SEC 1st Semester
UNIT: 5/ (Case studies)

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Separation of Binary Solid Mixture:

A binary solid mixture is a substance made up of two solid components. We can separate the components of a binary solid mixture using a variety of techniques, including:

Solvent extraction: Shake the mixture with a solvent that dissolves one of the components, then filter out the insoluble component

Sublimation: Heat the mixture until one component turns from a solid to a gas, leaving behind the other components

Also fractional crystallization, **steam distillation**, **chromatography** etc are also employed for the purpose

1. Separation of Benzoic acid and p-toluidine:

We can separate benzoic acid and p-toluidine using liquid-liquid extraction, which is a solvent extraction technique

The principle for separating benzoic acid and p-toluidine is solvent extraction, which is based on the difference in solubility of the two compounds in different solvents. . In this technique, we use an aqueous solution of a base to extract the acidic compound into the aqueous layer.

Procedure

An aqueous solution of a base, like sodium hydroxide or sodium bicarbonate is taken in a separating funnel. The mixture of benzoic acid and p-toluidine is added to the separating funnel and shaked vigorously and allow standing. Finally both the inorganic and organic layers are separated

Explanation

The base converts the acidic compound into its salt, which is more water-soluble. The salt is extracted into the aqueous layer.

Additional details

2. Separation of p-Nitrobenzoic acid and p-Aminobenzoic:

To separate p-nitrobenzoic acid from p-aminobenzoic acid, you can use a technique called acid-base extraction where you exploit the difference in their acidic properties: dissolve the mixture in an organic solvent like diethyl ether, then extract it with a dilute sodium bicarbonate solution; the p-nitrobenzoic acid will deprotonate and dissolve in the aqueous phase due to its stronger acidic nature, while the p-aminobenzoic acid will remain in the organic layer; finally, acidify the aqueous layer to recover the p-nitrobenzoic acid as a solid.

Explanation:

p-Nitrobenzoic acid is significantly more acidic than p-aminobenzoic acid because the nitro group ($-NO_2$) is a strong electron-withdrawing group, making the carboxylic acid proton more readily available for donation.

Steps involved in the separation process:

1. Dissolve the mixture:

Dissolve the mixture of p-nitrobenzoic acid and p-aminobenzoic acid in a suitable organic solvent like diethyl ether.

2. Add sodium bicarbonate solution:

Add a dilute aqueous solution of sodium bicarbonate ($NaHCO_3$) to the organic solution and shake well.

3. Separate layers:

The mixture will separate into two layers:

Aqueous layer: The p-nitrobenzoic acid will deprotonate and dissolve in the aqueous layer as the sodium salt.

Organic layer: The p-aminobenzoic acid will remain in the organic layer.

4. Acidify the aqueous layer:

To recover the p-nitrobenzoic acid, acidify the aqueous layer with a dilute mineral acid (like HCl) to precipitate the p-nitrobenzoic acid back out as a solid.

5. Isolate the p-aminobenzoic acid:
Evaporate the organic solvent from the organic layer to obtain the p-aminobenzoic acid.

3. Separation of the mixture of amino acid by ascending paper chromatography.

The aim of the experiment is to perform Ascending paper chromatography for the separation of amino acids present in the given sample.
Principle: Chromatography is used to separate mixtures of substances into their individual components. All forms of chromatography work on the same principle. They all have basic requirements of stationary phase (a solid or a liquid supported on a solid) and a mobile phase (a liquid or a gas). The mobile phase flows through the stationary phase and carries the components of the mixture with it. Different components travel at different rates based on their affinities toward stationary phase and mobile phase. In paper chromatography, the stationary phase is a very uniform adsorbent paper. The mobile phase is a suitable liquid solvent or mixture of solvents.
Retention or retardation factor (Rf): The distance the distance travelled by the solute to the distance travelled by solvent is called retention factor or retardation factor.
Requirements for Separation:
Apparatus: Glass beakers, Whatmann filter paper as chromatography paper, developing chamber and capillary tubes etc.

Chemicals: n-butanol, solvent system, amino acid mixture. ninhydrin reagents.

Solvents system:

A mixture of n-butanol, glacial acitic acid and water in the ratio 4:1:5

Procedure:

The procedure for ascending paper chromatography method is quite simple as compared to other methods of chromatography. The chromatography paper is cut into rectangular strips and marks a line on the paper with pencil at about 2 cm from the bottom. With the help of capillary tube, the samples are applied at different points on the starting line. Now, place the chromatography paper in the developing chamber, which contains the mobile phase. While placing the paper, it is important that the solvent level should not reach the starting line. After sometime the solvent rises up the paper or the stationary phase by capillary action and dissolves the sample. The components of the sample move along with the solvent in upward direction. The paper is removed when it reaches the top and marked the level with pencil. This level or height is called the "solvent front". By using **ninhydrin solution** examined the different spots of varied colors. Each spot represents a specific component of the amino acid. Finally the components are identified from their Rf valued by consulting standard literature

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