

Bsc 4 sem{separation techniques solvent}

Analytical Instrumental

Analytical chemistry is concerned with the chemical characterization of matter and the answer to two important questions: what is it ? (qualitative) and how much is it ? (quantitative).

SOLVENT EXTRACTION

THE LECTURE IS CLASSIFIED TO: 1) Introduction 2) Basic Principles of Solvent extraction Method 3) The important of Solvent Extraction 4) Classification of Extraction Systems 5) methods of Extraction 6) Factors Influencing the Extraction Efficiency 7) Analytical Applications

Introduction To Solvent Extraction Solvent extraction is a technique extensively utilized in both industrial applications and in the laboratory. It includes a variety of techniques such as liquid-liquid extraction (LLE), Liquid-solid extraction (LSE), supercritical fluid extraction (SFE), and other special techniques. LLE is an extraction technique applied to liquids, liquid samples, or samples in solution, using a liquid extracting medium.

Introduction To Solvent Extraction

The quality of manufactured products often depends on proper chemical proportions, and measurement of the constituents is a necessary part of quality control [1]. Solvent extraction technique is a part of analytical chemistry and has been recognized as an excellent separation method because of its ease, simplicity, speed, and wide scope. -----

1) G. D. Christian, Analytical chemistry 6th Ed. (2004).

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2-1) BASIC PRINCIPLES OF SOLVENT

Extraction Method An extractant, is a substance primarily responsible for the transfer of a solute (here metal) from one phase to the other. The extractant is dissolved in a suitable diluent and together act as a solvent. The diluent is immiscible with other phase which is usually water.

GIBBS PHASE RULE,

$P+V=C+2$ (1) Where, P = is the number of phases, V = is the variance or degree of freedom and C = is the number of components And the number 2 corresponds Temp. and Pressure

GIBBS PHASE RULE

, In solvent extraction, we have $P = 2$ two phases namely aqueous and organic phase, the component $C=1$, viz. solute, in solvent and water phase and at constant temperature and pressure $P=1$, thus, we therefore have $2+1=1+2$ i.e. $P+V= C+2$ (2)

Nernst Distribution Law

According to Nernst distribution law, If $[X]_1$ is concentration of solute in phase 1 and $[X]_2$ is the concentration of solute in phase 2 at equilibrium: $K_D = [x]_1 / [x]_2$ (3) (Where K_D is called as the partition coefficient or distribution coefficient

The Partition Coefficient Or Distribution Coefficient this partition or distribution coefficient (K_D) is independent of the total solute concentration in either of the phases

Distribution Ratio (D)

The distribution of a solute between two immiscible solvents in contact to each other can be described by the distribution Ratio (D) $D = [X]_1 / [X]_2$ Where $[X]$ represents the stoichiometric or formal concentration of a substance X and the subscripts 1 and 2 refer to the two phases.

Distribution Ratio (D)

Since in most cases, two-phase system is of analytical interest, an organic solvent and aqueous are involved, D will be understood to be; $D = [X]_{org} / [X]_{aq}$ The subscript org. and aq. refer to the organic and aqueous phases respectively Distribution ratio 'D' is dimensionless quantity, separation of two solutes by solvent extraction is expressed by the term, separation factor (α), which is related to individual distribution ratios,

Separation Factor

$\alpha = D_A / D_B$ D_A and D_B are the respective distribution ratios of solute A and B.

Percent Extraction (%E)

The more commonly used term for expressing the extraction efficiency by analytical chemist is the percent extraction "E", which is related to "D" as $\% \text{ Extraction } (E) = 100D / (D + V_{aq}/V_{org})$ Where, V represent solvent volume and the other quantities remain as previously defined. The percent extraction may be seen to vary with the volume ratio of the two phases as well as with D.

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3) Solvent Extraction May Serve the following three purposes

May Serve the following three purposes: i) Preconcentration of trace elements ii) Elimination of matrix interference iii) Differentiation of chemical species.

4) Classification Of Extraction Systems

The classification of extraction systems is based upon the process of extraction. Thus, based upon the process of extraction, extraction systems can be classified into four major classes

Classification Of Extraction Systems a)

Chelate extraction b) Extraction by solvation c) Extraction involving ion pair formation d) Synergic extraction

(A -1)

Chelate Extraction

In this class, extraction proceeds by the process of formation of chelate or closed ring structure between the chelating agent and the metal ion to be extracted. e.g. i) The extraction of Uranium with 8-hydroxyquinoline in chloroform

(A-2)

CHELATE EXTRACTI

The extraction of Iron with cupferron in carbon tetrachloride the ammonium salt of N-nitroso-Nphenylhydroxylamine, is a common reagent for the complexation of metal ions. Its formula is $\text{NH}_4 [\text{C}_6\text{H}_5\text{N}(\text{O})\text{NO}]$. The anion binds to metal cations through the two oxygen atoms, forming five-membered chelate rings. Cupferron is prepared source: from phenylhydroxylamine and an NO

B) EXTRACTION BY SOLVATION

In this class, the extraction proceeds by the process of solvation of the species which is extracted into organic phase. Oxygenated organic solvents such as alcohols (C-OH), ketones, ethers and esters show some basicity because of the lone pair of electron on the oxygen atom and can therefore directly solvate protons and metal ions and bring about their extraction. e.g. i) The extraction of Uranium with tributyl phosphate from nitric acid ii) The extraction of Iron(III) with diethyl ether from hydrochloric acid.

C) Extraction Involving Ion Pair Formation

The extraction proceeds with the formation of neutral uncharged species which in turn gets extracted in to the organic phase. The best example of this is the extraction of Scandium and Uranium with trioctyl amine from mineral acids. In this case an ion pair is formed between complex of metal ion with high molecular weight amine and anionic species of mineral acids

D) Synergic Extraction

In this case, there is enhancement in the extraction on account of use of two extractants. e.g. the extraction of Uranium with tributylphosphate (TBP) as well as 2-thionyltrifluoroacetone (TTA)

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5) Methods Of Extraction

Three basic methods of liquid-liquid extraction are generally utilized in the analytical laboratory.

A) BATCH EXTRACTION

Batch extraction, the simplest and most commonly used method, consists of extracting the solute from one immiscible layer into other by shaking the two layers until equilibrium is attained, after which the layers are allowed to settle before sampling. This is commonly used on the small scale in chemical laboratories. The most commonly employed apparatus for performing a batch extraction is a separatory funnel. The batch extractions may also be used with advantage when the distribution ratio is large

B-1) Continuous Extraction

The second type, continuous extraction, makes use of a continuous flow of immiscible solvent through the solution or a continuous countercurrent flow of both phases. Continuous extractions

are particularly applicable when the distribution ratio is relatively small. Continuous extraction devices operate on the same general principle, which consist of distilling the extracting solvent from a boiler flask and condensing it and passing it continuously through the solution being extracted.

B-2) Continuous Extraction .

The extracting liquid separates out and flows back into the receiving flask, where it is again evaporated and recycled while the extracted solute remains in the receiving flask. When the solvent cannot easily be distilled, a continuous supply of fresh solvent may be added from a reservoir

C) COUNTER CURRENT EXTRACTIONS

Extraction by continuous countercurrent distribution is the third general type and is used primarily for fractionation purposes. The separation through continuous countercurrent method is achieved by virtue of the density difference between the fluids in contact. In vertical columns, the denser phase enters at the top and flows downwards while the less dense phase enters from the bottom and flows upwards. The choice of method to be employed will depend primarily upon the value of the distribution ratio of the solute of interest, as well as on the separation factors of the

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6) Factors Influencing

The Extraction Efficiency Primary requirement of solvent extraction for separation /removal purposes is a high distribution ratio of the solute of interest between the two liquid phases. It is useful to employ a number of different techniques for enhancing the distribution ratio. It depends on the nature of the species being extracted and extraction system. The attainment of selectivity in an extraction procedure is also very important. Some of the factors, which affect the distribution of solute of interest, are given below.

6-1) Nonchemical Factors

Affecting Extraction factors address the nonchemical elements of the extraction, which include (1) the choice of the extraction technique, (2) the choice of solvent and aqueous phase volumes, (3) the time of extraction, (4) the solvent evaporation procedure, etc.

These factors are important for achieving the appropriate extraction efficiency . for a successful utilization of LLE in sample preparation. The choice of the extraction procedure (batch or continuous), the number of extractions when using the batch procedure, etc.,
