Separating a Basic Compound (e.g. Amine) From A Crude Mixture

Step 1: Dissolve crude mixture in organic solvent (e.g. diethyl ether, \(\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_3\))

Step 2: Transfer to Seperatory Funnel

Step 3: Add solution of aqueous acid (e.g. 2M \(\text{H}_2\text{SO}_4\)) and shake vigorously

- Water is more dense than ether, so it will be on the bottom layer. (note: chlorinated solvents like \(\text{CH}_2\text{Cl}_2\) are more dense than water & will be on bottom)
- Acid will protonate any amines in the crude mixture, forming salts, which will then dissolve in the aqueous layer

Step 4: Collect Aqueous Layer

[Set organic layer aside - it contains any neutral & acidic components of the crude mixture]

Step 5: Adjust pH of aqueous layer to pH >14 with concentrated base (e.g. 6M \(\text{NaOH}\))

Step 6: Transfer to separatory funnel, add organic solvent (e.g. \(\text{Et}_2\text{O}\)) and shake vigorously

- The organic layer (top if \(\text{Et}_2\text{O}\) is used) should contain the neutral amine
- A precipitate should form as the salt is neutralized to its water-insoluble free-base form.

Step 7: Collect the organic layer and remove solvent (e.g. using a rotary evaporator)

This should result in separation of any basic components from the rest of the crude mixture!

Separating an Acidic Compound (e.g. Carboxylic Acid) from A Crude Mixture

This is conceptually similar to the procedure on the left, except that we will convert the neutral compound to a salt by using base, and do the final neutralization using acid

Step 1: Dissolve crude mixture in organic solvent (e.g. diethyl ether, \(\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_3\))

Step 2: Transfer to Seperatory Funnel

Step 3: Add solution of strong base (e.g. 2 M \(\text{NaOH}\)) to adjust pH to 14 and shake vigorously

- Aqueous layer should be on bottom unless chlorinated organic solvents are used (e.g. \(\text{CH}_2\text{Cl}_2\))
- Strong base will convert any acidic compounds into their conjugate bases; these salts should dissove in the aqueous layer

Step 4: Collect Aqueous Layer (set organic layer aside)

Step 5: Adjust pH of aqueous layer to pH 1 with concentrated acid (e.g. 6M \(\text{HCl}\))

Step 6: Transfer to separatory funnel, add organic solvent (e.g. \(\text{Et}_2\text{O}\)) and shake vigorously

- The organic layer (top in most cases) should contain the neutral carboxylic acid
- A precipitate should form as the salt is neutralized to its water insoluble neutral form

Step 7: Collect the organic layer and remove solvent (e.g. using a rotary evaporator)

This should result in separation of any acidic components from the rest of the crude mixture!