

SYNOPSIS OF THE DISSERTATION ENTITLED "ANALYTICAL APPLICATIONS OF PYRIMIDINETHIOLS" SUBMITTED BY SHRI A.S. MOTAGI, FOR THE M.Phil. DEGREE IN CHEMISTRY TO SHIVAJI UNIVERSITY, KOLHAPUR.

The present investigation incorporates studies on the use of 1-(2',3'-dichlorophenyl)-4,4,6-trimethyl (1H, 4H)-2-Pyrimidinethiol (2',3'- dichloroPTPT) as analytical reagent in the extraction separation and determination of Tellurium(IV), Ruthenium(III) and Palladium(II). The dissertation consists of five chapters.

CHAPTER - I

In this chapter the theory of spectrophotometry is given covering the laws of absorption, limitation to applicability of Beer's law.

CHAPTER - II

The chapter second gives an account of synthesis and characterization of 1-(2',3'-dichlorophenyl)-4,4,6-trimethyl (1H, 4H)-2-Pyrimidinethiol.

The broad review of mercaptopyrimines reported in the literature is also given. The reagent was prepared by the method of mathes. The purity of the reagent was checked from their elemental analysis, melting point and spectral characteristics. Further the purity of the

pyrimidinethiols was confirmed by titrimetric method of analysis using standard solution of sodium methoxide (0.05 M) in benzene-methanol solution and Azo-violet as an indicator. It was found that the reagent is of 99.6 % pure.

CHAPTER - III

The chapter describes a method for the solvent extraction and spectrophotometric determination of microgram levels of tellurium(IV) from hydrochloric acid media with 1-(2',3'- dichlorophenyl) -4,4,6-trimethyl-(1H, 4H) pyrimidine-2-thiol (2',3'-dichloroPTPT) in chloroform. The yellow coloured complex absorbs at 430nm and the system obeys Beer's law in the concentration range of 2.5 to 12.5 ppm. The molar absorptivity and Sandell's sensitivity are $7562 \text{ L mol}^{-1} \text{ cm}^{-1}$ and 17 ng cm^{-2} respectively. The probable composition of extracted species is TeL_2 . The interference of various ions was studied and tellurium(IV) in various synthetic samples was determined.

CHAPTER - IV

The study of photometric determination of Ruthenium(III) using 2',3'-dichloroPTPT is described in

this chapter. Ruthenium(III) reacts with 2',3'-dichloroPTPT in dioxan solution (10 min heating on boiling water bath) at 8M HCl to form blue coloured complex extractable into chloroform. The complex is measured at 620nm against chloroform blank. The system obeys Beer's law in the range 4.8 to 17 ppm Ru(III) at 620nm. The molar extinction coefficient of the complex is $4211 \text{ L mol}^{-1} \text{ cm}^{-1}$ with Sandell's sensitivity 24 ng cm^{-2} . The interference study shows that Ru(III) can be determined without interference of many ions and fairly large amount of common anions except Te(IV), Se(IV), Zr(IV) and Ti(IV), Thiourea, $\text{S}_2\text{O}_3^{2-}$ and I^- .

The method is simple, selective and also reproducible.

CHAPTER - V

This chapter is devoted to describe the method for extraction and spectrophotometric determination of Palladium(II) using 2',3'-dichloroPTPT. Palladium(II) (2.5 - 25 ppm) can be extracted into 10ml of 0.01M reagent in chloroform in the acidity range 0.5 - 9M HCl, 0.5 - 9 M H_2SO_4 and 0.5 - 9 M HClO_4 within 15 min shaking. The absorption spectra of the complex extracted into chloroform show a maximum at 420nm against chloroform blank. The molar extinction coefficient is

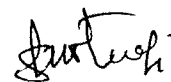
3405 L mol⁻¹ cm⁻¹ and the Sandell's sensitivity is 31ng cm⁻². The interference due to various cations and anions has been studied. The composition of the extracted species is determined by Job's continuous variation method and mole-ratio method and is found to ^{be} 1:2 (metal to ligand). The method has been employed to determine palladium in various alloys and the results are comparable with that obtained by other standard methods. This method is simple, sensitive and reproducible.

Every chapter from III to V opens up with brief but upto date literature survey of reagents reported for extractive photometric determination of the respective elements. An attempt has been made to study the method described critically with regard to sensitivity, accuracy and precision. The proposed ligand appears versatile and promising as an extractant and spectrophotometric reagent for Te(IV), Ru(III) and Pd(II).



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