

SYNOPSIS OF THE DISSERTATION ENTITLED
"ANALYTICAL APPLICATIONS OF SOME THIOLIGANDS"
SUBMITTED BY MISS HONMUTE, S. N. FOR THE M.Phil.
DEGREE IN CHEMISTRY TO SHIVAJI UNIVERSITY, KOLHAPUR-4.

The present investigation embodies the results of intensive studies on the use of 3,5-dichlorosalicylaldehyde thiosemicarbazone (3,5-dichloro SAT) as spectrophotometric reagent for Molybdenum(VI) and Iron(III). It also includes the use of 1-(4'-bromophenyl)-4,4,6-trimethyl(1H,4H)-2-pyrimidinethiol (4'-bromo-PTPT) as analytical reagent in the extraction separation and determination of Copper(II). The dissertation consist of four chapters.

Chapter I -

The chapter first gives an account of synthesis and characterisation of -

- 1) 3,5-dichlorosalicylaldehyde thiosemicarbazone and
- 2) 1-(4'-bromophenyl)-4,4,6-trimethyl(1H,4H)-2-pyrimidinethiol.

The broad review of thiosemicarbazones and mercaptopyrimidines reported in the literature is also given. These reagents form stable complexes with metal ions and possess properties desirable for spectrophotometry. The purity of the reagents was checked from their elemental analysis and melting point.

Chapter II -

In this chapter photometric procedure for determination of Mo(VI) using 3,5-dichloro SAT is presented. The Mo(VI) reacts with 3,5-dichloro SAT at 3.0N H₂SO₄ to form pink colour complex. The absorbance of pink colour complex of Mo(VI) is measured at 525nm against water as a blank. The system obeys Beer-Lambert's law upto 12ppm Mo at 525nm. The molar extinction coefficient of the complex is 6432 L mole⁻¹ cm⁻¹ with Sandell sensitivity 15ng/cm². The stability constant K of the complex is also calculated. The value of K is 6.945 x 10⁻¹⁶. The interference study is also reported. The method is simple, sensitive and reproducible.

Chapter III -

The study of photometric determination of Iron(III) using 3,5-dichloro SAT is described in this chapter. Iron(III) reacts with 3,5-dichloro SAT at pH 10 to form green complex. The complex is measured at 590nm against water as a blank. The system obeys Beer-Lambert's law upto 20.0ppm Fe at 590nm. The molar extinction coefficient of the complex is 2680 L mole⁻¹ cm⁻¹ with Sandell sensitivity and stability constant (K) of the complex 21ng/cm² and 3.0869 x 10⁻¹⁶ respectively. The effect of foreign ions on the complex is also studied. The method is simple, sensitive and reproducible.

Chapter IV -

This chapter is devoted to describe the use of 4'-bromo-PTPT in the extractive separation and determination of Copper(II) in presence and absence of pyridine. The yellow complex of Cu(II) with 4'-bromo-PTPT is formed at pH 5 in presence of pyridine. The complex is extracted with chloroform and is measured at 410nm against the solvent blank. The optimum conditions for full complexation and complete extraction of Cu(II) is established in presence and absence of pyridine. The system obeys Beer-Lambert's law upto 7ppm Cu(II) at 410nm. The molar extinction coefficient of the complex is $5084 \text{ L mole}^{-1} \text{ cm}^{-1}$ with Sandell sensitivity 12.5 ng/cm^2

The effect of all parameters and interference is made. The recovery of Cu(II) is quantitative and the results are reproducible.

Every chapter from II-IV opens up with a brief but upto date literature survey of reagents in tabular form reported for photometric and extractive determination of the respective elements. An attempt has been made to study the methods described critically with regard to sensitivity, accuracy and precision. The proposed ligands appear very versatile and promising as an extractant and photometric reagents.