

Physicochemical Characterization of Metal Complexes with Schiff Bases Ligand

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Abstract: In the Present work first we synthesise Schiff base of some heterocyclic amine with aromatic amine, the chelating ability of Schiff base help to form metal complexes, and therefore Schiff base further react with metal ion to form metal complexes. The physical measurement and structural elucidation by spectrum like UV-Vis, FT-IR, ¹H-NMR, used in this work.

Keywords: Aromatic Amine, Aromatic Aldehyde, Sulphuric Acid, Ethanol, and Metals Halide.

I. INTRODUCTION

Both the Schiff base and metal complexes are important class of compound due to wide range of their Biological activity and its biological importance. The compound obtain by direct condensation of aldehyde with primary aromatic amine are known as Schiff bases (SBs), the Schiff bases with bioactive metal such as Copper (II), Cobalt (II), Zinc (II), Manganese(II) form most stable complexes.

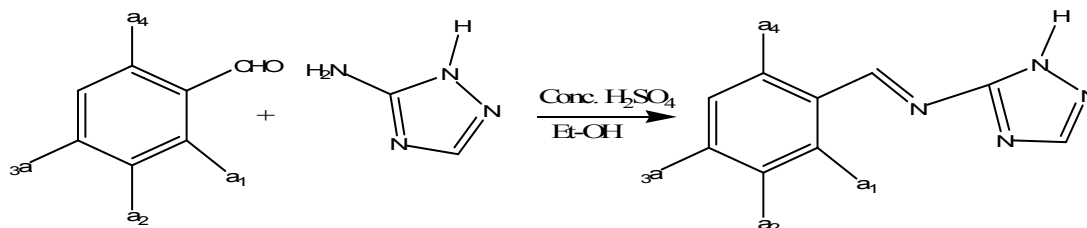
II. EXPERIMENTAL

2.1 Material and Methods

All solvents were laboured as commercial anhydrous mark without further Refining. The column chromatography was carried out over silica gel (100120esh). Melting points determined by open capillary tube. ¹H NMR spectra were recorded on a Bruker 300 MHz spectrometer in CDCl₃ solvent TMS as internal standard. The crude product was recrystallizing from 80 percentage ethanol.

Step I: General Procedure for the Synthesis of Schiff Base

A mixture of alcohol (20 ml) and aromatic aldehyde (0.02 mol) was taken into a 100 ml round bottom flask. The mixture was stirred until a homogeneous solution was obtained; aromatic amine (0.02 mol) was added with stirring. (As the reaction is exothermic it should be carried out by placing flask in a freezing mixture). Reaction mass is stirred for another 45 min. the Schiff base was precipitated out. The reaction mixture was cool with stirring. The isolated crude product is purified by the washing in acetone.

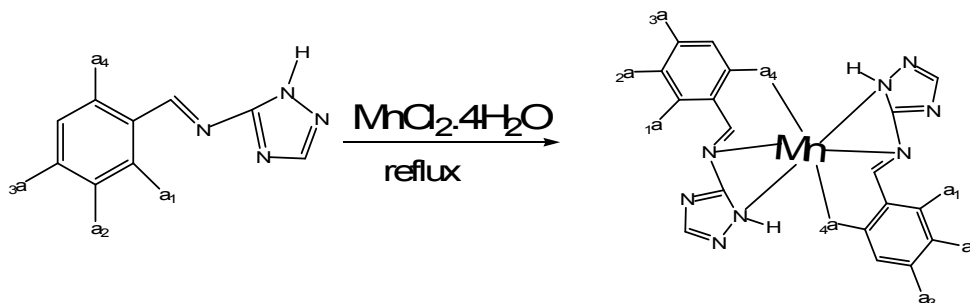


Scheme I

Compound also purify by silica gel column chromatography eluent ethyl acetate hexane reaction was. Monitored by TLC & spot were visualized in iodine.

Step II: General Procedure for the synthesis of Metal Complexes [MnCl₂ 4H₂O]

A mixture 0.1 mole of Schiff base in 30 ml alcohol and 0.5 mole of Manganese chloride was stir to form a homogeneous mixture then the reaction mixture was refluxed on a water bath for 6-8 hours. The precipitated complexes were filtered and dried.



Scheme II

1) **B7:-M.P.220 °C** (a₁=H, a₂=I, a₃=H, a₄= H)

FT-IR 760 cm⁻¹ for aromatic C-C stretching, 870 cm⁻¹ for C-I stretching, 1680 cm⁻¹ for C=N stretching, 1590 for C=C stretching, 1090 cm⁻¹ for C-N stretching, 2350 cm⁻¹ for CN stretching, 3170 for N-H stretching.

NMR : δ 7.2, s for Ar 8H, δ 8.0 m for Ar 2H, δ 8.5 for N 1H.

2) **B8 :-M.P.190 °C.** (a₁=CH₃, a₂=I, a₃=H, a₄= H)

FT-IR 550 cm⁻¹ for C-I stretching, 770 cm⁻¹ for aromatic C-C stretching, 1160 cm⁻¹ for C-N stretching, 1660 cm⁻¹ for Ar C=C stretching, 1720 cm⁻¹ for C=N stretching, 2960 cm⁻¹ Ar C-H stretching, 3090 for N-H stretching.

NMR: δ 2.6 s for 6H (Methyl group), δ 7.2 m for Ar 6H, δ 7.9 m for Ar 2H (Heterocyclic), δ 8.1 s for 2H (N-H).

3) **B9 M.P.140 °C.** (a₁=Cl, a₂=I, a₃=H, a₄= H)

FT-IR: 590 cm⁻¹ for C-I stretching, 770 cm⁻¹ for aromatic C-C stretching, 1090 cm⁻¹ for C-N stretching, 1650 cm⁻¹ for Ar C=C stretching, 1760 cm⁻¹ for C=N stretching, 3060 cm⁻¹ Ar C-H stretching, 3100 for N-H stretching.

NMR : δ 6.5 m for 6H, δ 7.3 s for 2H, δ 8.3 s for 2H(N-H).

4) **B10 M.P.198 °C.** (a₁=Br, a₂=I, a₃=H, a₄= H)

FT-IR 590 cm⁻¹ for C-I stretching, 770 cm⁻¹ for aromatic C-C stretching, 850 cm⁻¹ for aromatic C-Br stretching, 1310 cm⁻¹ for C-N stretching, 1610 cm⁻¹ for Ar C=C stretching, 1770 cm⁻¹ for C=C stretching, 3040 cm⁻¹ Ar C-H stretching, 3150 for N-H stretching.

NMR: δ 7.2 m for 6H, δ 7.7 s for 2H, δ 8.0 s for 2H(N-H).

5) **B11 M.P.210 °C.** (a₁=Br, a₂=I, a₃=H, a₄= CH₃)

FT-IR 610 cm⁻¹ for C-I stretching, 720 cm⁻¹ for aromatic C-C stretching, 1250 cm⁻¹ for C-N stretching, 1640 cm⁻¹ for Ar C=C stretching, 1690 cm⁻¹ for C=C stretching, 3120 cm⁻¹ Ar C-H stretching, 3150 for N-H stretching.

NMR: δ 2.9 s for 6H, δ 6.7 m for 6H, δ 7.7 s for 2H, δ 8.0 s for 2H(N-H).

Antibacterial properties of the synthesized Schiff base metal complex [Zone of inhibition (mm)]

IV. RESULTS AND DISCUSSION

All the six Schiff base metal complexes compounds 1B–4B containing nitrogen containing heterocyclic moiety were successfully synthesized in excellent yield and their structures are elucidated using elemental analysis, FTIR, & ¹HNMR spectroscopy. All the Synthesised Compound will be screened for their biological activity.

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