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**SYNTHESIS, CHARACTERIZATION OF SCHIFF BASE
COBALT (II) METAL COMPLEX**

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Abstract:

We have synthesized the Schiff base, 3-{(E)-[(2-hydroxy-4-methylphenyl) imino] methyl} quinoxalin-2(1H)-one, by condensation reaction. The Schiff base is an ONO donor and it can act as a monobasic or dibasic ligand due to the formation of tautomers. Based on the physicochemical and spectroscopic studies we propose octahedral structures for the complex

Key words: Schiff base, ONO donor, tautomers, physicochemical

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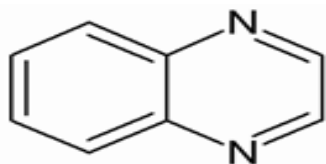
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INTRODUCTION:

Quinoxalines, also called benzopyrazines, are heterocyclic compounds containing a fused ring made up of a benzene ring and a pyrazine ring. They along with the isomers cinnolines, phthalazines and quinazolines belong to a class of heterocyclic compounds known as diazaphthalenes with two heteroatoms in the same or different rings. The fusion of a benzene ring, however, causes decrease in the aromaticity due to the bond alternation (Figure 1). Quinoxalines have 10- π electrons that are located in five bonding molecular orbitals. There are also two non-bonding orbitals that lie in the molecular plane and are confined to the nitrogen atoms. Each of these orbitals contains an electron pair and these electrons are responsible for the basic properties of quinoxalines [1-4]. Quinoxaline can act as Lewis base, form metal complexes, and can participate in hydrogen bonding with hydrogen atoms present in electronegative atom

**Fig 1: Quinoxalines**

Quinoxaline derivatives have been widely used in dyes, pharmaceuticals, and in electrical/photochemical materials. Quinoxaline ring moiety is a part of the chemical structures of various antibiotics such as echinomycin, levomycin and actinoleutin, which are known to inhibit growth of gram positive bacteria and are active against various transplantable tumors. A number of synthetic strategies have been developed for the preparation of substituted quinoxalines. Quinoxalines are readily made from 1,2-dicarbonyl compounds and aromatic 1,2-diamines. A well-known route to quinoxalines is the reaction of *o*-phenylenediamine with a 1,2-dicarbonyl compound [5-7].

During the past several years, quinoxalines have been used effectively as building blocks for metal-

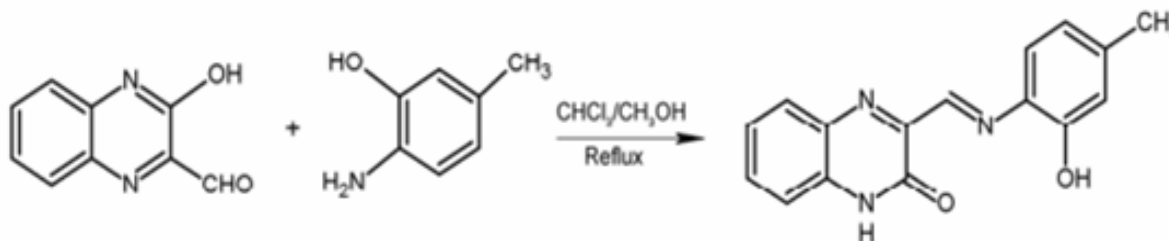
containing two dimensional networks [8]. Metal halides coordinate readily with quinoxalines to form interesting coordination polymers. Substituted quinoxalines, which have the potential to form novel three-dimensional structures upon coordination, have been synthesized. Quinoxaline heterocycles have attracted much attention owing to their natural occurrences [94] and biological activities [95]. Many quinoxaline derivatives display unusual solid-tumor selectivity against multi drug-resistant cancer cells. The synthetic utility and pharmacological importance of these compounds have prompted many scientists to synthesize and characterize novel quinoxaline derivatives [9].

Materials and Methods:

Cobalt (II) chloride hexahydrate (Merck), cobalt (II) acetate hexahydrate (Merck), 2-aminothiophenol, 2-amino-5-methylphenol were purchased from Sigma Aldrich Chemicals Private Limited, Delhi. Ortho phenylenediamine (Lobachemie), D- glucose (SD Fine Chem Limited), sodium sulphate (Merck), sodium metaperiodate (Merck), glacial acetic acid, hydrazine hydrate (Qualigens), sodium bicarbonate (Sisco Research Laboratories Limited), bromine (Merck) and sodium pyruvate (Sisco Research Laboratories Limited) were used in the present investigation. Uv spectra were recorded in methanol or DMSO- on a Bruker AVAVCE III spectrometer using

Synthesis of Schiff base:

To an aqueous solution of 3-hydroxyquinoxaline-2-carboxaldehyde (1.742 g, 10 mmol, in 500 mL distilled water), 3-4 drops of conc. HCl was added. To this solution was added drop wise 2-amino-5-methylphenol (1.231 g, 10 mmol) in methanol (20 mL). The solution was stirred for 6 hours. The red coloured Schiff base formed was filtered, washed with water and dried over anhydrous calcium chloride. The crude product was recrystallised from absolute ethanol. Formation of Schiff base is presented in scheme below (Yield: 75 %, MP: 200 °C)

**Fig 2: Formation of Schiff base**

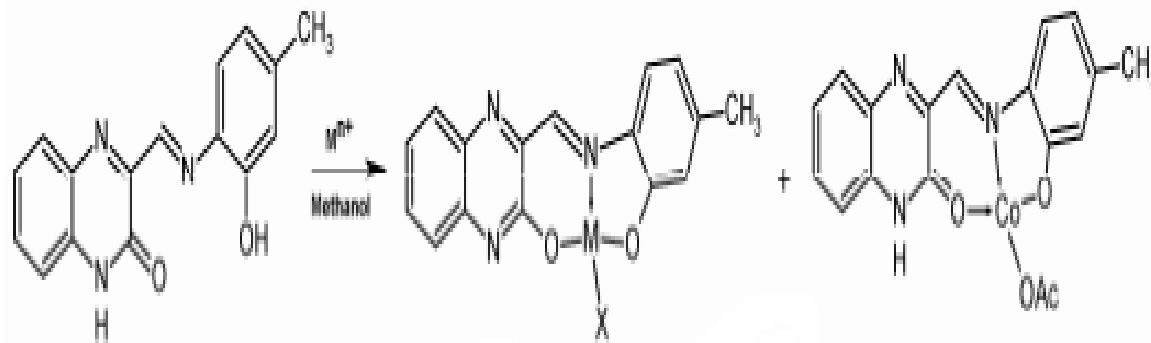


Fig 3: The formation of metal complexes

Synthesis of Schiff base metal complex

The Schiff base, hamp, (0.01 mol, 0.279 g) was dissolved in 50 mL. To this solution a solution of metal compound cobalt acetate hexahydrate 0.249 g, in methanol (30 mL) was added. The solution was then refluxed for an hour and was then kept at room temperature (28 ± 2 °C) after reducing its volume by evaporation. The precipitate formed was filtered, washed with methanol and kept in a desiccator. The formation of metal complexes is given in Scheme above.

RESULT AND DISCUSSION:

The complexes are stable in air and non-hygroscopic. They are soluble in common solvents like ethanol, methanol, acetonitrile and DMF. The analytical data (Table 5.1) reveal that all the complexes are formed in metal: ligand ratio of 1:1. Further the molecular formula of the complexes given in the table agrees well with the analytical data. The very low conductance values.

Electronic Spectra

The electronic spectral data of the Schiff base and complex are taken in methanol ($\sim 5 \times 10^{-4}$ molar) and are given in Figures a and b. The spectral bands due to solvent, which have been observed above 45000 cm^{-1} , are omitted [11]. The ligand shows absorptions at 30800 and 24000 cm^{-1} . These bands are attributed to benzene $\pi-\pi^*$, imino $\pi-\pi^*$ and quinoxaline $\pi-\pi^*$ transitions of the Schiff base. In complexes, the intense bands observed above 30000 cm^{-1} are due to charge transfer and intra ligand transitions. In the electronic spectrum of Co(II) complex the d-d transitions are almost masked by the high-intensity charge transfer bands. However, a shoulder is observed around 17000 cm^{-1} which may be assigned to the ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ transition and transitions in this region have been reported for octahedral Co (II) complexes [32]. Below is the figure showing the UV spectra of Schiff base and Cobalt Schiff base.

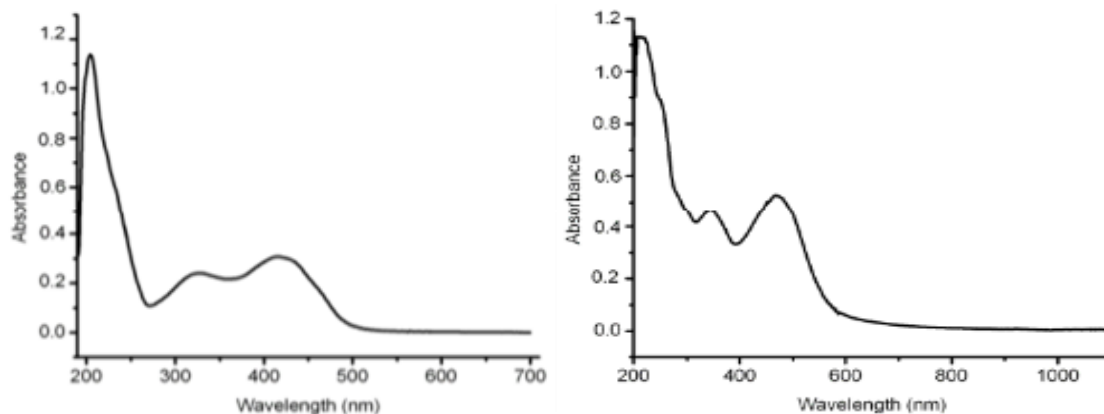
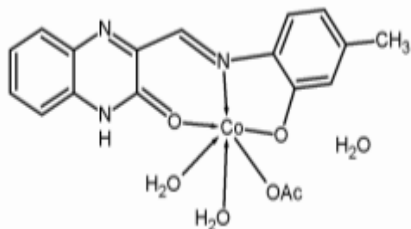


Fig 4: (a) UV of Schiff base ligand (b) UV-of Schiff base Cobalt metal complex

CONCLUSION:

A new Schiff base derived from 3-hydroxy quinoxaline-2-carboxaldehyde and 2-amino-5-methylphenol and its complex of Co (II,) have been synthesized and characterized. Based on the physicochemical and spectroscopic studies we propose the following structures for the complex.

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