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Synthesis and Characterization of Some 3D Metals Complexes Derived from Schiff Base of Benzoin

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ABSTRACT

We made a new Schiff base ligand by reacting benzoin with 4-aminophenol, then combined it with Ni(II), Cu(II), Zn(II), Mn(II), Co(II), and Mg(II) to get six different metal complexes. To check what we'd made, we looked at their melting points, measured conductivity, and ran FT-IR spectra.

The ligand itself is a brown, crystalline solid. It doesn't dissolve in water but goes into ethyl acetate and hexane just fine. It shows almost no electrical conductivity (around 0.13 $\mu S \cdot cm^{-1}$ in 1 mM MeOH), so it's definitely non-electrolytic. In the FT-IR spectrum, the carbonyl band from benzoin (about 1696 cm^{-1}) disappears and a new band pops up at 1568 cm^{-1}, which tells us the Schiff base formed like we planned.

When we added the metals, each complex came out as a colored solid—green with Ni and Cu, off-white with Zn, and a clay color for Mn, Co, and Mg. Their conductivities were all super low too (under 0.20 $\mu S \cdot cm^{-1}$), so none of them act like electrolytes.

The FT-IR spectra gave us more to go on. The imine C=N band in the ligand, which shows up near 1570 cm^{-1}, shifts down to about 1420–1430 cm^{-1} in the complexes, and new bands for metal–nitrogen bonds appear (Ni at 530 cm^{-1}, Cu at 433 cm^{-1}, Zn at 447 cm^{-1}, Mn at 486 cm^{-1}, Co and Mg both at 447 cm^{-1}). This shift matches what you'd expect if the metal binds through the azomethine nitrogen. A bunch of the complexes also show broad FT-IR bands between 2550 and 3615 cm^{-1}, which points to coordinated water.

All in all, the data confirm we made the Schiff base ligand and its complexes with these 3d metals.

KEYWORDS: Schiff Base; Benzoin; Transition Metal Complexes; FT-IR Spectroscopy; Coordination Chemistry; Metal Complexes

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INTRODUCTION

Metal bearing organic moieties, metal complexes and organometallics play very crucial roles in medicines, agriculture and industrial arenas. A metal complex consists of metal atoms or ions present in center surrounded by ligands. These ligands can be neutral or negative. Metal complexes are also known as coordination complexes [1]. Transition metal complex can be anionic cationic or neutral species [2]. Transition metals interact with negatively charged ions or neutral molecules in variety of modes because metal exhibit different oxidation sates. This property leads to the development of metal based drugs that have number of pharmacological applications [1]. Because of the diversity in their optical, chemical and magnetic properties, transition metal complexes are used as therapeutic agents and these also play important role in catalysis, biological systems and in photo-chemistry [3]. Ligands have vital role in determining the properties of the complexes. Schiff bases are important ligands which have been employed to synthesize different complexes which exhibit range of biological activities such as antifungal, antibacterial, antimalarial, antiproliferative and antiviral properties [4].

Metals have a unique place in chemistry of medicines. Research has revealed significance development in the employment of transition metals as drugs. Most important use of the transition metals is as anticancer drugs [5]. Recently a lot of work has been done for the development of therapeutic metal complexes. Although metal complexes have played a significant role for modern chemotherapy and pharmacy but there is minor use of these complexes in today market yet [1]. Recent works on the discovery of chemotherapeutic base and their metal complexes are now attracting the attention of medicinal chemist. New developments in the field of metallodrugs and novel methods are being employed to improve delivery of the drugs [6].

When a group of cells show abnormal growth and invasion this abnormal growth leads to cancer. This is a serious problem of the world and second leading cause of death throughout the world. Treatments of cancer include surgery and chemotherapy but the drugs are not effective enough. These drugs have various side effects. Various metal complexes show anti-cancer activity. Many Schiff bases proved to be effective in cancer treatment [7]. Au et al., 2008 recognized that transition metal complexes with gold showed more anti-cancer activity against breast cancer by different mode of action as compared to platinum complexes [8]. Among Pt complexes cisplatin is most active anti-cancer drug [9].

Besides these complexes nanoparticles of gold makes more DNA damage and make the treatment of target cells more specific. Their activity found to be enhanced when they are used with radio and chemotherapy [10]. Some complexes of Mn(III) have also been found to be active in causing selective apoptosis of tumor cells in human body [11]. Recent study revealed that ruthenium (II) complexes also have anticancer activity because they have low toxicity and stronger affinity for cancer cells. Half sandwiched complexes of ruthenium (II) with arene came out as candidate for anticancer activity because of the availability of three coordination positions that give stability to metal center. These complexes have excellent anticancer activity but much lower than cisplatin and destruction of DNA is one of the targets in their mode of action [12].

From 2-acetylpyridine and L-tryptophan ($C_{18}H_{16}N_3O_2$) three metal complexes have

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been prepared $Cu(C_{18}H_{16}N_3O_2)_2CH_3$ OH , $Zn(C_{18}H_{16}N_3O_2)_2$.$2CH_3$ CH_2 OH and $Cd(C_{18}H_{16}N_3O_2)_2$.$2CH_3OH$ and their activities has been investigated on breast cancer cells. The results show that complex Cd $(C_{18}H_{16}N_3O_2)_2$.$2CH_3$ OH has more anticancer activity than others [10].

Many metal complexes found to be very active in antibacterial activity. Pyrimethamine complexes of Ag (I), Co (II), Cu (II) was prepared by Idemudia and Ajibade. These complexes were found to be air stabled. By antimicrobial screening they found that Ag (I) complexes have much more antimicrobial potential at an MIC value of 0.03125mg/ml [13].

Jayaseelan, et al, (2010) tested the antimicrobial activity of Cu (II), Ni (II), Co (II) and Mn (II) cyt-a/a3 complexes against gram positive and gram-negative bacteria. In case of gram-positive bacteria, the rate of activity of these complexes was Co > Ni >Cu > Mn, and in case of gram-negative bacteria the order was Cu > Co > Ni >Mn. They showed that the chelation of these complexes made the complexes more powerful antibacterial agents [14]. Another Schiff base 2-Aminomethylthiophenyl-4-bromosalicylaldehyde has been tested against bacteria, the results showed this Schiff base has more inhibitory activity than the parent compound. It was suggested that their antibacterial activity involves the inhibition of a step involved in the synthesis of peptidoglycan, so that the Schiff base become able to kill the bacteria [15].

Malaria is one of the parasitic diseases in the world. A female Anopheles mosquito is responsible for the spread of this disease. Metal complexes have also been proved as effective anti-malarial agents. Numbers of potential antimalarial drugs are available. Numbers of metal complexes such as gold, iron, cobalt, ruthenium, iridium, zinc and palladium have antimalarial activity, there activity involves metal complexation by heme inhibition. There enhanced activity is due to the structural modifications in metal complexes. For the treatment of malarial infections metal chelating agents are very active [16]. Because of the unique properties of ruthenium, ruthenium centered antimalarial drugs have been prepared. These found to be active for antimalarial activity [17]. Parasite need iron for the reproduction, if anything inhibit this iron the reproduction can be stopped. Iron chelating agents have ability to capture iron available for reproduction, that's why iron chelating agents can also be used as anti-malarial drugs [18].

Many metal complexes have been prepared and characterized for the treatment of inflammatory disorders. Gold and silver complexes have been used clinically for the treatment of rheumatoid arthritis [19]. Iron, copper and Mn complexes can also be used anti-inflammatory drugs because these can cause dismutation of superoxide anion. For the treatment of heart diseases sodium nitropruside is applied because it gives NO but CN toxicity its activity. However, ruthenium nitrosyl complexes have proven to be more active than sodium nitropruside [20]. Metal complexes of tolfanamic acids (Figure1) have also been investigated as anti-inflammatory agents [21]. Vanadium (II) complexes have been recognized for showing sulphonomide group at 1 position in the benzene ring that increases its anti-inflammatory activity [22]. Of 4-aminoantipyrine (4-amino- 1, 5-dimethyl-2-phenylpyrazole-3-one) and benzaldehyde derivative also show anti-inflammatory activity. This promising anti-inflammatory activity is beneficial for use in the treatment of inflammatory diseases. The results of analysis of these ligands can lead to the development of a new

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therapeutic agent that are useful against diseases caused by oxidative stress and inflammation [23].

About 2-8 % of the people of world are in state of undergoing pain due diabetes [24]. In diabetes body does not produce insulin for the absorption of glucose in cells [25]. For the treatment of diabetes number of vanadium complexes have prepared that are less toxic and have more insulin-mimetic ability. Vanadium complexes cause the activation of components in insulin-signaling pathway and increase tyrosine phosphorylation of insulin receptor substrate [26].

Metal complexes can act as catalyst in many reactions like in biological systems, dyes and for polymerization. Many aromatic bases and their complexes can be used as effective catalyst [27]. Ruthenium complexes have versatile applications in organic transformation. But Ruthenium Pincer Complexes have proven to be more active in catalysis s compares to traditional ruthenium complexes. Studies revealed that Ruthenium Pincer Complexes are more efficient and selective. Many novel and important reactions have been done successfully by using Ruthenium Pincer Complexes [28]. Many Schiff bases that obtained from metal complexes can be applied in many biological, polymer and dye industry [04]. These Schiff bases are highly selective, sensitive and stable, therefore are used as carrier of cations in many reactions [29].

A series of Schiff base complexes prepared by the condensation of 1,2-diaminocyclohexane with salicylaldehyde, 2-pyridinecarboxaldehyde, and 2-hydroxy-1- naphthaldehyde, and their metallation with manganese cobalt, copper and iron salts yield complexes that are catalytically active. Under moderate circumstances, styrene is catalytically epoxidized using the Mn complex that results from the N,N0-bis (2-hydroxy-1-naphthalidene) cyclohexanediamine ligand. The greatest styrene oxide production, 91.2 mol%, was much greater than that of the straightforward salt catalysts Mn (Ac) 2.4H2O and MnSO4.water. Nevertheless, under the same experimental conditions, two other salene Mn complexes, 1 and 2, comprising N,N0-bis (salicylidene)cyclohexanediamine and N,N0-bis (2-pyridinecarboxalidene) cyclohexanediamine ligands exhibit comparatively low activity.
[30].

Schiff bases possess photo and thermo chromic properties that make Schiff bases a valuable material for use in modern technology. The photochromic properties make Schiff bases to be used as photostablizer and dye for solar collector. These are also used in optical sound recording technology [31].

Schiff bases are used to prevent corrosion in steel, copper and zinc in acidic medium. This property is used to the presence of imine group nitrogen, oxygen and sulfur atoms [32]. In cosmetics hair fiber are used. These fibers contain 85% keratin and 7% water along these some metals such as aluminum (Al), calcium (Ca), and many transition metals such as iron (Fe), manganese are in high concentrations [33]. Another transition metal Ni is used as skin sensitizer that penetrates into stratum corneum and with endogenous proteins makes hapten–carrier conjugate [03]. Another application of transition metal complexes is the doping into halide crystals. For the synthesis of salt, the saline water hexacyanoferrate (II) is used to prevent the cake formation. In photographic industry for the development of latent image transition metal complexes are incorporated into the silver halide microcrystals [34]. For the

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first reaction at 358 K, they provide a high yield (90–100%) of epoxidation; for the epoxidation of 1-octene, the yield was between 15 and 74%. More effective and selective catalysts are [Trans-N, N0-bis-(4, 6-dimethoxysalicylidene)-1, 2-cyclohexanediaminato]-dioxomolybdenum (VI) and [Trans-N, N0-bis-(2-hydroxynaphth-ylidene)-1, 2-cyclohexanediaminato]-dioxomolybdenum[35].

The metal ions, especially of transition metals, play essential roles in biological systems. In many cellular and subcellular functions metals play crucial role. For the regulation of cell activities Mg^{+2} and Ca^{+2} are involved. Metallothionin is a protein that is also rich in metals. Similarly, copper is involved in in the functioning of Cu dependent cellular energy. Zn^{+2}, Ca^{+2} and Mg^{+2} inhibit cytotoxicity and antagonize the effect of Cd induced carcinogenesis. In body protein complex are responsible for the delivery of iron in body [36].

One of the a-Hydroxy ketones that is useful in the creation of Schiff base ligands with proven industrial, antimicrobial, and medicinal applications is benzoin. Schiff bases metal-based compounds with a benzoin moiety are still accounted for in terms of their spectroscopic and electrochemical activities. This research has focused on the spectroscopic and chemical characteristics of the novel Schiff base ligand made from benzoin and its complexes with three-dimensional metals [37].

Benzoin and o-amino benzoic acid have been combined to create a complex Schiff base compound with nickel (II) salt; however, this molecule has not been well described, and its antibacterial qualities have not been documented. The production, complete characterisation, and antibacterial characteristics of the nickel (II) Schiff base complex are reported in this work [38].

Physical and chemical approaches have been used to synthesize and analyze a series of metal complexes with three novel tetradentate Schiff bases generated from benzoin and benzil with o-toluidine and benzil with diaminoethane. While the Hg n and Cd n complexes have tetrahedral geometry, the Co n and Ni II complexes seem to have octahedral geometry based on electronic spectra and room temperature magnetic moment values. The complexes of Cu and N are square planar. All of the other complexes, with the exception of the Schiff base complexes made from benzoin, have high molar conductance values, indicating that they are electrolytes. A few fungal infections have been filtered out of the complexes [39]. Thus, once the first reaction has occurred, the intermediate complex is properly oriented for intramolecular reaction [40].

Literature review

Synthesis of tridentate ligand:

In 2017, Gayathri N. et al. synthesized the tridentate ligand.Benzoin and anthranilic acid are used to make the Schiff bases.The disclosed process was used to manufacture the ligand.After adding an ethanolic solution of 2-aminobenzoic acid and anhydrous sodium acetate to the ethanolic solution of benzoin, the combination was refluxed for an hour. After pouring the heated solution into ice-cold water, the Schiff base's grayish white precipitate separated, was suction-filtered, rinsed with water, recrystallized from rectified spirit, and vacuum-dried [41].

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Synthesis of schiff base (HDEA) using (HDEAP) ligand:
Ibtihal Khadim et al. successfully synthesized the ligand (HDEA) in 2019.There are two phases involved in ligand (HDEAP) preparation: Condensation of 4-aminoantipyrine and benzoin produced the Schiff base (HDEA), which is the first step. The second stage included creating the novel Schiff base ligand by concentrating the first step's result (HDEA) with 3-aminoacetophenone in 100% ethanol using an acid catalyst [42].

Synthesis of Schiff base by using 2, 6-diaminopyridine:
Qiuqun Liang et al. synthesized schiff bases in 2019 using 2,6-diaminopyridine. To get the schiff bases ready, they used the following technique. 2,6-diaminopyridine was utilized as the starting material. It reacted with o-, m-, and p-carboxy benzaldehyde to form 2,6-diaminopyridine-o-carboxyl and 2,6-diaminopyridine-p-carboxyl benzaldehyde bis-schiff bases, which are denoted as L1, L2, and L3, respectively [43].

Synthesis of schiff bases from benzoin and o-phenylenediamine (bop)
Using a homogenized ethanolic solution of benzoin (4.42g, 0.02mol in 20ml ethanol) and an ethanolic solution of o-phenylene diamine (1.08g, 0.01mol in 20ml ethanol), Gayathri and M.S. Suresh created the schiff bases. They then added sodium acetate (4g) and refluxed the mixture for an hour. When the heated solution was added to ice-cold water, the Schiff base's yellow precipitate separated. After filtering, water washing, drying, and re-crystallization from ethanol, [44].

Schiff Bases synthesized From Benzoin and Ethylendiamine (BED)
The schiff bases were created by Gayathri and M.S. Suresh using the same technique for the ethylendiamine complex, which substituted 4.48g,0.01mol of ethylendiamine diluted in 20ml of alcohol for o-phenylenediamine [45].

Synthesis of 4-(4-Substituted phenyl)-N-(4-substituted benzylidene)-1,3-thiazole-2-amines
First, using the traditional Hantzsch and Weber technique, the intermediate 4-Substituted phenyl-1, 3-thiazole-2-amines were produced (Hantzsch and Weber, 1887). Thus, using piperidine as a catalyst, the corresponding 4-Substituted phenyl-1, 3-thiazole-2-amine reacted with various aldehydes at an equimolar ratio to yield the Schiff bases (Geronikaki et al., 2004). The resulting products were purified using liquid chromatography with silica gel (hexane: ethyl acetate, 7:3) [46].

Synthesis of Schiff bases (9a-l) of 3-amino-6, 8-dibromo-2-phenyl-quinazolin-4(3H)-ones9
Using 3-amino-6, 8-dibromo-2-phenyl-quinazolin-4(3H)-ones with various substituted aldehydes, Paneersalvam et al. (2010) produced the schiff base by refluxing their equimolar dry ethanol solution for six hours. The Schiff bases (9a–l) were filtered, dried, and recrystallized using absolute ethanol. Comprehensive spectroscopy (IR, 1H NMR, 13C NMR, Mass) and elemental analysis were used to analyze each of the chemicals (9a–l). [47].

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Synthesis of the (Z)-4-(4-amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-ylideneamino) benzoic acid

The (Z)-4-(4-amino-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-ylideneamino) benzoic acid (HL1) was synthesized by Al-Shimaray et al. in 2016.After adding three to four drops of glacial acetic acid, a 50 mL ethanolic solution containing 4-aminoantipyrine (0.01 mol, 2.03 g) and 4-aminobenzoic acid (0.01 mol, 1.37 g) was refluxed for 36 hours. After filtering the combination, a water bath was used to concentrate the resultant solution to 20 milliliters.The reaction mixture cooled, and a bright yellow crystal product separated out (yield 85%, m.p. 240 0C). This product was recovered by filtering, completely cleaned with ethanol, and vacuum-dried.The TLC technique was used to verify the product's purity, and elemental and spectral studies were used to describe it [48].

Synthesis of Schiff base using methoxybenzaldehyde:

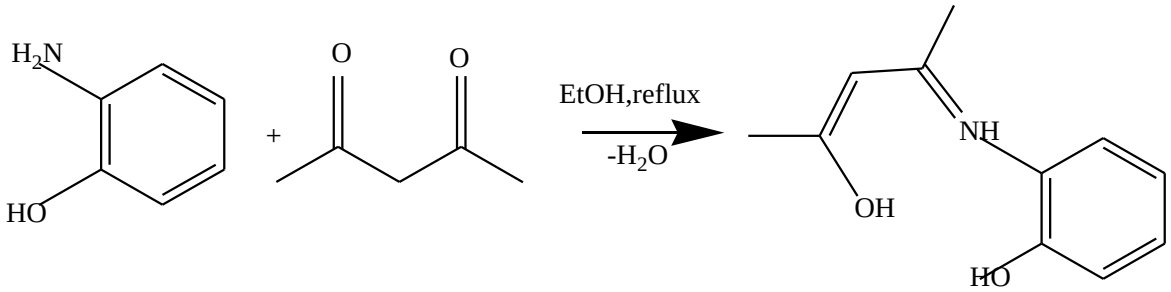
Senarli et al. created the schiff base in 2020 by combining a suitable methoxybenzaldehyde (such as 4-hydroxy-3-methoxy-5-nitrobenzaldehyde for 6, 985 mg; 5 mmol) with an ethanolic solution (10 mL) of 4-chloro-2-aminophenol (720 mg; 5 mmol) in an equivalent volume of ethanol. After three hours of refluxing with stirring, the liquid was left to crystallize the Schiff bases at room temperature [49]

Synthesis of schiff base using aminophenol and trimethinium salt

Samani et al in 2021 Synthesize the Schiff bases 3 via the reaction between trimethinium salts 1 and diverse aminophenols 2 in the presence of Et3N in EtOH at reflux. [50]

Synthesis of Schiff base by using ortho-aminophenol:

Me2SnCl2 (Fluka), Ph2SnO (Aldrich), ortho-aminophenol (Lobachemie), acetyl acetone (Sisco), and 20 -hydroxyacetophenone (Aldrich) were utilized by Basu et al. (2010) without any further purification. 2-hydroxy-3-methylacetophenone was a gift sample. AR-grade solvents were used in the reactions, and they were dried according to established protocols. The literature approach was used to synthesis Me2SnO. [51]



Chemicals and Instruments

The chemicals used in this work were of analytical grades and purchased from Sigma-Aldrich Pakistan. These were used without further purification except the solvents which were purified according to the standard procedures.

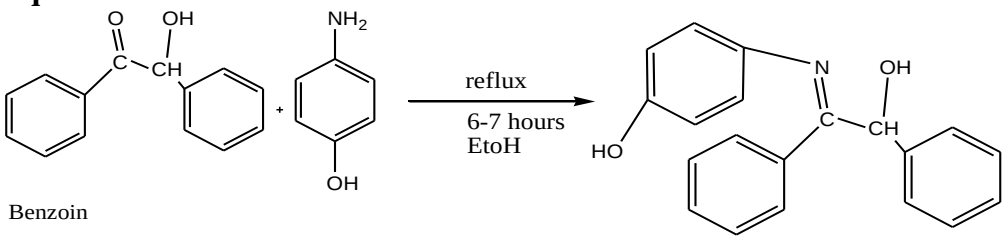
Gallenkamp digital melting apparatus was used for melting point determination and FTIR spectra were recorded prestige-21(200) FT-IR spectrometer. All the prepared

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products were present on solid, the IR Spectra were measured by using KBr discs.
Reaction progress was checked by thin layer chromatography (TLC) by using aluminium backed plates coated with thin layer silica gel (0.25nm) which shows fluorescent indication (GF $_{254}$) active at wavelength 254nm.
The mobile phase employed usually was made up of n-hexane, chloroform, ethyl acetate, methanol etc.

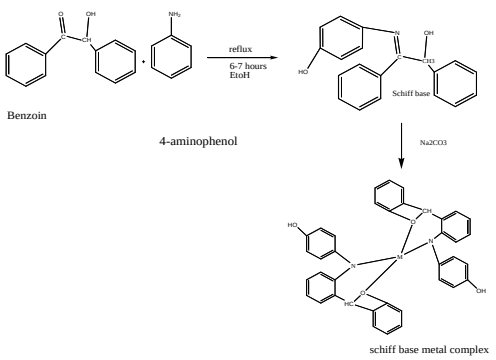
Scheme of work
Sythesis of schiff base ligand:
Equation:



Benzoin 4-aminophenol **Schiff base**

Method:
In a 1:1 molar ratio, ethanol was used to dissolve the benzoin and 4-amino phenol.The mixture of the ethanolic solution.Reflux on an oil bath for three hours was the remedy. Filtration was used to separate the crystalline complex after cooling, and the crystals were then cleaned with ethanol, hydrous Et2O, and dried CaCl2.

Synthesis of Metal Complexes from schiff base
Schiff bases can coordinate with transition metals because of the presence of lone pair in donor group. Schiff bases are good chelating agents and can form compounds containing six or five membered ring. These complexes can be synthesized by the reaction of bases with metal nitrate, acetates, chloride or sulphates. Na_2CO_3
Equation:

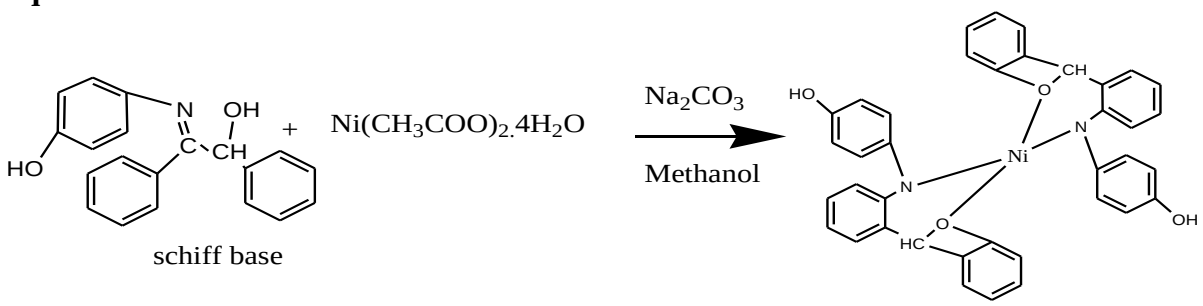


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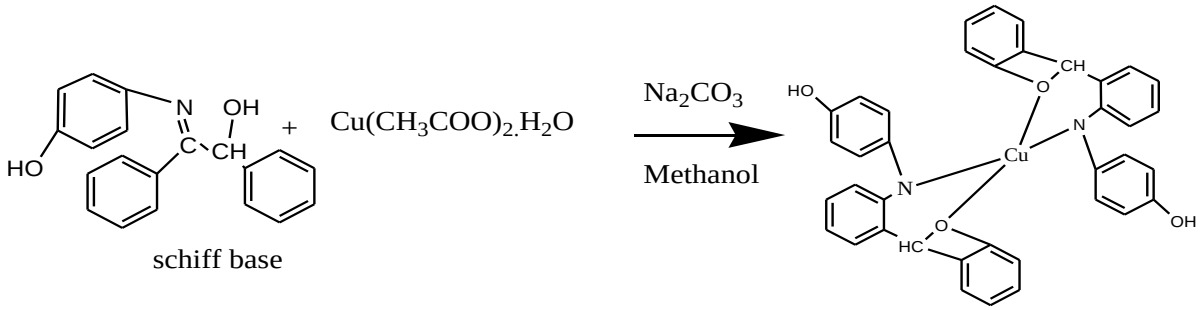
Synthesis of Nickel (II) complex with ligand
Equation:



schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction mixture with continuous stirring.The reaction mixture cooled down at room temperature.Nickel acetate(0.164g,0.659mmoles) dissolved in methanol was added dropwise in the reaction mixture under continuous stirring.

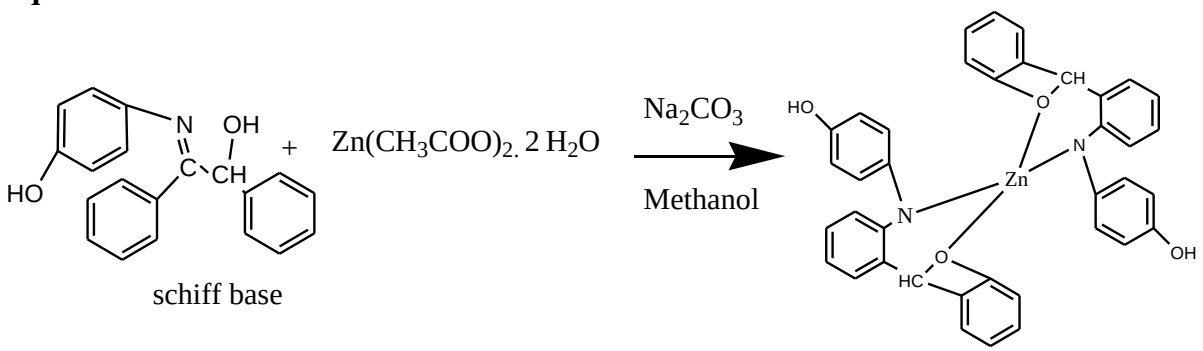
Synthesis of Copper (II) complex with ligand
Equation:



schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction mixture with continuous stirring. The reaction mixture cooled down at room temperature. Copper acetate (0.164g,0.82mmoles) dissolved in methanol was added drop wise in the reaction mixture under continuous stirring.

Synthesis of Zinc (II) complex with ligand
Equation:



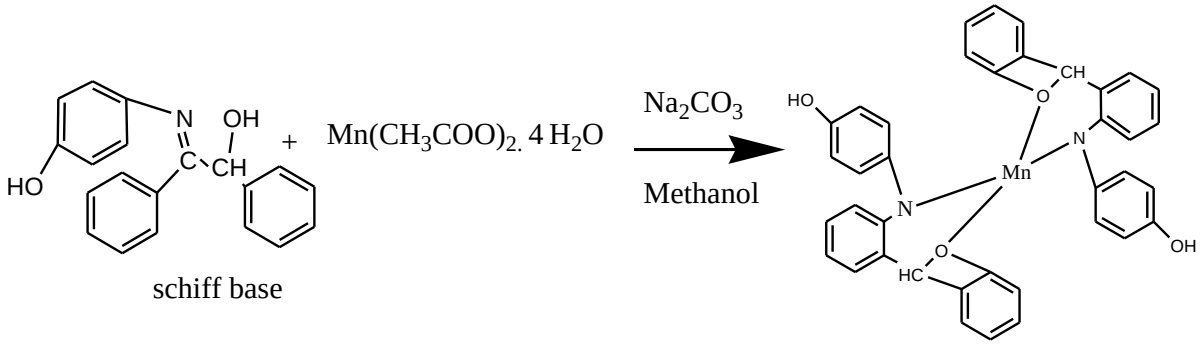
schiff base

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Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction mixture with continuous stirring. The reaction mixture cooled down at room temperature. Zinc acetate (0.164g,0.894mmoles) dissolved in methanol was added drop wise in the reaction mixture under continuous stirring. The mobile phase employed for TLC which was ethyl acetate and n-hexane in ratio 1:1.

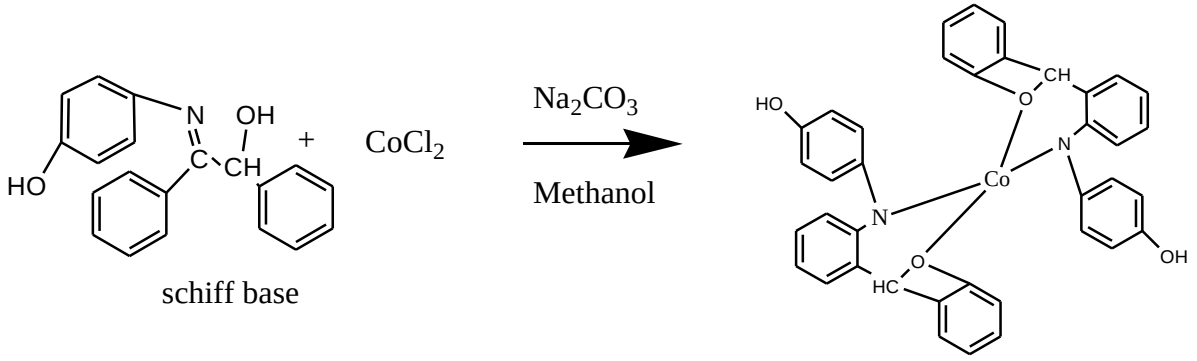
Synthesis of Manganese (II) complex with ligand
Equation:



schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction misture with continuous stirring .The reaction mixture cooled down at room temperature.Manganese acetate (0.164g,0.669mmoles) dissolved in methanol was added dropwise in the reaction mixture under continuous stirring. The reaction mixture was reflux for 2 hours.The clay colored precipitates obtained which were washed with water and then dried at room temperature. The mobile phase employed for TLC which was ethyl acetate and n-hexane in ratio 1:1.

Synthesis of Cobalt (III) complex with ligand
Equation:



schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction misture with continuous stirring. The reaction mixture cooled down at room

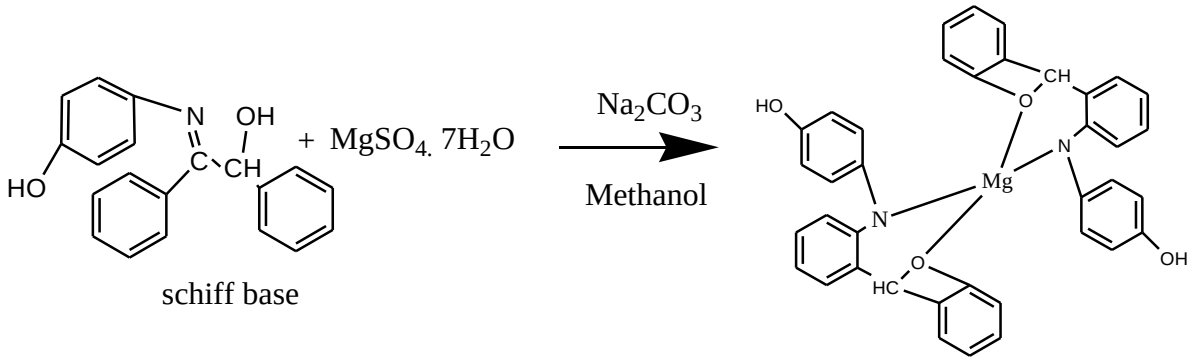
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temperature.Cobalt Chloride(0.164g,1.263mmoles) dissolved in methanol was added dropwise in the reaction mixture under continuous stirring. The reaction mixture was reflux for 2 hours. The clay colored precipitates obtained which were washed with water and then dried at room temperature. The mobile phase employed for TLC which was ethyl acetate and n-hexane in ratio 1:1.

3.3.6 Synthesis of Magnesium sulfate (VI) complex from schiff base

Equation:

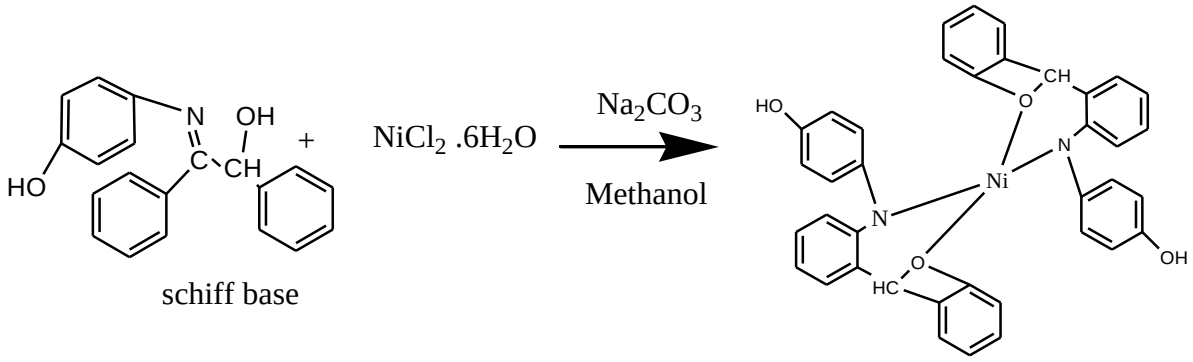


schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction misture with continuous stirring.The reaction mixture cooled down at room temperature. Magnesium sulfate (0.164g,0.665mmoles) dissolved in water was added drop wise in the reaction mixture under continuous stirring.

Synthesis of Nickel (II) chloride complex from schiff base

Equation:



schiff base

Ligand (0.5g,1.64mmoles) was dissolved in 15mL of methanol. Na2CO3 (0.179g,0.00164mmoles) was dissolved in 2mL of water and added into the reaction misture with continuous stirring. The reaction mixture cooled down at room temperature.Nickel Chloride(0.164g,0.689mmoles) dissolved in methanol was added drop wise in the reaction mixture under continuous stirring. The reaction mixture was reflux for 2 hours. The green colored precipitates obtained which were washed with water and then dried at room temperature. The mobile phase employed for TLC which was ethyl acetate and n-hexane in ratio 1:1.

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Volume 4, Issue21 (2026)

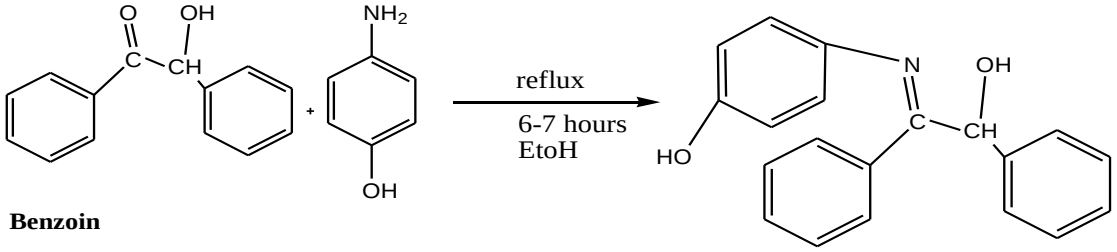
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RESULT AND DISCUSSION

The derivation of new ligand and their different 3D Transition metal complexes were examined and characterized by different types of physical methods and spectroscopic technique like FT-IR is described in this chapter.

Synthesis of ligand:

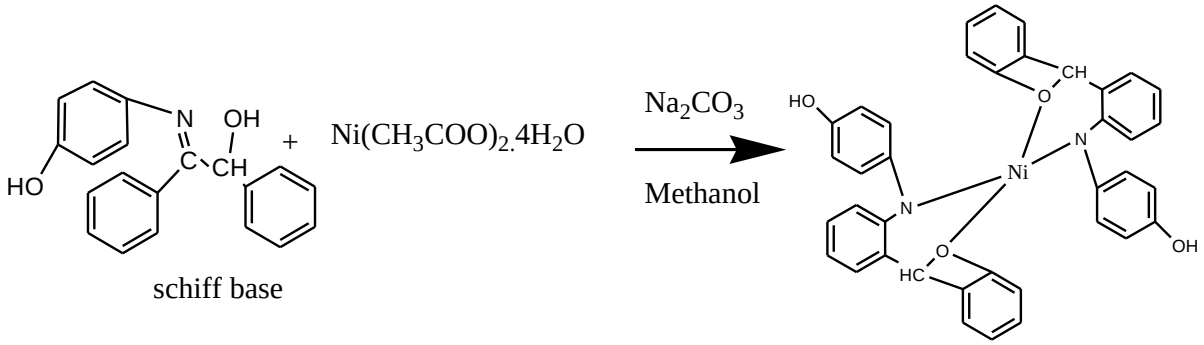


Benzoin **4-aminophenol** **Ligand**

Melting point=
Physical Appearance= Brown color
Conductivity value= (1 Mm Solution of MeOH) 0.13µS/cm
So, value of conductivity is low which proved that almost insignificance value of conductivity equal to zero. Clearly results confirmed that ligand nature was non-ionic.Practically ,at room temperature it is insoluble in water,soluble in ethyl acetate and hexane.

FT-IR=KBR (DISC,cm^{-1}): 1568(α,β –unsat ketones),2992,3039(CH $_{aliphatic}$,CH $_{arpmatic}$),2671 (C-C $_{aliphatic}$),1701 (C=C $_{aromatic}$)
Melting results and FTIR results indicated the information of new compound.This is showed by the disappearing peak of aromatic Benzoin stretching frequency at 1696 cm^{-1} which was found in the spectrum of the reactant.Shifting of 4-aminophenol peak towards narrow peak at 1568 cm^{-1},this showed the sysnthesis of α,β-unsaturated diketones.Peaks are present in the broad region at 2992 cm^{-1} and 3039 cm^{-1} which was stretching frequency of aromatic C=C and aliphatic C-C groups present at 1701 cm^{-1} and 2671 cm^{-1} respectively.4.2 Synthesis of M-L Complexes

Synthesis of Nickel metal complexes with Ligand
Equation:



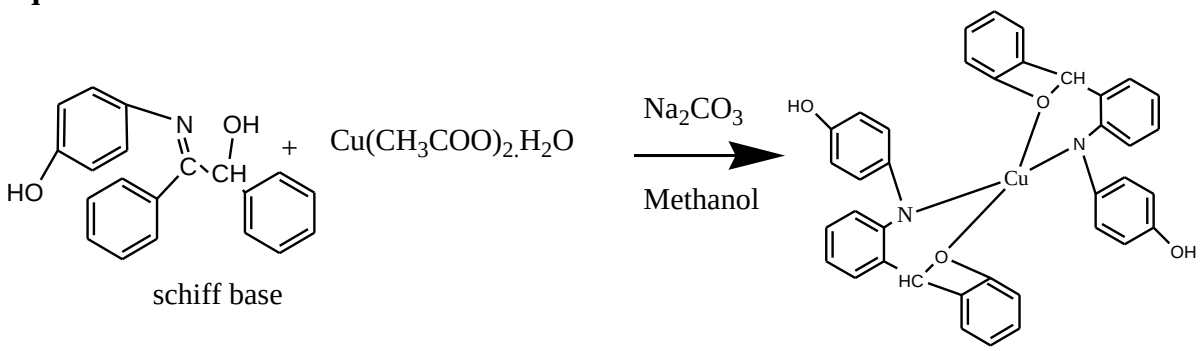
schiff base

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Volume 4, Issue21 (2026)

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Melting point:
Physical appearance: Light Green
Conductivity Value: (1mM solution of MeOH) 0.09 µS/cm
So, value of conductivity is low which proved that almost insignificance value of conductivity equal to zero. Clearly results confirmed that ligand nature was non-ionic.Practically ,at room temperature it is insoluble in water,soluble in ethyl acetate and hexane.
FT-IR = KBr (Disc,cm^{-1}) : 2550-3615 (water coordinated) 1560 (C-N $_{aromatic}$) 1427 (C= N $_{aromatic}$)
3022 (CH $_{aromatic}$),1703 (C=C $_{aromatic}$) 2700 (C-C $_{aliphatic}$)2426 (C-C)530 (Ni-Ni).
Melting point and FTIR results have been supported the formation of new proposed Ni complex.The shifting of strtching frequency of C=N $_{aromatic}$ at 1571 cm-1 to 1427 cm-1 which prove that coordination present in iminic nitrogen and metal ion.A narrow peak present at 530 cm-1 is of Ni-N value of stretching frequency.Bond appreared in broad region at the value of 2550-3615 cm-1 which shows the coordination of water.

FTIR result of Nickel metal complex with Ligand
Synthesis of Copper metal complex with Ligand
Equation:



schiff base

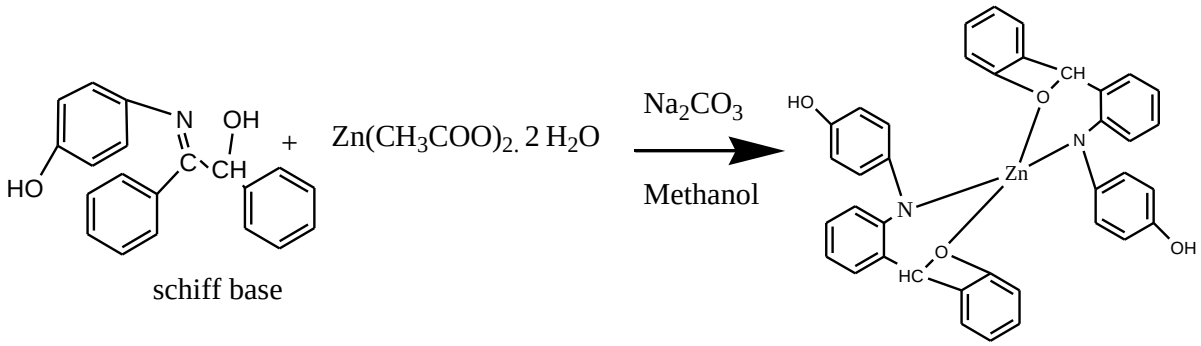
Melting Point:
Physical Appearance: Green
Conductivity Value: (1Mm ,MeOH + DMSO)0.04µS/cm
So,value of conductivity is low which proved that almost insignificance value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-ionic .Practically ,at room temperature it is insoluble in water, in ethyl acetate and hexane.
FT-IR = KBr (Disc cm^{-1}): 152550-3615 (water coordinated) 1568 (C-N $_{aromatic}$) 1421(C=$N_{aromatic}$) 3037 ($CH_{aromatic}$) ,2927 (CH $_{aliphatic}$),1878 (C=C $_{aromatic}$)2364 (C-$C_{aliphatic}$) 1691 (H-N $_{aliphatic}$) 433 (Cu-N)
Melting point and FTIR results have been supported the formation of new proposed Cu complex.The shifting of stretching frequency of C=N aromatic at 1571 cm-1 which proved that coordination present in iminic nitrogen and Cu metal ion.A narrow peak present at 433cm-1 is of Cu-N value of stretching frequ ency.Bond appeared in broad region at the value of 2550-3615cm-1 which shows the coordination of water.

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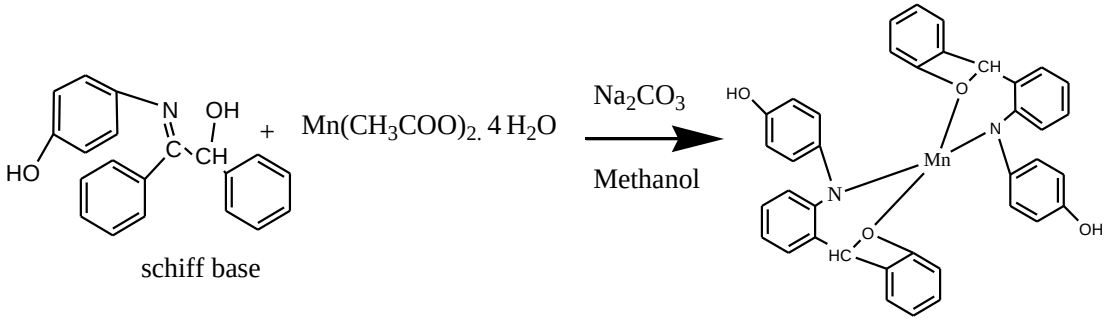
FTIR result of Copper metal complexes with Ligand
Synthesis of Zinc metal complex with ligand
Equation:



schiff base

Melting Point:
Physical Appearance: off white
Conductivity value: (1Nm,MeOH + DMSO) 0.18 µs/cm
So,Value of conductivity is low which proved that aimost insignificant value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-ionic.Practically,at room temperature it is insoluble in water,soluble in water,soluble in ethyl acetate and hexane.
FT-IR =KBr (disc, cm-1): 2550 -3615 (water coordinated) 1301 (C-N aromatic) 1427 (C=N aromatic) 3034 (CH aromatic),2926 (CH aliphatic),1710 (C=C aromatic)2314 (C=C aliphatic) 2408 (C-C)1568 (H-N aliphatic)447 (Zn-N)
Melting point and FTIR results have been supported the formation of new proposed Zn complex.The shifting of stretching frequency of C=N aromatic at 1571 cm^{-1} to 1427 which proved that coordination present in iminic nitrogen and Zn metal ion.A narrow peak present at 447 cm-1 is of Zn-N value of stretching frequency.Bond appeared in broad region at the value of 2250-3615 cm-1 which shows the coordination of water.

FTIR result of Zn metal complex with ligand
Synthesis of Manganese metal complex with Ligand
Equation:



schiff base

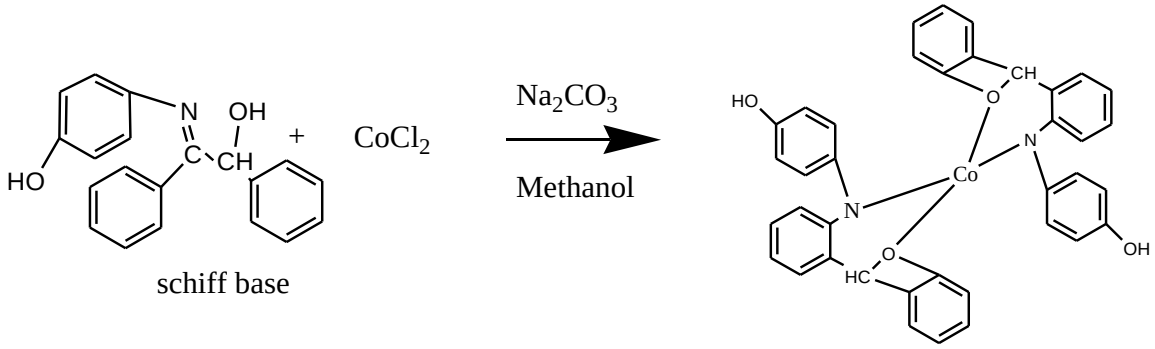
Melting point:
Physical appearance:clay color

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Volume 4, Issue21 (2026)

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Conductivity value: (1Mm, MeOH + DMSO) 0.15 µS/cm
So,Value of conductivity is low which proved that aimost insignificant value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-ionic.Practically,at room temperature it is insoluble in water,soluble in water,soluble in ethyl acetate and hexane.
FT-IR =KBr (disc, cm-1): 2550 -3615 (water coordinated) 1662 (C-N aromatic) 1427 (C=N aromatic) 3041 (CH aromatic),2964 (CH aliphatic),1921 (C=C aromatic)2970-2950 (C-C aliphatic) 2931 (C-C) 486 (Mn-N)
Melting point and FTIR results have been supported the formation of new proposed Mn complex. The shifting of stretching frequency of C=N aromatic at 1571 cm^{-1} to 1406 which proved that coordination present in iminic nitrogen and Zn metal ion.A narrow peak present at 486 cm-1 is of Zn-N value of stretching frequency. Bond appeared in broad region at the value of 2550-3615 cm-1 which shows the coordination of water.

FTIR result of Manganese metal complex wih Ligand
Synthesis of cobalt metal complex with Ligand
Equation:



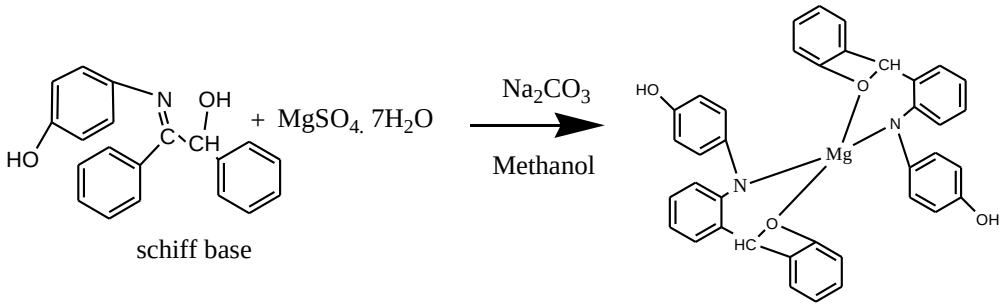
schiff base

Melting Point =
Physical appearance = Clay color
Conductivity Value = (1Mm ,MeOH + DMSO) 0.08 µS/cm
So,Value of conductivity is low which proved that aimost insignificant value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-ionic.Practically,at room temperature it is insoluble in water,soluble in water,soluble in ethyl acetate and hexane.
FT-IR =KBr (disc,cm-1) : 2550 -3615 (water coordinated) 1301 (C-N aromatic) 1427 (C=N aromatic) 3034 (CH aromatic),2926 (CH aliphatic),1710 (C=C aromatic)2314 (C-C aliphatic) 2408 (C-C)1568 (H-N aliphatic)447 (Zn-N)
Melting point and FTIR results have been supported the formation of new proposed Co complex.The shifting of stretching frequency of C=N aromatic at 1571 cm^{-1} to 1427 which proved that coordination present in iminic nitrogen and Co metal ion.A narrow peak present at 447 cm-1 is of Co-N value of stretching frequency.Bond appeared in broad region at the value of 2250-3615 cm-1 which shows the coordination of water.

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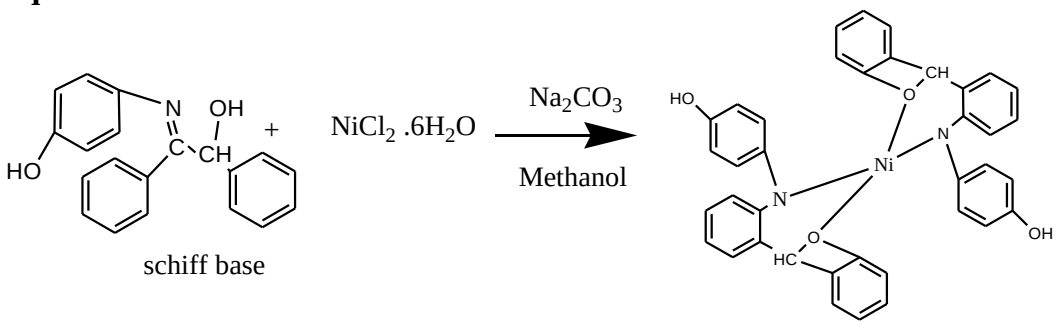
FTIR result of cobalt complex with ligand
Synthesis of Magnesium Sulfate complex with Ligand
Equation:



schiff base

Melting Point =
Physical appearance = Clay color
Conductivity Value = (1Mm ,MeOH + DMSO) 0.08 µS/cm
So,Value of conductivity is low which proved that aimost insignificant value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-ionic.Practically,at room temperature it is insoluble in water,soluble in water,soluble in ethyl acetate and hexane.
FT-IR =KBr (disc,cm-1) : 2550 -3615 (water coordinated) 1301 (C-N aromatic) 1427 (C=N aromatic) 3034 (CH aromatic),2926 (CH aliphatic),1710 (C=C aromatic)2314 (C-C aliphatic) 2408 (C-C)1568 (H-N aliphatic)
Melting point and FTIR results have been supported the formation of new proposed Magnesium complex.The shifting of stretching frequency of C=N aromatic at 1571 cm^{-1} to 1427 which proved that coordination present in iminic nitrogen and Mg metal ion.A narrow peak present at 447 cm-1 is of Mg-N value of stretching frequency.Bond appeared in broad region at the value of 2250-3615 cm-1 which shows the coordination of water.

FTIR result of Magnesium sulfate complex with Ligand
Synthesis of Nickel chloride complex with Ligand
Equation:



schiff base

Melting Point =
Physical appearance = Clay color
Conductivity Value = (1Mm ,MeOH + DMSO) 0.08 µS/cm
So,Value of conductivity is low which proved that aimost insignificant value of conductivity equal to zero.Clearly results confirmed that ligand nature was non-

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Volume 4, Issue21 (2026)

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ionic.Practically,at room temperature it is insoluble in water,soluble in water,soluble in ethyl acetate and hexane.

FT-IR =KBr (disc,cm-1) : 2550 -3615 (water coordinated) 1301 (C-N aromatic) 1427 (C=N aromatic) 3034 (CH aromatic),2926 (CH aliphatic),1710 (C=C aromatic)2314 (C-C aliphatic) 2408 (C-C)1568 (H-N aliphatic)

Melting point and FTIR results have been supported the formation of new proposed Ni complex.The shifting of stretching frequency of C=N aromatic at 1571 cm^{-1} to 1427 which proved that coordination present in iminic nitrogen and Ni metal ion.A narrow peak present at 447 cm-1 is of Ni-N value of stretching frequency.Bond appeared in broad region at the value of 2250-3615 cm-1 which shows the coordination of water.

FTIR result of Nickel complex with Ligand

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