

## SYNTHESIS, STABILITY AND TGA STUDY OF SCHIFF-BASE LIGAND AND CORRESPONDING METAL COMPLEXES.

Assist. Prof. Premlata M. Sonparote

Department of chemistry, B. B. Arts, N. B. Commerce and B. P. Science College, Digras

Dist-Yavatmal, Maharashtra 445203

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### ABSTRACT

In the present study the Schiff- base chelates of 2,5dihydroxyphenone and 2,4dihydroxy phenone with p-phenyl diamine were prepared by the literature method. The complex with the metal Mn(III) and Al (III) were also prepared. The TGA study was done to study the stability of the resultant complexes.

**Keywords**—Schiff-Base, TGA, complexes, chelate's, metals.

### INTRODUCTION

The importance of Schiff- base complexes is increasing day to day. Hence it is essential to study the formation of such complexes with different metal ions. It is noted that Schiff-Bases are selective and sensitive towards central metal atom. The Schiff –Base ligand 2,5dihydroxy-acetophenone and 2,4dihydroxyacetophenone with some diamines have been reported to form complexes with transition metal ions. It is important to study the thermal stability of such complexes. Hence in the present work TGA study was undertaken.

### METHODOLOGY

All the chemicals and solvents used were of A R grade. 2,5 dihydroxy acetophenone and 2,4 dihydroxy acetophenone were prepared according to literature methods. p-Phenylene diamine was used. Elemental analysis were performed at R. S. I. C, C. D. R. I, Lucknow. Metal contents were estimated using standard method. I.R. spectra (KBr) was recorded on a Perkin Elmer spectro photometer. <sup>1</sup>HNMR spectra (CDCl<sub>3</sub>) at R.S.I.C., Chandigarh and diffuse reflectance spectra at I.I.T, Chennai. Magnetic measurements were carried out by the GOWY method at room temperature. The mannetometer was calibrated using Hg[Co(NcS)<sub>4</sub>]. Diamagnetic corrections were made by using Pascal' constants. Thermal studies were done with a Perkin – Elmer TGS-2 thermo balance in air with a heating rate 10<sup>0</sup> min<sup>-1</sup>. The d. c. electrical conductivity was measured by voltage drop technique using a micro voltammeter. The synthesis and characterization of ligands H<sub>2</sub>L<sup>4</sup> have been reported earlier.

### SYNTHESIS OF COMPLEXES

To a boiling solution of the ligand (0.002 mol) in ethanol (40ml), appropriate metal salt ( $\text{MnCoAC} \cdot 3 \cdot 2\text{H}_2\text{O}$  and  $\text{AlSO}_4 \cdot 0.002$  mol solution) was added. The reaction mixture was refluxed on a water bath for 2-3 hours. The resulting solid was washed with hot water, ethanol and methanol. The metal complexes were dried under reduced pressure over anhydrous  $\text{CaCl}_2$  in a desiccators.

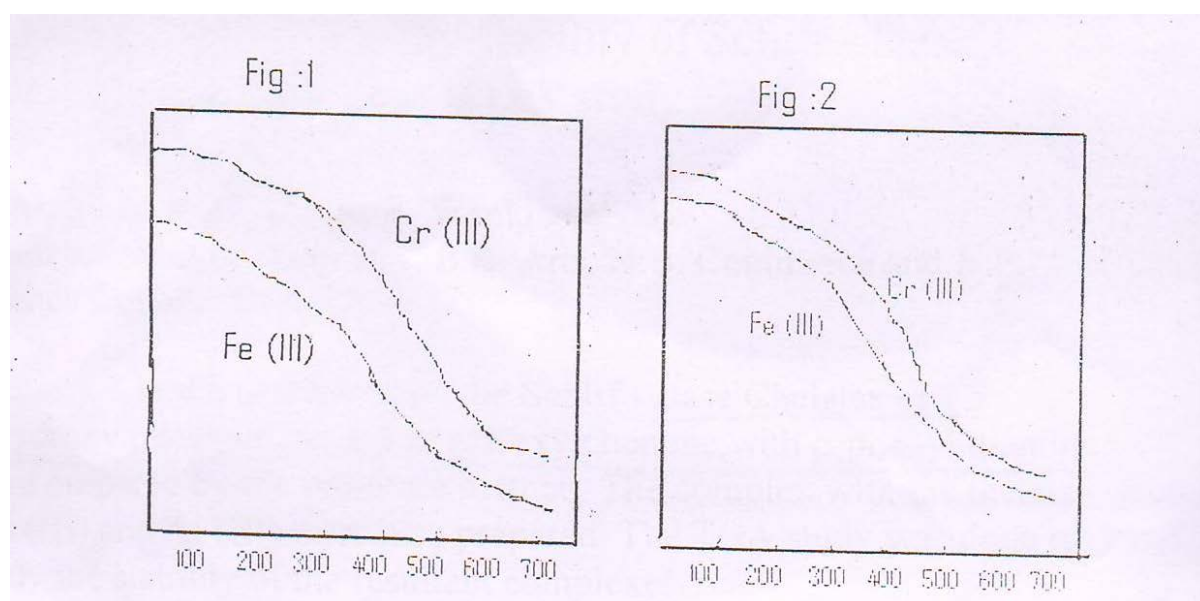
## RESULT AND DISCUSSION

Thermo gravimetric analysis studies of the complexes formed was done to verify the presence of co-ordinated water molecule or water crystallisation and relative thermal stability of the complexes of Mn(III) and Al(III). The formed TGA curves are given in the fig 1 and 2.

TGA curves of Al(III) complex showed three steps of decomposition. The TGA curves shows weight loss 7.60 % in the temperature range of  $140-185^\circ\text{C}$ , which is equivalent to theoretical value of two water molecules (7.80%). This result indicates that the water of co-ordination is present in these complexes. At  $650^\circ\text{C}$ , the stable metallic oxide is formed in each complex (fig 1).

The thermo grams of Mn(III) show two steps of decomposition. The first step begins at  $T > 150^\circ$ . This suggests the absence of any water. No attempts were made to characterize the products formed at the end of first stage. At the end of second step i. e. at  $650^\circ$  stable manganese oxide was formed. The complexes are found to be thermally more stable than the corresponding Schiff base ligand (fig 2)

Chelates of Fe(III) shows three or four step decomposition. The first decomposition step shows the weight loss in the range of  $90-190^\circ\text{C}$  equal to 7.25, 6.83, 8.62 and 7.16% respectively. These values formed<sup>1</sup>.



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