SUMMARY OF WORK DONE

The dissertation entitled " EXTRACTIVE SEPARATION STUDIES OF LEAD FROM ASSOCIATED ELEMENTS WITH DIBENZO-18-CROWN-6 " deals the research work carried out under the guidance of Dr.B.S.Mohite A brief account of the main features of the research work is given below.

The dissertation is divided in to two chapters, Chapter-1 and Chapter-2. Chapter-1 deals with Introduction to Chromatography and Crown Ethers And its Use in Chromatography, while Chapter-2 deals with Extraction Chromatographic Separation Studies of Lead Using Dibenzo-18-Crown-6 From Picric Acid Medium.

CHAPTER-1

INTRODUCTION TO CHROMATOGRAPHY AND CROWN ETHERS AND ITS USE IN CHROMATOGRAPHY

This chapter includes historical perspective of chromatography, classification of chromatographic methods, theory of extraction chromatography , classification of extraction systems, separation techniques, optimisation of chromatographic process crown ethers, synthesis and properties of crown ethers and its use in extraction chromatographic methods. This chapter also includes the summary of column chromatographic separation studies of various elements with crown ethers.

CHAPTER-2

EXTRACTION CHROMATOGRAPHIC SEPARATION STUDIES OF LEAD USING DIBENZO-18-CROWN-6 FROM PICRIC ACID MEDIUM :

In this chapter the experimental details are covered. For the experimental work 60-120 mesh silica gel was used which was made hydrophobic by silanization with dimethyl-dichloro-silane. After silanization, silica gel was coated with dibenzo-18-crown-6 (6 %) using rotary vacuum evaporator. About 2.0 gm of dibenzo-18-crown-6 coated silanised silica gel was slurried with distilled deionised water and was poured in to Pyrex glass chromatographic column to get a bed height of about 10 cm. After preconditioning the column with picric acid it was use for the separation studies.

Extraction chromatographic separation studies of lead were carried out on dibenzo-18-crown-6 column from picric acid medium. The concentration of picric acid was varied from 0.01-0.05 M. There was 20 % extraction of lead with 0.01 M picric acid. With increase in the concentration of picric acid, there was increase in the % extraction of lead. There was 76 % extraction of lead with 0.025 M picric acid whereas lead was extracted quantitatively from 0.04-0.05 M picric acid. Further extraction studies of lead were carried out from 0.045 M picric acid.

After extraction, lead was eluted with different eluting agents which included nitric acid , hydrochloric acid, hydrobromic acid and perchloric acid. The concentration of eluent was

varied from 0.01-4.0 M . It was found that all the eluting agents were found to be the best eluents for the elution of lead. The total volume of the eluent'Vt' required for the recovery of lead and ' Vmax' was also found out.

To ascertain the amount of lead that could be extracted with 2.0 gm of dibenzo-18-crown-6 coated silanised silica gel, various experiments were carried out from 0.045 M picric acid by varying the concentration of lead. It was found that there was quantitative extraction of lead up to 9500 ug of lead/10 ml of load solution.

Lead was separated from number of elements in binary mixtures. It was found that most of the alkali metals were tolerated in higher proportions. Along with lead, potassium, rubidium, and cesium were coextracted, which were eluted by washing the column with water. Amongst alkaline earth metals strontium and barium were coextracted along with lead. The separation of lead from barium was also achieved. Most of the alkaline earth metals also showed high tolerance limits. Most of the d-block and p-block elements also the anions of inorganic and organic acids showed very high tolerance limit.

By exploiting the extraction and elution behavior of lead and other elements , clean cut separation of lead from other elements has been achieved. Lead was separated from number of elements in multicomponent mixtures. The mixtures included the following

a) X , Pb , Ba. b) X , Rb , Pb. & c) X , K , Pb .

X = U(VI) / Th(IV) / Ge(IV) / La(III) / Ce(III).

The method was extended for the determination of lead in number of environmental samples and alloy sample. The environmental samples included grass , bark of trees and soil samples. The alloy sample which was used for the lead content was Tin base white metal alloy (BCS-CRM No.178/2).

The most important feature of this method is that, the method is very simple , rapid , selective and reproducible. The recovery of lead in all instances from triplicate determinations is 100 % +/- 2 % and the reproducibility of the method is +/- 2 % .

Dr.B.S.Mohite
Research Guide

Miss. V.S.Kapali
Research Student.