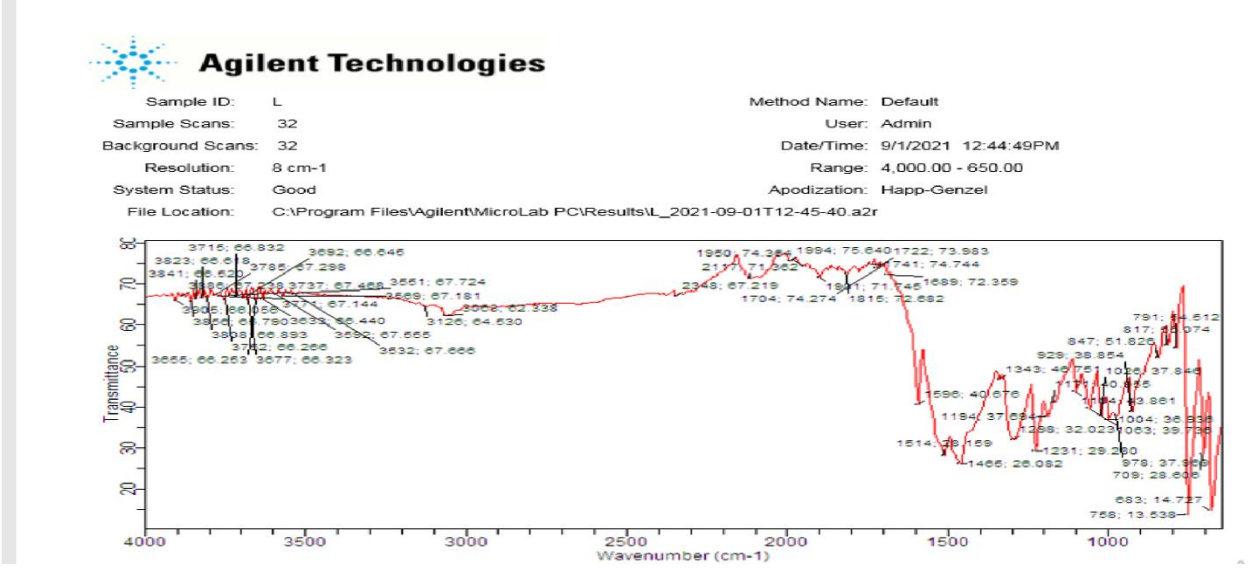

Figure 2. FT-IR spectra of Ligand

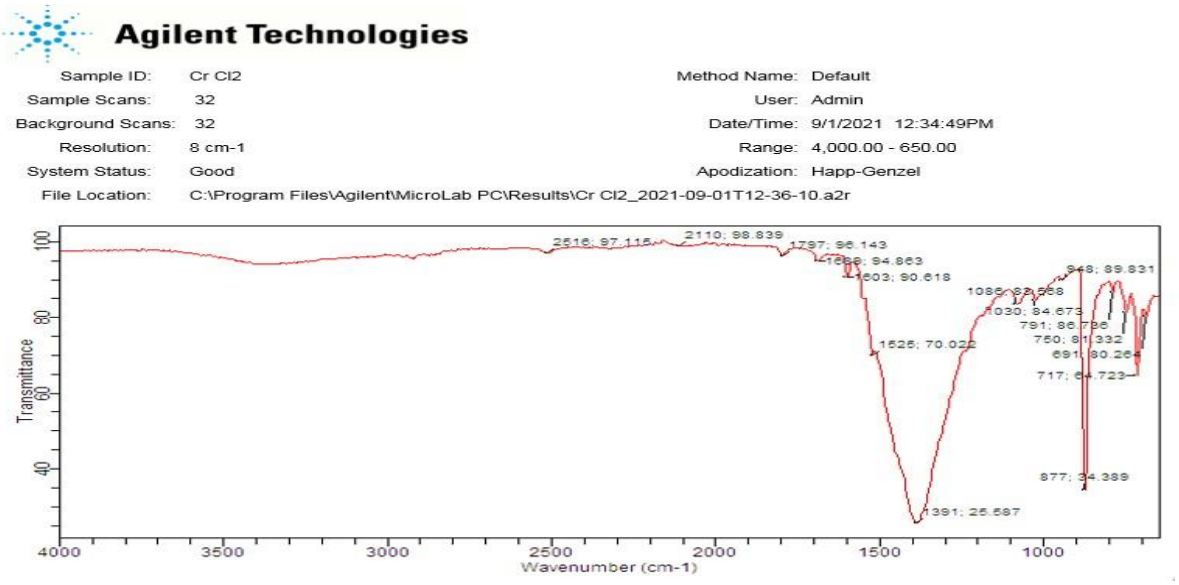

Figure 3. *FT-IR spectra of $CrCl_2$*

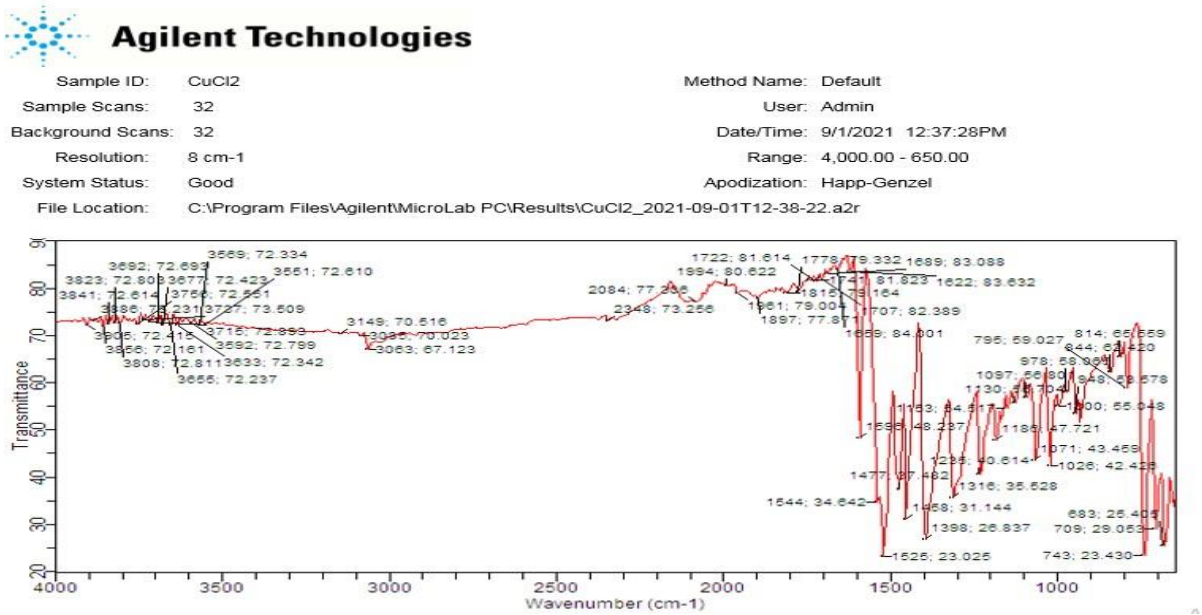

Figure 4. *FT-IR spectra of $CuCl_2$*

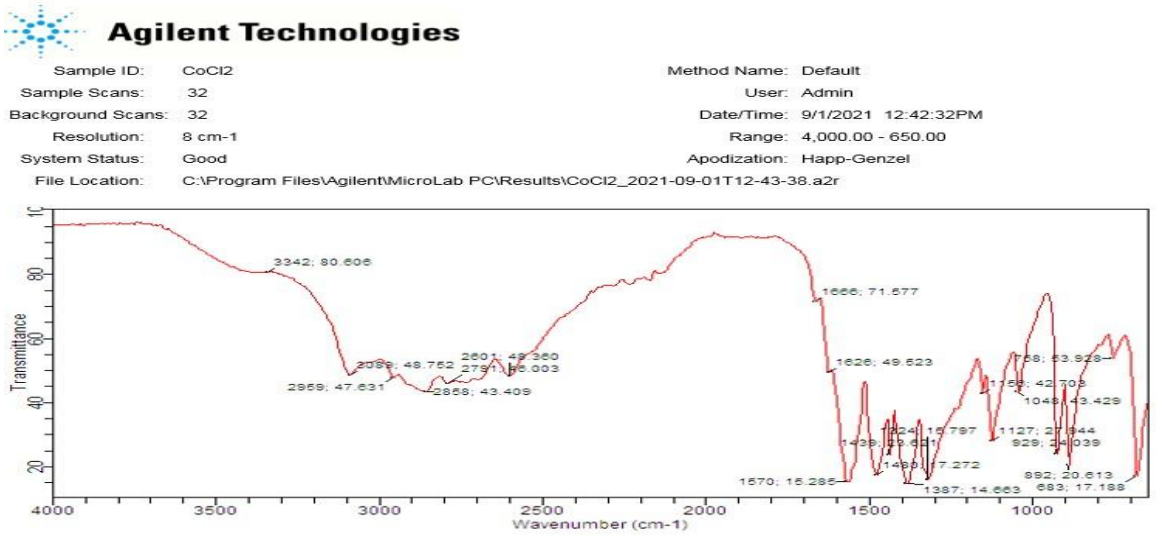

Figure 5. *FT-IR spectra of $CoCl_2$*

Table 5: *Infrared Absorption Frequencies (cm^{-1}) of the Schiff base and its Metal (II) Complexes*

S/N	COMPOUND	υ (O-H)	υ (C=O)	υ (C=N)	υ (M-N)	υ (M-O)
1	Ligand	3551	1704	1689	1343	1004
2	$[Cr(L)]Cl_2$	3349	1797	1603	1391	1030
3	$[Co(L)]Cl_2$	3342	1666	1626	1224	1048
4	$[Ni(L)]Cl_2$	3331	1707	1679	1298	1026
5	$[Cu(L)]Cl_2$	3083	1722	1659	1316	1071

Where L = $C_{19}H_{18}N_2O_4$

Conclusion

This study successfully synthesized and characterized a Schiff base ligand derived from 1,3-diphenylpropane-1,3-dione and glycine, along with its Co(II), Ni(II), Cu(II), and Cr(II) complexes. The results confirm the coordination of the ligand through azomethine nitrogen and, in some cases, carbonyl/enolate oxygen, forming thermally stable, non-electrolytic mononuclear complexes. Characterization data revealed distinct color changes, higher decomposition temperatures, and improved solubility of the

complexes compared to the free ligand. Antibacterial evaluation demonstrated that complexation significantly enhanced activity against both Gram-positive and Gram-negative bacteria, with Ni(II) and Co(II) complexes showing the highest inhibition zones. The improved biological activity of the complexes can be rationalized by chelation theory, which enhances lipophilicity, facilitates cell membrane penetration, and potentially disrupts microbial metabolic processes. Overall, this research underscores the potential of Schiff base transition metal complexes as lead structures for the development of new antibacterial agents, particularly in addressing the growing challenge of antimicrobial resistance. Further studies involving minimum inhibitory concentration (MIC), DNA binding, and molecular docking are recommended to better understand their mechanisms of action.

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