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SYNOPSIS OF THE DISSERTATION ENTITLED "ANALYTICAL APPLICATIONS OF 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-2-pyrimidinethiol" SUBMITTED BY SHRI LOKHANDE, T. 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 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-2-pyrimidinethiol (4'-bromo-PTPT) as analytical reagent in the extraction separation and determination of Palladium(II), Ruthenium(III) and Osmium(VIII). The dissertation consists of four chapters.

Chapter I :-

This chapter gives an account of the synthesis and characterisation of 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-2-pyrimidinethiol. The broad review of mercaptopyrimidines reported in the literature is also given. The mercaptopyrimidines also known as cyclicthioureas react with platinum metals to form sufficiently stable and coloured complexes which are easily extractable. Hence, these are suitable for use in spectrophotometric measurements. The purity of the reagent was checked from the elemental analysis, melting point, TLC and also by non aqueous titrimetric determination of thiol group of 4'-bromo-PTPT.

Chapter II :-

In this chapter extractive photometric procedure for determination of Pd(II) using 4'-bromo-PTPT is presented.

The Pd(II) in the aqueous medium, 2-3 M in HCl is extractable with 10ml of 0.01 M reagent in chloroform within 10 min shaking. The yellow complex of Pd(II) with 4'-bromo-PTPT is measured at 420 nm against the solvent blank. The system obeys Beer-Lambert's law in the range 7-24 ppm Pd at 420 nm. The molar extinction coefficient of the complex is $2930 \text{ L mol}^{-1} \text{ cm}^{-1}$ with Sandell sensitivity 36 ng/cm^2 . The interference study is also reported. This method is simple, sensitive and reproducible and permits the determination of palladium in palladised carbon catalysts.

Chapter III :-

The study of photometric determination of ruthenium (III) using 4'-bromo-PTPT is described in this chapter. Ruthenium (III) reacts with 4'-bromo-PTPT in hot alcoholic solution (16 min heating on water-bath) at 6.0 M HCl to form blue complex extractable with chloroform. The complex is measured at 620 nm against the solvent blank. The system obeys Beer-Lambert's law in the range 5-15 ppm Ru at 620 nm. The molar extinction coefficient of the complex is $5660 \text{ L mol}^{-1} \text{ cm}^{-1}$ with Sandell sensitivity 18 nm/cm^2 . The interference study shows that Ru(III) can be determined without interference of many ions and fairly large amount of common anions. The method is simple, sensitive and also reproducible.

Chapter IV :-

This chapter is devoted to describe the use of 4'-bromo-PTPT in the extractive separation and determination of

Os (VIII) from aqueous-perchloric acid media. The pink complex of Os(VIII) with 4'-bromo-PTPT is formed when an aqueous solution of Os(VIII) which is 2-3 M in HClO_4 is heated for 3 min on water-bath. The complex is extracted with chloroform and is measured at 520 nm against the solvent blank. The optimal conditions for full complexation and complete extraction of Os(VIII) is established by varying parameters such as acidity of aqueous phase, reagent concentration, heating time, stability of the complex etc. The system obeys Beer-Lambert's law in the range 7-30 ppm Os at 520 nm. The molar extinction coefficient of the complex is $5325 \text{ L mole}^{-1} \text{ cm}^{-1}$ with Sandell sensitivity 36 ng/cm^2 . The 1:1 complex with respect to metal to reagent is established by Job's continuous variation and Mole ratio methods. The effect of all parameters and interference is made.

Every chapter from 2-4 opens up with a 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 methods described critically with regard to sensitivity, accuracy and precision. The proposed ligand appears versatile and promising as an extractant and photometric reagent for Pd(II), Ru(III) and Os(VIII).