SYNOPSIS

n-OCTYLANILINE AS AN EXTRACTANT FOR SOME ELEMENTS

Solvent extraction also called as liquid-liquid extraction is one of the most versatile and popular method of separation used in analytical chemistry. The main reason for its versatility and popularity lies in the fact that it is simple, rapid and is applicable at tracer and macrogram concentrations of metal ions.

In recent years solvent extraction by high molecular weight organic amines has occupied important position in the study of metal complexes. High molecular weight amines or so called liquid anion exchangers combine many of the advantages of both solvent extraction and ion exchange phenomena. They extract selectively the anionic metal complexes which are formed reversibly in the aqueous phase, hence the name “liquid anion exchangers”. Best separations are possible with good diluents with negligible solubility in aqueous phase. The extent of extraction depends upon the structure and branching of the amine and the nature of the diluent. It is found that primary and secondary amines are good for the extraction of anionic complexes with the organic carboxylic acid. In regard of diluent used, the solubility of primary long chain amines in non-polar solvents increases with increasing chain length. Hence, n-Octylaniline is a primary amine has high solubility in organic solvents while negligible solubility in water. The extraction equilibria for ion pair formation can be represented as,

\[ [\text{RNH}_2]_{\text{org}} + \text{HX}_{\text{aq.}} \Leftrightarrow [\text{RNH}_3^+ \cdot \text{X}^-]_{\text{org}} \]  ... (1)

\[ [\text{RNH}_3^+ \cdot \text{X}^-]_{\text{org}} + \text{Y}^-_{\text{aq.}} \Leftrightarrow [\text{RNH}_3^+ \cdot \text{Y}^-]_{\text{org}} + \text{X}^-_{\text{aq.}} \]  ... (2)

Where \( X^- \) = Anion of simple complex metal acid.
\( Y^- \) = Anion of metal complex.
\( R = -\text{C}_6\text{H}_5 \)
The amine salt formed in equation (1) can undergo anion exchange with an anion \( Y^- \) in the aqueous phase.

In the thesis, there are seven chapters. At the beginning a brief introduction of liquid-liquid extraction covering the theoretical aspects with special reference to principle and classification of extraction system is given. Chapter one gives a concise account of basic extractants and the actual experimental results obtained during different phases of investigation are included in chapter 2 to 7. An up to date literature survey of the reagents reported for solvent extraction of respective metals is given at the beginning of each chapter. The details about these are given below:

Chapter 1, describes the solvent extraction with basic extractants and high molecular weight amines used for liquid liquid extraction of metals from aqueous solutions. The method of synthesis, characterization of n-Octylaniline and general features for selective extraction of metal is enumerated in this chapter.

Chapter 2, deals with extraction separation of aluminium(III) from associated elements with n-Octylaniline from sodium succinate media. Selective and quantitative extraction of aluminium(III) by 2% n-Octylaniline into toluene takes place from 0.04M sodium succinate medium. The effect of concentration of succinate, pH, n-Octylaniline, role of various diluents, stripping agents and foreign ions on the extraction of aluminium(III) has been studied. The procedure offers distinct improvements in the analysis of dolomite, magnalum, bauxite and haematite ores and alloys. The pharmaceutical samples like gelusil, digene, acidin, alucinol and almacorb also analysed. The procedure is safety on environmental basis as it is carried out in weak organic acid media.

Chapter 3, introduces the use of n-Octylaniline for extraction of gallium(III) from 0.005M succinate media at pH 4.0. Gallium(III) extracted in the
organic phase was stripped with water and was determined complexometrically by E.D.T.A. The extraction system is studied as a function of pH, equilibrium time, diluent, reagent concentration and diverse ions. Experimental data have been analysed graphically to determine the stoichiometry of the extracted species \([RNH_3^+. Ga (C_4H_4O_4)_{2}]_{\text{org}}\). The method is simple, rapid and selective.

Chapter 4, explains how a n-Octylaniline is useful for the selective extraction of indium(III) from sodium succinate media. The extraction system is studied as a function of various parameters. Experimental data have been analysed graphically to determine the stoichiometry of extracted species. Indium(III) was extracted into the organic phase by ion pair formation of the \([RNH_3^+. In(C_4H_4O_4)_{2}]_{\text{org}}\). The method is free from interferences of commonly associated metal ions. The results are employed in the determination of the metal ions in several synthetic mixtures and alloys.

Chapter 5, describes the extraction separation of thallium(III) from sodium salicylate media (0.02M - 0.05M) at pH range 2.3 - 2.7 with 1-6% n-Octylaniline in toluene. Thallium(III) extracted in organic phase was stripped with acetate buffer (pH 4.70) and determined complexometrically by E.D.T.A. The dependence of percentage extraction of thallium(III) has been studied by using different parameters. The method is free from interferences of large number of cations and anions. The method is successfully applied for sequential separation of thallium(III) from thallium(I) and analysis of synthetic mixtures and alloys.

Chapter 6, deals with the extraction of lead(II) from 0.005M sodium succinate with 5% n-Octylaniline at pH 8.2. The metal ion was stripped out from organic phase with water and determined complexometrically with E.D.T.A. or spectrophotometrically with PAR. The stoichiometry of the extracted species was determined on the basis of slope analysis and found to be 1:2:2 (metal:acid:
extractant). The system was used for selective extraction of lead(II) from its binary mixture with Zn(II), Tl(I), Tl(III), Bi(III), Al(III), Cu(II), Th(IV), Sn(IV), Sb(III) and Ca(II). The developed method was successfully applied for the analysis of lead(II) in real samples such as solder, type metal, gun metal, 5 g nonferrous alloy; galena ore, zinc ore and pharmaceutical samples like Nag Bhasma, Ekangvira, Garbhapal Rasa etc.

Chapter 7, explains the quantitative extraction and separation of aluminium (III), gallium(III), indium(III) and thallium(III) from each other. The separation scheme is based on use of masking agent strippants etc. The extraction scheme was developed and is used for binary, ternary and multicomponent mixtures in presence of masking agents, use of different weak organic acids and pH. The methods were readily combined with suitable complexometric methods for their determination after their extraction and separation.

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