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Synthesis, Characterization of some Schiff's bases derived from phenylhydrazine

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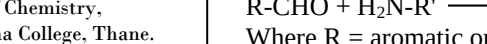
Abstract
A Schiff's Base is a neutral molecule with an electro pair and contains a carbon-nitrogen double bond. This class of compounds was discovered in 1864 by Hugo Schiff. The condensation reactions of an aromatic aldehyde and aromatic amines carried out efficiently in a solvent medium. The formation of Imines or Azomethine are carried out without catalyst using ethanol as solvent. In this method, yields are high, reactions are fast and products are isolated easily by filtration. The chemical structure of compounds were confirmed by IR Spectroscopy.

Keywords: salicaldehyde, phenylhydrazine, Ethanol, Schiff base, Complexation

1. Introduction
Compounds containing an azomethine group (-CH=N-) are known as SCHIFF'S BASE. This class of compounds was discovered in 1864 by Hugo Schiff. Schiff's bases are usually prepared by the condensation of carbonyl compound with a primary amine. Schiff's bases of aromatic aldehydes having an effective conjugated system are more stable and an aliphatic aldehydes are relatively unstable and readily polymerizable.



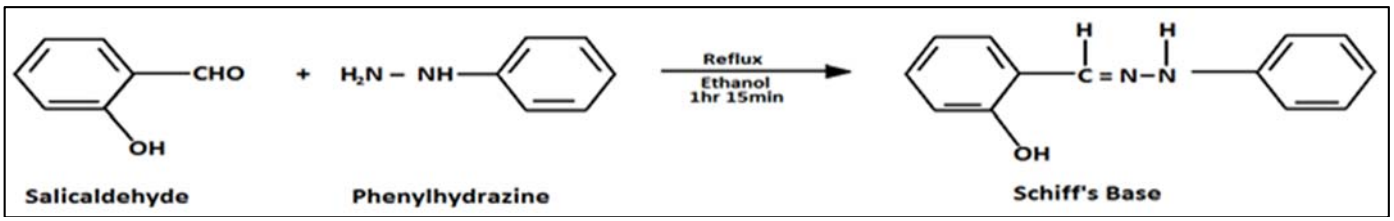
Where R = aromatic or aliphatic compounds.
Schiff's bases is a neutral molecule with an electron pair and contains a carbon-nitrogen double bond. The Schiff's bases are also called as IMINES, ANILIS and AZOMETHINE.
The Schiff base is derived from anilines and its derivative with aromatic aldehyde have wide varieties of application in biological and analytical chemistry.
Schiff's bases formation involves a two-step reaction between the carbonyl compound and the amino compound. First addition takes place to form carbinolamine which then undergoes dehydration. Both steps are reversible and subject to general acid-base catalyst.
Schiff's bases are known to be neoplasm inhibitors, antiviral, anticonvulsant, antimicrobial, anticancer, Plant growth regulator and antitubercular agents.
On the other hand, they are fundamental material for the synthesis of various Schiff's base ligands which used as chiral auxiliaries in asymmetric synthesis. Metal complex Schiff's bases have also been used in oxidation reaction.
The great deal of work has been reported on the complexation of metal ions with Schiff's bases for the study of structure and stability of the complexes. The catalytic effect of hydrogen, hydroxyl and metal ions on the formation and hydrolysis of imines have been studied, In the present work we reported here kinetic study and formation of Schiff's bases using phenylhydrazine and p-nitroaniline in ethanol medium.

2. Experimental
Synthesis of Schiff's Base Ligand
Step-I: Preparation of Schiff's base
Take salicaldehyde (0.9mmol) with phenylhydrazine (1.5mmol) in ethanol reflux the reaction mixture for one and an hours and in between take TLC, for the progress of the reaction. After completion of the reaction the hot solution was poured into ice-cold water. Then, the product was filtered under suction and washed with distilled water. The product obtained was yellow coloured solid, were dried at ambient temperature and recrystallised with ethanol.
Yield range 70-75%, Melting point-185 ºC.

Step-II: Synthesis of Co (II) Complexes
The Schiff's base complexes were synthesized by mixing the Schiff's base (1.5 g) in ethanolic solution of Cobalt nitrate [$Co(NO_3)_2$]. This reaction is refluxed in a water bath for two hours and their volume were reduced to 70% of it's original volume and residue was obtained. The coloured product obtained was filtered under suction, washed with ethanol. The product were recrystallized from ethanol. Their yields ranges from 50-55%, the product obtained were pinkish colour and melting point was 202 ^{0}C.

Step-III: Synthesis of Cr(II) Complexes
The Schiff's base complexes were synthesized by mixing the Schiff's base (1.5 g) in ethanolic solution of Chromium chloride [$CrCl_2$]. This reaction is refluxed in a water bath for two hours and their volume were reduced to 70% of it's original volume and residue was obtained. The coloured product obtained was filtered under suction, washed with ethanol. The product were recrystallized from ethanol. Their yields ranges from 50-55%, the product obtained were light green colour and melting point was 210 ^{0}C.

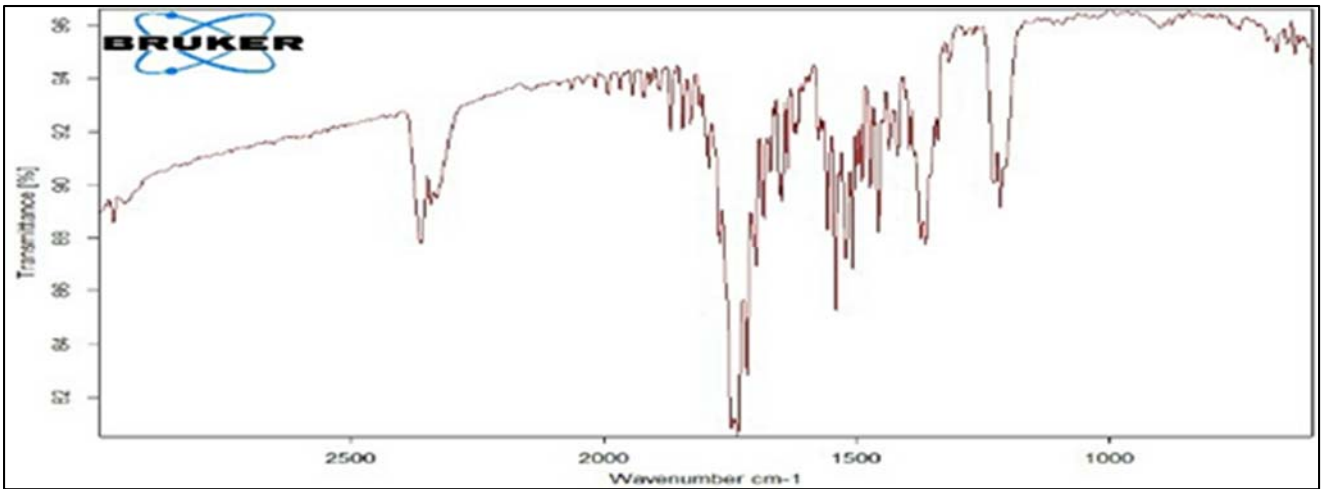
Step IV: Synthesis of Pb(II) Complexes
The Schiff's base complexes were synthesized by mixing the Schiff's base (1.5 g) in ethanolic solution of Lead nitrate [$Pb(NO_3)_2$]. This reaction is refluxed in a water bath for two hours and their volume were reduced to 70% of it's original volume and residue was obtained. The coloured product obtained was filtered under suction, washed with ethanol. The product were recrystallized from ethanol. Their yields ranges from 50-55%, the product obtained were white colour and melting point was 205 ^{0}C.



3. Results and Discussion
Physicochemical studies have been used in the elucidation of the metal complexes with the newly synthesized Schiff's base complexes.

Characterization of the Schiff's base Ligand
The newly synthesized ligands were characterized by IR-Spectroscopy. The data for the Schiff's base Ligand proposed the molecular formula.

Infrared spectra of the Schiff's base Ligand
The Infrared spectra of the synthesized Schiff's base ligand were scanned. The IR spectra of the Schiff's base ligands were recorded for the identification of their donor sites. A partial listing of the IR spectra of the Schiff's base ligand is given. Schiff's base ligands exhibit following assignments.
A strong absorption band at 1600-1700 cm^{-1} is due to C=N (imine) stretching vibration frequency.
Aromatic C-H band at 3000-2900 cm^{-1} is due to delocalization.
A band at 3500-3400 cm^{-1} is attributed to intramolecular hydrogen bonded -OH group.
A medium absorption band at 700-650 cm^{-1} is due to N-H (1^0, 2^0- amine) stretching vibration of NH group of the Schiff's base ligand.



4. Conclusion
From the experiment it was concluded that the methods could be used for production of Schiff's bases and has several advantages. It gives higher production and resulted into more economic production. The synthesized Schiff's base ligand were characterized on the basis of melting point, IR-Spectroscopy. Also the complexes of newly synthesized Schiff's base ligand with transition metal ions is form and characterized on the basis of IR-spectroscopic studies.

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