



# Schiff Base Ligand System Containing ONO Donor Atoms and Synthesis of Metal Complexes by Direct and Template Methods: A Review

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## KEYWORDS

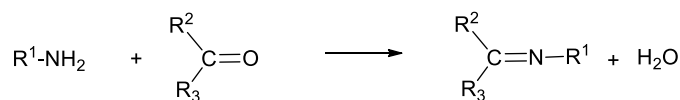
Schiff base, metal complex, Azomethine, antibacterial, Antifungal

## ABSTRACT:

Schiff bases are obtained by condensation reaction of primary amine with an aldehyde or ketone. Schiff base contains azomethine (>C=N-) functional group which play major role in the progress of coordination chemistry. These contain donor atoms like Oxygen, Nitrogen and Sulphur. Their ability to react with transition metals to form stable complexes makes them very useful in wide range of synthesis. These find numerous applications in various fields, including medicinal chemistry, catalysis and chemosensing. This review focused on Schiff base system containing ONO donor atoms and methods employed to form complexes with metals along their brief spectral aspects and applications.

## Introduction

A Schiff base is nitrogen equivalent of an aldehyde or ketone in which carbonyl (>C=O) group is replaced by azomethine (>C=N-R) group. Schiff bases are generally obtained by the condensation reaction of an aldehyde or ketone with a primary amine. They are formed by the following reaction Scheme 1.



**Scheme 1. General scheme for synthesis of Schiff bases (R<sup>1</sup>, R<sup>2</sup> and R<sup>3</sup> may be an alkyl or aryl group).**

Schiff bases having aryl substituents can be prepared easily and due to effective conjugation found to be more stable, whereas those containing alkyl substituents are comparatively less stable.

The synthesis of Schiff bases have numerous ligating ability has drawn a lot of interest because of their vital use as analytical reagents and metal sensors in complexometric titration [1]. The azomethine group – N=CH- enables the process of transamination and racemization reaction in biological systems [2].

Therefore, they possess significant biological activities like antifungal and antibacterial properties, antifertility and insecticidal activities [3-7]. Some Schiff bases demonstrate high biocidal activity, cytotoxic and antitumor activities [8-10].

Schiff bases have high thermal stability; therefore, they can be used as stationary phases in chromatography [11]. They have coordinating sites which can capture the metal ions and impart colors with some specific metal ions; hence they are used to sense some particular ions by naked eye. In the past, Schiff base moieties are used for designing very efficient and sensitive chemosensors for recognition of metal ions in aqueous and non aqueous medium [12,13].

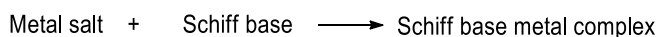
Their metal coordinating property makes them useful in the catalysis. They form homogenous and heterogeneous catalysts and are widely used in medicinal and pharmaceutical fields [14,15]. The heterogeneous catalysis using Schiff bases involves immobilization of Schiff bases on to silica surfaces by grafting and co-condensation. Their diverse nature for binding metal ions results in the varying coordination number of metal ions which is useful for catalyzing a chemical reaction. Till date, they have been used for



catalyzing numerous organic reactions like C-C bond formation, cross-coupling reactions, oxidation-reduction reactions etc.

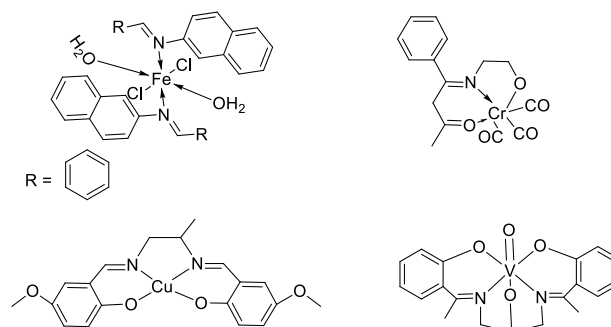
### Schiff Base metal complexes

Schiff bases are recognized as one of the important class of compounds, and have been widely studied for over one and half century. Their importance has been increased over the time due to their easy approach of synthesis, their versatility and high complexing ability once they formed. Most of the transition metals generally contain partially filled orbitals, which accept lone pair of electron from ligands to form stable metal complexes. Schiff base-metal complexes have been reported by nearly all the transition metals in the periodic table [16]. In general metal complexes of Schiff base can be synthesized by following general method.



The property of Schiff base metal complexes are significantly influenced by metal center and ligands. The Schiff base obtained from the condensation reaction of diamine and ketones generally give a macrocyclic ligand with cavity of different dimensions.

Schiff base ligands contain Nitrogen, Oxygen and sulphur donor atoms which are used to coordinate with different metal ions. Schiff base ligands coordinate with metal through lone pair present on azomethine nitrogen and other groups generally contains oxygen or sulphur. Tetradentate Schiff base containing N<sub>2</sub>O<sub>2</sub> donor atoms are extremely significant in coordination with different metal ions. They have garnered considerable attention in recent years because of their rich coordination chemistry [17-22]. The Schiff base ligands typically coordinated with metal centers via two imine nitrogen and two oxygen atoms from phenolic oxygen atoms. These four donor site makes them particularly effective for equatorial coordination with additional availability of two vacant sites for coordination with auxiliary ligands around metal center Figure 1.



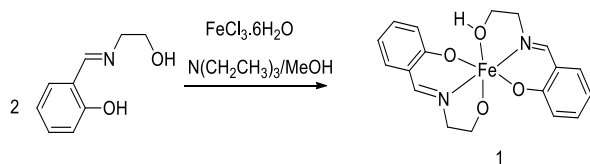
**Figure 1. Structure of some Schiff base metal complexes**

### Synthesis of metal complexes

The synthesis of Schiff base metal complexes can be achieved by direct method or by template synthesis. In the direct method, Schiff bases are prepared and isolated in the first step followed by reaction with metal salts to obtain corresponding complexes. However, in template synthesis, all the reactants i.e. aldehyde or ketone, carbonyl and metal salts are reacted in a single step without undergoing the step of isolation of Schiff base. These methods are discussed below in detail.

#### Direct synthesis

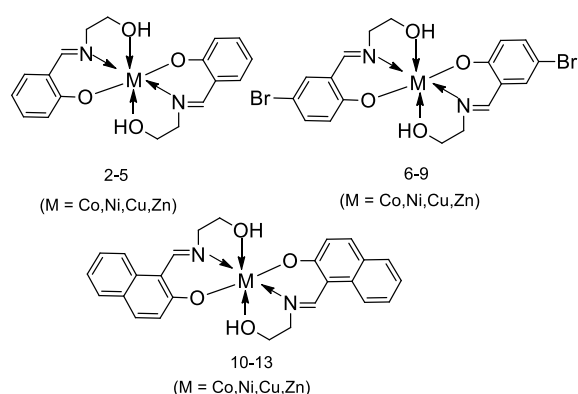
Metal salts like metal chlorides, metal nitrates, metal acetates etc., when dissolved in the polar solvents such as alcohols and allowed to react with Schiff base moieties led to the formation of colored complexes. In some cases a small amount of base or acid increased the rate of reaction. For example A red colored iron(III) phenoxy-imine complex formed after the addition of FeCl<sub>3</sub>·6H<sub>2</sub>O to methanolic solution of 2-[(2-hydroxyethyl)imino]methyl]-phenol followed by addition of triethylamine have been reported Scheme 2 [23]. The complex 1 was characterized by different spectral techniques. The studies shows octahedral geometry around Iron with the six positions occupied by two iminic-N atoms, one ethanolate-O atoms and two hydroxyl-OH atoms a strong H-bond between ethanolate and ethanol group of neighbouring molecules stabilized the crystal packing.



**Scheme 2. Synthesis of phenoxy-imine iron complex 1**

The reported iron complex can be used to start opening of cyclic ring polymerization of L-lactide in the presence of initiator (benzyl alcohol). Despite the lower electrophilic nature of metal centre in hexa-coordinated environment as compared to less saturated structures, it offered an interesting example to produce polylactide, believes to occur through coordination-insertion mechanism.

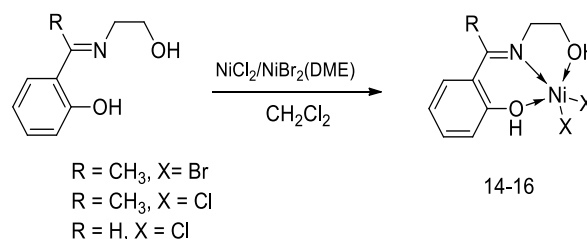
In addition Amjad and coworkers have reported mixed complexes of Cu(II), Co(II), Ni(II) and Zn(II) with ligands containing ONO donors, obtained by reaction of 2-hydroxybenzaldehyde, 5-bromo-2-hydroxybenzaldehyde and 2-hydroxy-1-naphthaldehyde with ethanol amine Figure 2 [24]. All the metal complexes showed intense color except Zn(II) complex which was white in color.



**Figure 2. Schiff base metal complexes 2-13**

The reported metal complexes 2-13 were characterized by various spectroscopic techniques. Their low molar conductance value showed non-electrolytic behavior. Schiff base ligands were found to be less potent than their corresponding metal complexes, when investigated for their antibacterial and anti fungal activities.

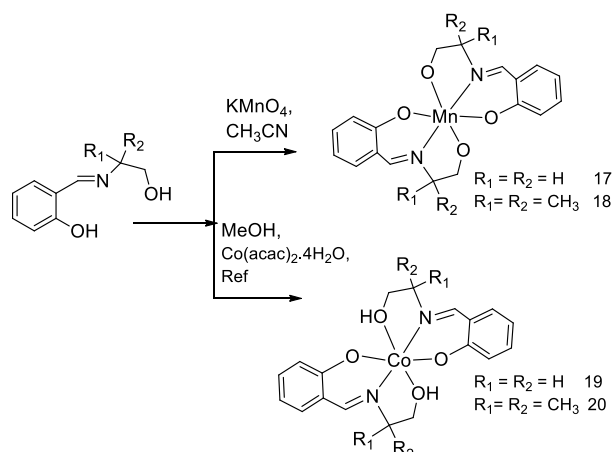
Green colored nickel(II) complexes 14-16 were obtained from the Schiff base ligands, prepared by condensation reaction of substituted aldehyde with 2-aminoethanol by Ngocobo and co-workers Scheme 3 [25]. Out of these, the complex 16 was recrystallized from methanol/diethylether solution for single crystal X-ray studies.



**Scheme 3. Synthesis of Ni(II) complexes 14-16**

The single crystal X-ray studies of complex 16 established monoclinic system with P2<sub>1</sub>/n space group. Further catalytic activity of these complexes was investigated for ethylene oligomerization reaction by using EtAlCl<sub>2</sub> as activator in chlorobenzene. The results showed that structure and other reaction parameters influence the catalytic activity of Ni(II) complexes.

More Mn(II) and Co(II) complexes 17-20 of tridentate –OH rich Schiff base which were synthesized and compared with their analogues of Schiff base reported from salicylaldehyde and 2-amino-2-methyl-propanol Scheme 4 [26].

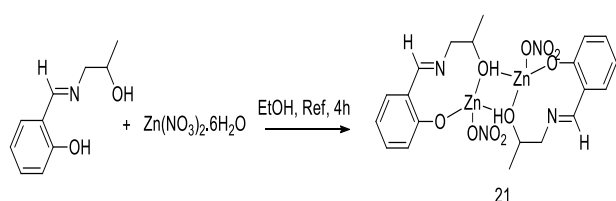


**Scheme 4. Preparation of complexes 17-20**

The single crystal X-ray studies of complex 20 possessed monoclinic system with P2<sub>1</sub> space group and



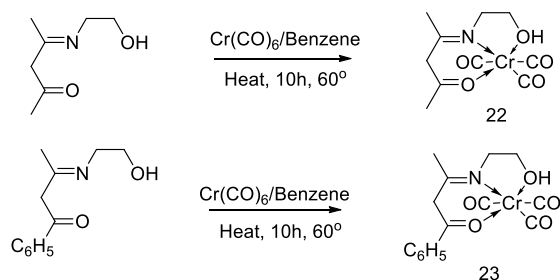
revealed distorted octahedral structure of the Co(III) complex with  $O_4N_2$  coordination sphere. In another approach a block-shaped crystal of Zn(II) complex 21 of Schiff base (2-[(2-hydroxypropylimino)methyl]phenol) have been reported and was investigated by different spectral studies Scheme 5 [27]. The Zn(II) complex was further explored for its photoluminescence properties and to fabricate ZnO nanoparticles by solid state transformation of complex at  $700^\circ$ .



**Scheme 5. Synthesis of di-nuclear Zn(II) complex 21**

The synthesized complex was a dimer consisting of two Zn(II) ions and two tridentate Schiff base ligand. The metal was coordinated to ligand *via* ethanolic oxygen atoms, imine nitrogen, deprotonated phenolic oxygen and two monodentate nitrate anions. The coordination around Zn is five and its geometry lies between trigonal bipyramidal and square pyramidal.

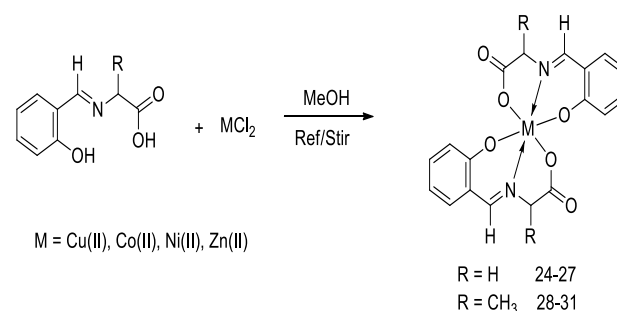
Soleman and co-workers synthesized another brown colored complexes 22, 23 were obtained when a mixture of 2,4-diketones and  $Cr(CO)_6$  dissolved in benzene was heated in a sealed tube at  $60^\circ C$  for 10 hrs Scheme 6 [28]. The shift in vibrational frequency of imine  $-HC=N-$  to lower values indicated the participation of imine group in coordination.



**Scheme 6. Synthesis of complexes 22,23**

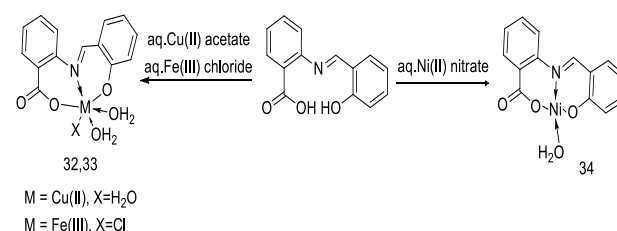
Chohan and others reported another series of Schiff bases, synthesized from salicylaldehyde and corresponding amino acids (like alanine and glycine)

were used for the formation of their metal complexes with Zn(II), Cu(II), Ni(II) and Co(II) 24-31 Scheme 7 [29]. The formation of metal complexes was supported by different spectral methods. The reported ligands along their metal complexes were checked for their antifungal and antibacterial activity against different fungal and bacterial strains. The study revealed that upon metal complexation antifungal and antibacterial activities were significantly increased in comparison of Schiff base alone.



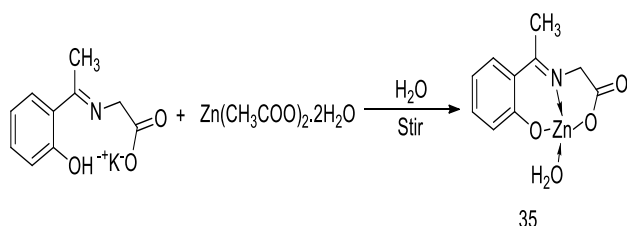
**Scheme 7. Structure of metal (II) complexes 24-31 of amino acid Schiff base ligand**

Similarly, anthranillic acid based Schiff base and its metal complexes 32-34 of Ni(II), Fe(III) and Cu(II) have been investigated for in-vitro antibacterial (*Escherichia coli*) and *Bacillus cereus*) and antifungal actions by Mawgoud and co-workers Scheme 8 [30]. Studies indicated that metal complexes were found to be more biological active than parent Schiff bases.



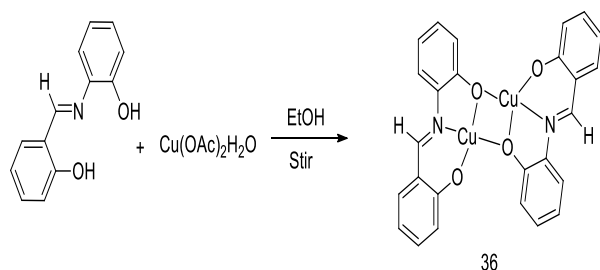
**Scheme 8. Synthesis of amino acid based metal complexes (32-34)**

In another approach a white colored Zn(II) metal complex 35 of tridentate Schiff base prepared from potassium (N-2-hydroxyacetophenone)glycinate and zinc acetate dehydrate Scheme 9 [31]. The formation of metal complex was revealed by its spectral studies.



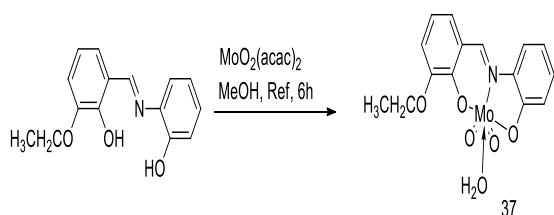
**Scheme 9. Synthesis of Zn(II) complex 35**

In addition, a green colored Cu(II) complex 36 of tridentate Schiff base prepared by refluxing an ethanolic mixture of salicylaldehyde with 2-aminophenol have been reported [32]. The Cu(II) complex shows square planar geometry and was attained by coordination of Schiff base ligand to the azomethine N, phenolic O atoms and bridging *via* one of enolic oxygen as shown in Scheme 10.



**Scheme 10. Preparation of Cu(II) complex 36**

An orange coloured Mo(VI) complex 37 which was synthesized by the addition of  $\text{MoO}_2(\text{acac})_2$  to the ethanolic solution of Schiff base obtained from 3-ethoxysalicylaldehyde and 2-aminophenol Scheme 11 [33].

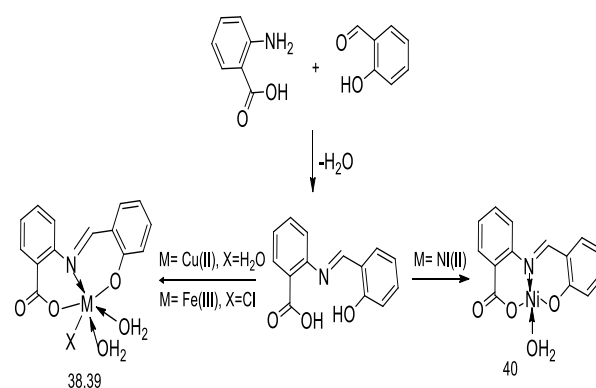


**Scheme 11. Preparation of Mo(VI) complex 37**

The confirmation of synthesized Mo(VI) complex 37 was accomplished by various spectral techniques. The single crystal X-ray study of Mo(VI) complex confirmed monoclinic system for complex with  $P2_1/c$

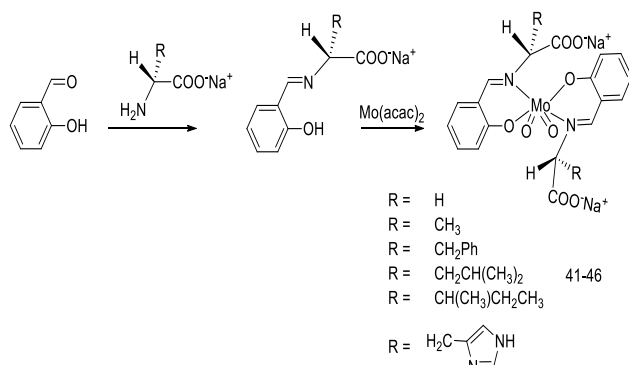
space group. The asymmetric unit was consisted of two molecules of complex besides a non-coordinated water molecule.

Mawgoud and others explored an anthranilic acid based Schiff base ligand for the synthesis of Cu(II), Ni(II) and Fe(III) complexes 38-40 Scheme 12 [34]. Different spectral studies confirmed the metal complexation occur through oxygen atom of hydroxyl part of phenolic and carboxylic groups. The studies shows octahedral geometry for Cu(II) and Fe(III) complexes whereas square planar geometry was confirmed Ni(II) complexes. Furthermore the synthesized Schiff base along with its complexes was investigated for their *in-vitro* antibacterial activities. The studies revealed a significant increase in activity upon complexation.



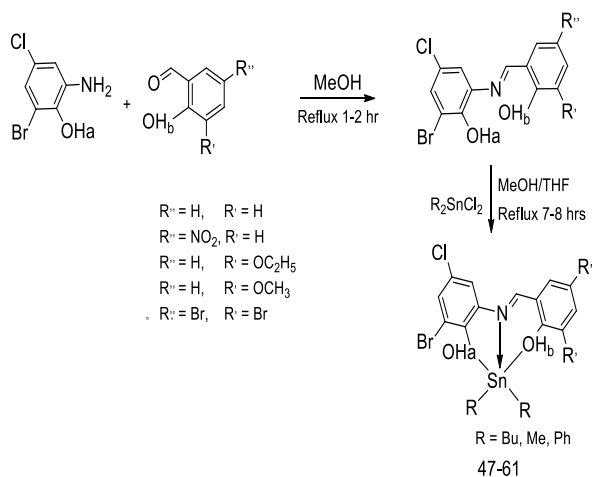
**Scheme 12. Structure of Cu(II), Fe(III) and Ni(II) complexes 38,39 and 40**

Mo(VI) complexes 41-46 acts as heterogeneous catalysts were reported with Schiff base ligands obtained from amino acids (glycine, alanine, phenyl alanine, leucine, isoleucine, histidine) and salicylaldehyde were synthesized by Farahani and co-workers Scheme 13 [35]. The reported metal complexes immobilized into the SBA-16 nanocages followed by silylation were used as heterogeneous catalysts for olefin epoxidation of with TBHP. The studies revealed high activity and selectivity for epoxidation reaction. Moreover the synthesized catalysts can be reused several times without losing their activities and selectivity.



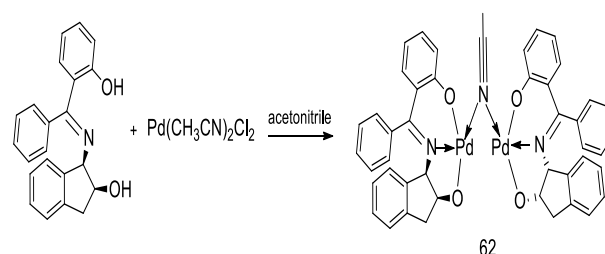
**Scheme 13. Synthesis of amino based Mo(VI) complexes 41-46**

A series of colored diorganotin(IV) complexes 47-61 with another Schiff base ligands have been reported, which were synthesized by the reaction of salicylaldehyde derivatives with 2-amino-6-bromo-4-chlorophenol Scheme 14 [36]. The reported Schiff bases along their metal complexes were further investigated for their antimicrobial and antifungal activities against different bacterial and fungal strains. The studies revealed that phenyl substituted metal complexes showed higher activity in comparison of methyl and butyl substituted complexes. Moreover these complexes were also investigated for their anticancer activities against MCF7, HepG2 and A549 cell lines, in which maximum activity was found against A549 cell line.



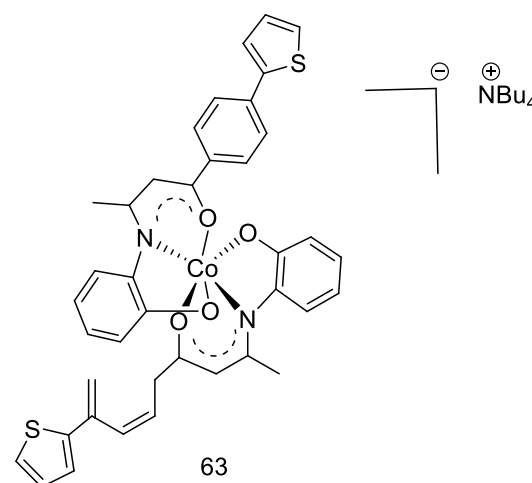
**Scheme 14. Synthesis of organotin(IV) complexes (47-61)**

Recently a new acetonitrile-bridged dinuclear Pd(II) complex 62 have been reported with a chiral Schiff base ligand containing ONO donor atoms Scheme 15 [37]. The synthesized dinuclear metal complex was connected by a bridging acetonitrile.



**Scheme 15. Synthesis of acetonitrile-bridged dinuclear Pd(II) complex 62**

A thiophenyl based Schiff base ligand containing ONO donor atoms have been reported by Gonzalez and co-workers and was further reacted with Co(II) acetate tetrahydrate to form a cobaltate(III) complex 63 Figure 3 [38]. Various spectral and X-ray study confirmed the formation of metal complex having hexa coordination around Co(III) ion. The reported complex acts as a high performance electrochemical glucose sensor.



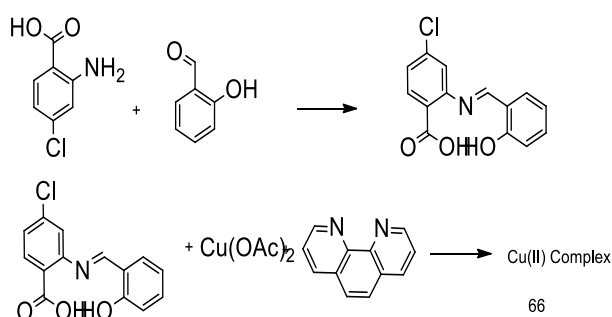
**Figure 3. Structure of Co(III) complex 63**

Tang and coworker have reported another Schiff base ligand which was prepared by stirring an ethanolic solution salicylaldehyde and 4-amino antipyrine for 6hrs, was further used to react with NiCl<sub>2</sub>.6H<sub>2</sub>O and Neodymium nitrate to form respective metal complexes



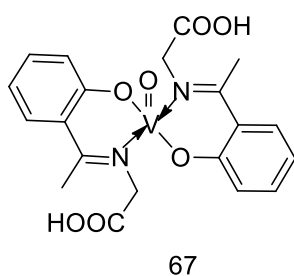
64,65 [39]. The single crystal X-ray diffraction studies showed that Both Ni(II) and Nd(III) complexes crystallized in monoclinic space group  $P_2(1)/n$ . Both complexes were screened for their antimicrobial activity. The Results showed that upon complexation antimicrobial activity was significant increased. Moreover Ni(II) complex displayed better antimicrobial activity in comparison of Nd(III) complex.

Li and coworkers have reported a ternary Cu(II) complex 66 with Schiff base, obtained from salicylaldehyde and 4-chloroanthranilic acid and was characterized by various spectral techniques Scheme 16 [40]. Further the reported complex was found to be effective catalyst for the degradation of organophosphorus pesticides.



**Scheme 16. Preparation of Schiff base and its Cu(II) complex 66**

A yellow colored Schiff base ligand obtained from glycine and 2-hydroxyacetophenone and was further used to prepare vanadium complex 67 and was characterized by UV-Vis, FT-IR, and NMR techniques Figure 7 [41].

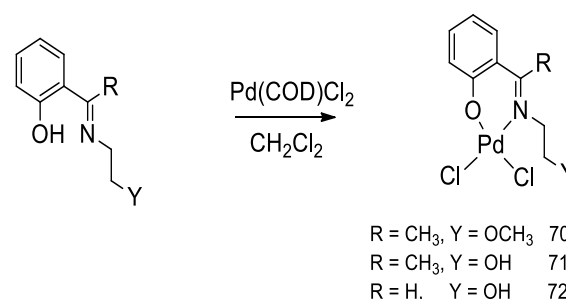


**Figure 4. Structure of vanadium complex 67**

The reported vanadium complex was found to be effective anticancer agent without any significant toxic affect when tested on female Swiss albino mice.

Mixed metal complexes of Cu(II) and Fe(III) 68,69 with Schiff base ligand obtained by condensation reaction of orthoaminophenol with orthohydroxyl acetophenone have been reported and were characterized by FT-IR, XRD and VSM techniques [42]. The studies confirmed that at high temperature Fe(III) complex was found to be paramagnetic but changes to antiferromagnetic at lower temperature.

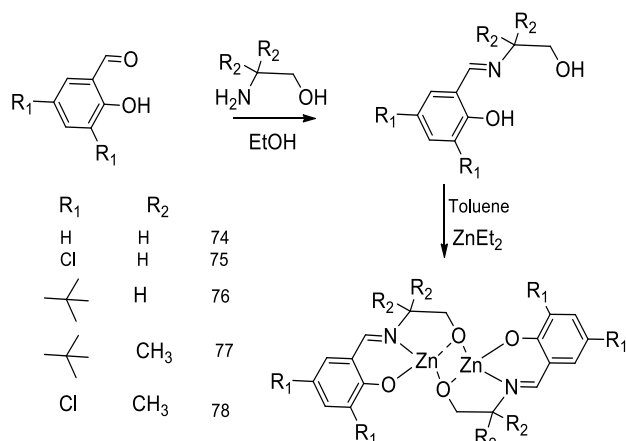
Pd(II) complexes 70-72 with iminophenol Schiff base ligands displayed catalytic properties have been reported [43]. The reported Pd(II) complexes have been used as an effective catalyst for hydrogenation reaction of alkene and alkynes Scheme 17.



**Scheme 17. Synthesis of 70-72 complexes**

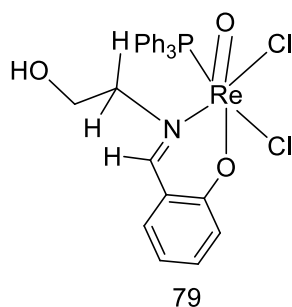
In another approach a green color tetranuclear clustered Ni(II) complex 73 with Schiff base ligand salicylidene-2-ethanolamine and its magnetic anisotropy studies were conducted by polarized neutron diffraction techniques [44]. Its single X-ray crystal studies confirmed monoclinic system with  $P2_1/n$  space group

Ranlong and others have reported the synthesis of five Zn(II) complexes 74-78 with Schiff base ligand containing ONO donor atoms Scheme 18 [45]. The reported Zn(II) complexes were explored for their catalytic activity in ring opening polymerization(ROP) of L-lactide and rac-lactide. The studies show that presence of bulky groups on Schiff base ligand reduced the polymersization activity.



**Scheme 18. Preparation of metal complexes 74-78**

Another Schiff base ligand obtained from salicylaldehyde and ethanolamine were explored for the synthesis of distorted octahedral oxorehenium(V) complexes 79 by smit and co-workers Figure 5 [46]. The reported complex was characterized by different spectral and structural techniques which confirmed monoclinic crystal system with  $P2_1/n$  space group.



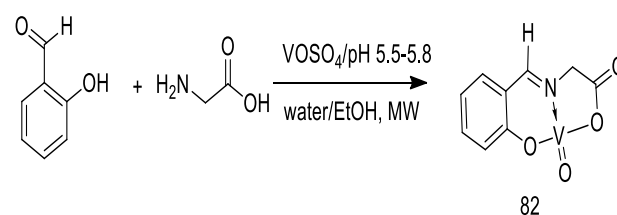
**Figure 5. Structure of complex 79**

More Ni(II) and Cu(II) complexes 80,81 with N-salicylidene-glycine Schiff base ligand have been reported [47]. The studies show square pyramidal geometry for Cu(II) complex whereas Ni(II) complex exhibited distorted octahedral geometry.

In the above-mentioned methods, ligands were isolated in the pure form and then reacted with metal ions, therefore, mononuclear and pure products were reported in most of the cases. However, synthesis of some of the Schiff base ligands was found difficult directly, so the alternative method for their synthesis have been reported.

### Template synthesis

Another method employed for the synthesis of Schiff base metal complexes is template synthesis. The term “template” has been introduced by Daryle Busch in 1963 in the field of coordination chemistry. The word “template” in the *Oxford English Dictionary* is defined as “an instrument used as a mentor in bringing any exercise to the desired shape”. In template processes, the metal ion with a definite stereochemistry, serves as mould, pattern or the matrix for forming reaction products which are difficult under normal conditions. The template synthesis of transition metal complexes provides different and additional advantage to swift the complexation process. Many researchers have taken the advantage of template synthesis. Wazalwar and others have synthesized an oxo-vanadium complex 82 by template synthesis [48]. For this, warm aqueous solution of glycine (pH ranges from 5.5 to 5.8 using buffer solution, acetic acid and sodium acetate) was mixed with vanadyl sulphate Scheme 19. Then, alcoholic solution of salicylaldehyde was added drop-wise and the resulting mixture was irradiated with microwaves for 10 seconds in the installments at 210-240W.



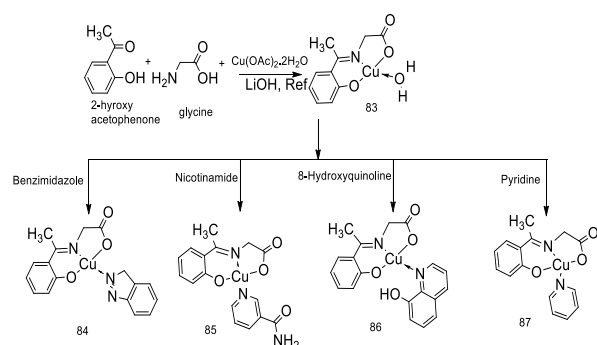
**Scheme 19. Synthesis of oxo-vanadium complex 82 via template synthesis**

The reported complex was characterized by different spectroscopic techniques. The studies show that blue colored vanadium complex exhibited monoclinic crystal system with  $P2_1$  space group. The complex also possessed antioxidant properties.

Similarly, Cu(II) complexes 83-87 of glycine Schiff base were synthesized by template synthesis by stirring an ethanolic solution of 2-hydroxyacetophenone with hot aqueous solution of LiOH and glycine for 2h at 50°C by [49]. A green coloured copper complex was formed after addition of  $\text{Cu}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ . An ethanolic

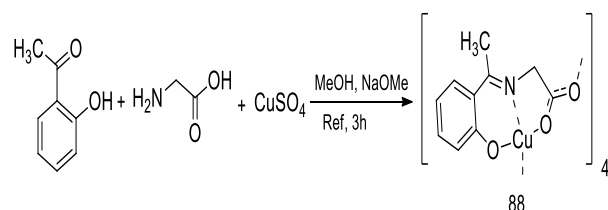


solution of copper complex was further heated to reflux with ethanolic solutions of benzimidazole, pyridine, 8-hydroxyquinoline and nicotinamide for 1 h to obtain Scheme 20. The reported complexes were investigated for their potential against some bacterial strains.



**Scheme 20. Synthesis of mixed ligand Schiff base Cu(II) complexes 83-87**

One more example of in situ synthesis of Cu(II) complex 88 from methanolic solution of 2-hydroxyacetophenone, glycine and copper sulphate in the presence of sodium methoxide was reported Scheme-21 [50].

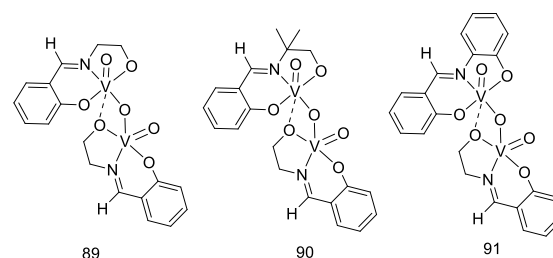


**Scheme 21. Synthesis of Cu(II) complex 88**

The single crystal X-ray studies of complex showed tetragonal system with  $P4_{2/n}$ . The coordination sphere was consisted of a cyclic tetrameric structure comprising four Cu(II) ions bonded to three ligand molecules. The synthesized complex was investigated for catalytic activity of cyclopropanation of styrene by ethyl diazoacetate to 2-phenyl-cyclopropanecarboxylate. In addition to catalytic properties, it was also tested for antioxidant activity.

In another approach Cordelle and coworkers reported oxo-based dinuclear(V) complexes 89-91 synthesized from Schiff base ligand containing ONO donor atoms through one pot process Figure 6 [51]. Various

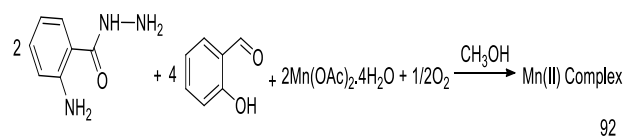
spectroscopic studies confirmed the formation of metal complexes.



**Figure 6. Structure of complexes 89, 90 and 91**

The complex 91 was found to be effective catalyst for the epoxidation of cyclooctene using aqueous TBHP oxidant in absence of organic solvents.

Recently a new black colored dinuclear Mn(II) complex 92 has been synthesized by one-pot condensation reaction salicylaldehyde and 2-aminobenzohydrazide with  $Mn(OAc)_2 \cdot 4H_2O$  in methanol Scheme 22 [52]. Various spectroscopic techniques confirmed the formation of metal complex. The single crystal X-ray analysis shows that dinuclear Mn(II) complex crystallized in  $P_1$  space group with triclinic system.



**Scheme 22. Synthesis of dinuclear Mn(II) complex 92**

Similarly a dark brown Co(II) complex 93 synthesized by template synthesis by reacting  $\beta$ -alanine, salicylaldehyde and cobalt acetate in the presence of potassium acetate in water-alcohol mixture [53]. The single crystal X-ray studies of complex indicated monoclinic crystal system for complex having  $C2/c$  space group. This complex contain two terminal Co(II) ions, coordinated by two tridentate ligands sal- $\beta$ -sal.

Another dark green colored Cu(II) complex 94 was obtained by one pot reaction of  $\beta$ -alanine, copper acetate monohydrate and salicylaldehyde by Vanco and coworkers [54]. The single crystal X-ray analysis of the complex revealed that the complex was consisted of two copper atoms; the first Cu(II) adopted a twisted



square-planar whereas other Cu(II) adopted square-pyramidal geometry and the existence of one H<sub>2</sub>O molecule at apical position with a distance of 2.241(2)Å. Further synthesized complex was tested for its antioxidant and anti-diabetic activities

### Conclusion

Schiff base along with their metal complexes have been widely explored due to their simple method of synthesis, versatile nature and stability. Lot of Efforts has been made by different research groups to explore new Schiff base along their metal complexes with modified properties. These find applications in various fields, including biological systems, catalysis, pharmaceutical and environment monitoring. In this review a brief literature survey of Schiff base ligand system containing ONO donors along with metal complexation methods, characterization and their applications have been discussed.

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