EXTRACTION :

Separation of aspirin, β-naphthol, and naphthalene by extraction

Extraction relies on the solubility of substances into solvents and the insolubility of the solvents into each other. It also means the selective transfer of any solute or impurity from one solvent to another. One of the solvents is usually water and the other is a water-insoluble solvent such as ether or chloroform. Removal of the solvent yields the substance to be isolated.

Frequently, the solute is not completely transferred from one solvent to another. In these cases, repeated extractions would be required to transfer the solute from one solvent to the other.

When a water solution of a substance "X" is shaken with an organic solvent (such as ether), substance "X" will distribute itself between water and ether. The concentration of "X" in each solvent is proportional to its solubility.

The ratio of concentrations in each solvent is the Distribution Coefficient: $K_D$.

$$K_D = \frac{\text{Concentration of "X" in Organic Solvent}}{\text{Concentration of "X" in Water}} = \frac{\text{Solubility of "X" in Org. Solvent}}{\text{Solubility of "X" in water}}$$

For example:

a) Given the solubility of solute "x" is 0.56 g/100 ml of ether and 0.14 g/100 ml of water, find $K_D$.

$$K_D = \frac{\text{Solubility -Org.Solvent}}{\text{Solubility in water}} = \frac{0.56 \text{ g/100 ml}}{0.14 \text{ g/100 ml}}$$

b) If a 40 mg of "x" were present in50 ml of water and extracted with 50 ml of ether, then the amount of "x" removed[extracted] by ether can be calculated:

Assume the amount of "x" removed by ether =Y mg

$$K_D = \frac{\text{Concentration of "x" in Organic Solvent}}{\text{Concentration of "x" in Water}} = \frac{\text{Y mg/ 50 ml of ether}}{\text{(40 mg -Y )/ 50 ml of water}} = 4$$

Y= 32 mg of solute extracted by ether. Remaining amount in water = 40-32= 8mg

In the above example, ethyl ether was used as an extracting solvent. It has several advantages: high solvent power, relative inertness, low boiling point.
Ether has some disadvantages such as its high flammability.

Another minor problem is that ether dissolves some water. One needs to treat the ether solution with a drying agent to remove traces of water.

The capacity of the drying agent refers to the amount of water absorbed by the drying agent( per unit weight).

A good drying agent should have a high efficiency and a rapid rate of drying. Examples of commonly used drying agents include magnesium sulfate, sodium sulfate and calcium chloride.

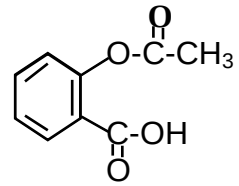
In this experiment, three organic compounds (aspirin, β-naphthol, and naphthalene) will be separated from each other. The three compounds are all soluble in ethyl ether (an organic solvent). By selectively reacting each organic compound, we can make it soluble in water and insoluble in ethyl ether. Since ethyl ether and water are insoluble in each other, they will form two layers and can be separated from each other using a separatory funnel.

The reacted organic compound which is in the aqueous portion is then converted back into the insoluble organic compound which precipitates out of the aqueous portion.
Aspirin, β-naphthol, and naphthalene are all soluble in ether.
Sodium bicarbonate (aqueous) will be added to the ether solution. Only the stronger acid { aspirin} reacts with the sodium bicarbonate.
The aqueous layer is separated from the organic layer (extraction). The aqueous layer contains the aspirin salt. The naphthalene and β-naphthol remain in the ether layer.
Aspirin is then precipitated out of the aqueous layer by reacting it with HCl.

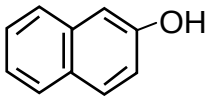
The β-naphthol is then reacted with sodium hydroxide (aqueous) to form an aqueous soluble salt.

The aqueous layer is then separated from the organic layer. The sodium salt of β-naphthol can be converted back to the insoluble β-naphthol and precipitated out.
The remaining ether layer is then evaporated leaving naphthalene behind.

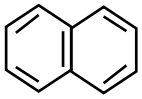
The following scheme illustrate the separation method:



Aspirin



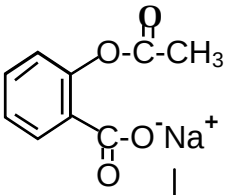
ß-naphthol



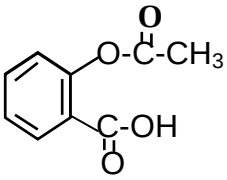
naphthalene

***Dissolve in Ether, Add 20 ml $NaHCO_3$ and Shake***
**Separate the two layers**

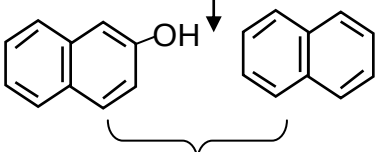
***Aqueous layer***



**acidify**
**( 6M HCl)**

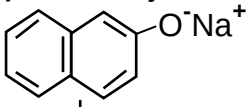


***Ether layer***

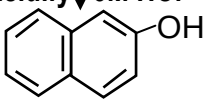


**add 20 ml**
**NaOH**
**Separate the two layers**

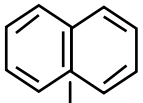
***Aqueous layer***



**acidify 6M HCl**



***Ether layer***



**Add a drying agent, filter, evaporate the solvent**

Using the Separatory Funnel