

# Synthesis and Characterization of Iron Complexes with Bidentate Ketoanils

Richa\*, Sanidhya Upadhyay\*\* and Rajkumar Upadhyay\*\*

\*Department of Physics, N.R.E.C. College, Khurja (UP), India

\*\*Department of Chemistry, N.R.E.C. College, Khurja (UP), India

(Corresponding author: Richa)

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**ABSTRACT:** Nine complexes of iron (III) chloride, bromide, iodide, nitrate, and acetate with two bidentate ligands, 3-hydroxyanil of 2-hydroxyphenylglyoxal and 2-pyridine anil of acetyl glyoxal, have been synthesized and characterized by their elemental analysis, magnetic measurements, and infrared, electronic and electronic spin resonance spectroscopy. Based on magneto-spectral studies all the complexes were assigned octahedral geometry.

**Key words:** Ketoanils, magnetic moment, electronic spin resonance, ligand field parameters

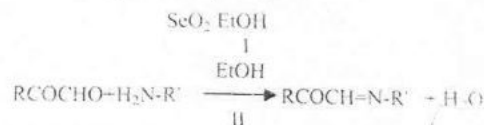
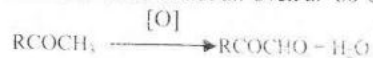
## I. INTRODUCTION

Schiff's bases obtained by condensation of aldehydes, ketones or glyoxals with primary amines, are preponderant over variety of other organics owing to their multifarious roles in diverse disciplines, viz. in chemistry as starting materials in the synthesis of heterocycles [1-4], as novel ligands in forming complexes of unusual stereochemistries [5-6] and isomeric structures [5,6] and as analytical reagents [8,9], in industries as dyes [10], in medical science as bactericide, fungicide, anticancer, antimalarial, antituberculosis, analgesic etc. agents [11-13] and in agriculture as herbicides [14].

The enhanced biological properties of metal-based organic compounds [14-16] observed generally and novel ligation properties of Schiff's bases aroused our interest to synthesize and characterize coordination complexes of chloride, bromide, iodide, nitrate and acetate salts of iron (III) with 3-hydroxy anil of 2-hydroxyphenylglyoxal (HAHPG) and 2-pyridine anil of acetyl glyoxal (PAAG) bidentate (ON) ligands by elemental analysis, magnetic measurements, and IR, ESR and electronic spectra and report in the present communication.

## II. EXPERIMENTAL

**Synthesis of ligands:** Both ligands, HAHPG and PAAG, were synthesized by the common method in two steps (Scheme 1). 2-Hydroxy phenyl glyoxal and acetyl glyoxal prepared by oxidation of 2-hydroxy acetophenone and diacetyl with selenium dioxide respectively in alcohol by reported methods [18,19] were mixed in equimolar quantities with 2-aminophenol and 2-aminopyridine respectively in alcohol and reaction mixtures were evaporated on water bath. The residue of HAHPG was washed with ice cold alcohol and benzene successively whereas residue of PAAG was washed with ether. Both the products were dried in hot air oven at 60°C.



Where, R = C<sub>6</sub>H<sub>4</sub>(OH)- or CH<sub>3</sub>CO- and R' = C<sub>6</sub>H<sub>4</sub>(OH)- or C<sub>4</sub>H<sub>3</sub>N

**Synthesis of complexes:** All the complexes of HAHPG with Fe (III) salts were prepared by mixing warm saturated solution of ligand with saturated solution of each of the metal salts in stoichiometric quantities in acetone or acetone-H<sub>2</sub>O-HCl (9:1.5: 0.5 v/v/v) and the reaction mixtures were evaporated to dryness on water bath after refluxing for 1h. Residues were washed with water and filtered. Water insoluble residues of bromo, iodo, and nitro complexes were finally washed with alcohol and alcohol-carbon tetrachloride (5:2, v/v) successively and dried in oven at 60°C. Filtrates containing chloro and acetato complexes were evaporated to dryness on water bath and residues were washed with acetone-benzene-methanol (4:3:4, v/v/v) and ether solvents successively as identified by TLC to recover the products; the Fe(III)-PAAG complex precipitated while mixing the reactants in acetone was washed with acetone and dried in oven. All the complexes are non-hygroscopic and fairly stable in air. All chemicals used in the synthetic work were BDH E. Mark laboratory reagents whereas in TLC work BDH solvents were used after their distillation.

**Analysis and physical measurements:** Carbon, hydrogen and nitrogen contents of the samples were estimated microanalytically at RSIC, Punjab University, Chandigarh. Infrared spectra were recorded in 4000-400cm<sup>-1</sup> range on Bruker IFS-66 VFT-IR spectrometer in KBR discs. Magnetic susceptibility measurements were performed on the vibrating sample magnetometer model-155 at RSIC, I.I.T Madras in magnetic field 6 KGauss at room temperature. Electron spin resonance spectra were recorded on EPR, E-4 spectrometer operating at 9.410 GHz, 9.475 GHz, frequency at room temperature at R.S.I.C. I.I.T Madras; value of Lande's splitting