SYNOPSIS

ANALYTICAL APPLICATIONS OF SOME THIOLIGANDS: EXTRACTION, SEPARATION AND SPECTROPHOTOMETRIC DETERMINATION OF SOME METALS.

The present investigation embodies the results of intensive studies on the use of 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-pyrimidine-2-thiol (4'-bromoPTPT) as analytical reagent in the extraction, separation and spectrophotometric determination of Selenium(IV), Tellurium(IV), Bismuth(III), Antimony(III), Thallium(I) and Rhenium(VII). In the thesis, there are seven different chapters.

CHAPTER - 1: This chapter gives an account of synthesis and characterisation of 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-pyrimidine-2-thiol. The purity of reagent was checked by titrimetric method, elemental analysis, melting point and spectral characteristics.

CHAPTER - 2: The operating conditions for the spectrophotometric determination of selenium(IV) with 4'-bromoPTPT as a ligand by liquid-liquid extraction techniques are presented. In acidic conditions selenium(IV) forms a yellow complex with the ligand which can be extracted in chloroform with an absorption maxima at 350 nm and system obeys Beer's law in the concentration range 5-70 ppm. The interference of various ions was studied and selenium(IV) was determined in synthetic mixtures, alloys and pharmaceutical samples.
CHAPTER - 3: The chapter describes a method for the extraction, separation and spectrophotometric determination of trace levels of tellurium(IV) from HCl media with 4'-bromoPTPT in chloroform. The complex is measured at 440 nm. Various parameters such as effect of acidity, reagent concentration, equilibration time etc, have been established. The method has been applied for the determination of tellurium (IV) in various synthetic samples, corresponding to alloys and also provides binary separation. It is also applicable for the determination of selenium(IV) and tellurium(IV) simultaneously.

CHAPTER - 4: The study of spectrophotometric determination of bismuth(III) with 1-(4'-bromophenyl)-4,4,6-trimethyl (1H,4H)-pyrimidine-2-thiol is described in this chapter. Bismuth(III) reacts with 4'-bromoPTPT in HCl medium to form yellow complex measured at 410 nm. The molar absorptivity is $15 \times 10^4$ l mol$^{-1}$ cm$^{-1}$ and Sandell's sensitivity is 14.3 ng cm$^{-2}$. The difference in the absorbance between the chloroform blank and bismuth(III) sample increases linearly in the concentration range 2-14 ppm. The method is extremely sensitive, rapid and reproducible.

CHAPTER - 5: This chapter is devoted to describe the method for the solvent extraction separation and spectrophotometric determination of microgram levels of antimony(III) from HBr media with 4'-bromoPTPT in chloroform. The yellow coloured complex absorbs maximally at 360 nm and system obeys Beer's range 5-35 ppm. The molar absorptivity and Sandell sensitivity are 3166 l mol$^{-1}$ cm$^{-1}$ and 38 ng cm$^{-2}$ respectively. Interference of various ions was studied. The method has been applied for
the determination of antimony(III) in various synthetic mixtures, real samples. It provides for binary separation of Sb(III) from Pb(II), Pd(II), Cu(II), Se(IV), Te(IV) and Bi(III).

CHAPTER - 6: It deals with extraction spectrophotometric determination of thallium(I) with 4'-bromoPTPT. Studies reveal that thallium(I) can be extracted quantitatively from 2.3 to 2.8 M HBr media. Common anions and cations do not interfere in determination of thallium(I). Thallium(I) is extracted from synthetic mixtures with composition corresponding to alloy. Various parameter like effect of acidity, equilibration time, reagent concentration was studied.

CHAPTER - 7: It includes the study of solvent extraction and spectrophotometric determination of rhenium(VII) using 4'-bromoPTPT as a chromogenic reagent which forms a yellow coloured complex at 0.7 - 1.0 M HCl extractable into isobutyl methyl ketone (MIBK). The complex absorbs at 395 nm and obeys Beer's law in the concentration range of 2.5 - 35.0 ppm. The probable composition of the extractable species is ReL₂. The method is applicable for the determination of rhenium(VII) in synthetic mixtures and alloys.

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