Chapter:

Solvent Extraction

Prepared By:

M Junaid Sahoo

Lecturer Chemistry in HED (Govt. College)

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Chemistry with MJS

Chemistry Preparation by MJS

Separation Techniques

★ **Filteration:** (Basic technique)
↳ separation of the whole components.

e.g Separation of AgCl

★ **Crystallization:** → separation technique (used when crude substance solid like sublimation)
↳ used when Solid substance Decompose at high Temp.

★ **Sublimation:** ↳ transition of substance directly From solid to gaseous Form (used when solid does not decompose at T↑)

★ **Extraction:**
Separation of a desired component → from solution.

★ Every techenique has own specificity / Domain

- **Analyte** → wanted material in extraction.
- **Raffinate** → unwanted materiall
OR
Remaining portion after extraction

- Extraction techniques give the better Results as compared to simple techniques.

Liq - Liq Extraction
OR
Solvent Extraction

↳ substances are separated on the basis of affinities / solubilities in two diff immiscible phases / liquids
↓
Solvent May be

- Aquous + NON-Aquous
- Aquous / organic phase
- polar / non polar
- Generally we use Separating Funnel-

liq - Solid Extraction

↳ Solvent is added to a solid

solvent may be;

★ Hydrophilic | Hydrophobic
★ Acidic | Basic

BEST Example;
Solid extraction
↳ Excellent Separating technique

- Soxhlet appratus is used.

★ Extraction is Reversible phenomena

★ in Extraction weak Forces are present (Wander walls)

✓ Liquid-Liquid Extraction: / Solvent Extraction:

↳ Involves the distribution of solute b/w two immiscible liquid phases.

- This is simple, clean, rapid and convenient
- Also Applicable to the trace level and large amount of materials.

✓ Nernest law of Distribution: (principle)

This law states that;

If a system containing two immiscible liquids, and solute is distributed itself b/w two liquids in such a way that equilibrium ratio of molar concentrations remains constant provided its molecular state same in both liquids & T remains constant.

$$\boxed{K_D = \frac{S_{organic}}{S_{aqueous}}}$$

→ Ratio of solubilities in two solvents

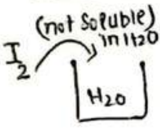
→ Ratio Remains constant (Thermodynamic Equilibrium constant) has a fixed value for phases

K_D ↓ Distribution coefficient OR partition coefficient

- K_D ↑ solute will move go toward organic phase
- K_D ↓ solute move toward Aqueous phase

Example

Extraction of I_2 from Aqueous layer



→ so we add salt KI

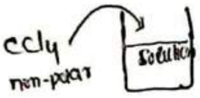
$$KI \longrightarrow K^+ + I^-$$

$$I^- + I_2 \rightleftharpoons I_3^{\ominus}$$

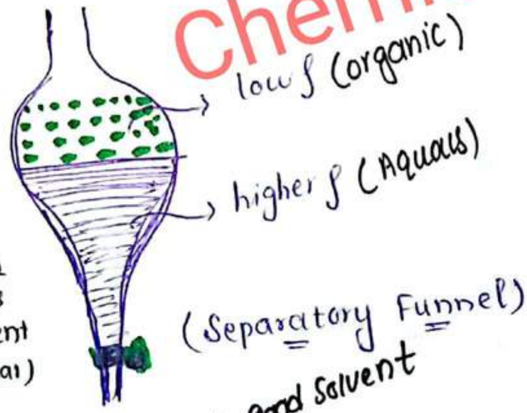
I_2 (insoluble) in H_2O

$I_3^{\ominus}$ → soluble in H_2O

↓ Now we can separate I_2 from Aqueous By using solvent CCl_4 (non-polar)



→ I_2 will go back to CCl_4 layer



(Separatory Funnel)

★ organic solvents → Diethyl ether*, Isopropyl ether, petroleum ether, benzene or CCl_4

* → good solvent

> ★ This technique is prefferely used, when the products are volatile and Thermally unstable.

A good Solvent; ★ It should have favourable temp. coefficient → At room temp, evaporate

- ★ Substance should be highly soluble in the solvent
- ★ Easily separable from water & solute
- ★ immescible & volatile in nature ★ should dissolve more in solute than 1st solvent

∴ If $K_D\uparrow\uparrow$ → Single extraction is used

e.g separation of metal chelates in organic solvents

∴ If $K_D\downarrow\downarrow$ → Then we have to use multiple extractions

Distribution Ratio & Extraction Efficiency:

Ratio of Amount OR concentration
↓
changes with solution conditions
↓
depends upon amounts OR concentration.

$$D = \frac{\text{Amount of Solute in organic phase}}{\text{Amount of Solute in Aqueous phase}}$$

$$D = S_{org}/S_{aq}$$

$$\boxed{\%E = \frac{D}{D+\left(\frac{V_{aq}}{V_{org}}\right)} \times 100}$$

★ If we use equal volumes of both layers;

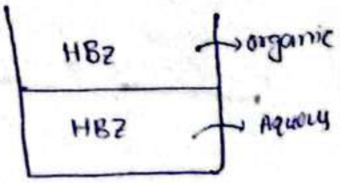
Then; Extraction Efficiency;

$$\boxed{\%E = \frac{100\,D}{D+1}}$$

Example;

Extraction of Benzoic Acid From an Aquous solution

$$K_D = \frac{[C_6H_5COOH]_{organic}}{[C_6H_5COOH]_{Aquous}}$$



Benzoic Acid ionizes in H_2O

$$C_6H_5COOH_{(aq)} \rightleftharpoons C_6H_5COO^- + \boxed{H^+}$$

$$K_a = \frac{[C_6H_5COO^-][H^+]}{[C_6H_5COOH]_{aq}} \checkmark$$

Benzoic acid in Aquous phase gives un $C_6H_5COO^-$

So,

$$D = \frac{[C_6H_5COOH]_{organic}}{[C_6H_5COOH]_{aq} + [C_6H_5COO^-]_{aq}} = \frac{[HBz]_{org}}{[HBz]_{aq} + [Bz^-]_{aq}}$$

~~Ka=~~ ~~D=~~ $\therefore \boxed{[HBz]_{org} = K_D [HBz]_{aq}}$

$$D = \frac{K_D [C_6H_5COOH]_{aq}}{[C_6H_5COOH]_{aq} + K_a [C_6H_5COOH]_{aq} / [H^+]_{aq})}$$

$$D = \frac{K_D [C_6H_5\cancel{COOH}]_{aq}}{[C_6H_5\cancel{COOH}]_{aq} [1 + K_a / [H^+]_{aq}}$$

$$\boxed{D = \frac{K_D}{1 + K_a / [H^+]_{aq}}}$$

Result

①: If $[H^+]_{aq} >> K_a$ $D \simeq K_D$

If D ↑ → [HBz] → go toward organic phase.

②: If $[H^+] << K_a$ → Then D ↓ → [HBz] → go toward Aquous phase

: Types OF Liquid-Liquid Extraction:

- Batch Extraction
- continuous Extraction
- counter-current Extraction

(i) Batch-Extraction:

- ★ Carried out by using separating Funnels.
- ★ Extraction By this method is carried out when when partion cofficient of solute K_D ↑↑ very high.
- ★ Generally It is called Singel Extraction due to high K_D value, but multiple extractions can be performed to improve the Efficiency.
- ★ For more Efficiency Fresh reactant is added to Raffinate and Extraction is continued several times.

- ★ This Method is simple & quick
- ★ This method is widely used For extraction on small scale. It can be generally performed in college Laboratories using Separating Funnels.
- ★ In this Extraction, Equilibrium is achieved B/W two phases, After this two immissible layers are allowed to settle and separated.
- ★ It can be single, Double & Multiple Extractions.

↱ Extraction → carried out to smaller volume, two or three times.

✓ Multiple Extraction: | Repeated Extraction |

- More Efficient and Convenient method.
- complete separation of solute.

✓ Smaller volume extraction

Let; of x - particles extracted in 100 ml of of organic phase & aquous phae.

$$K_D = 2 = \frac{x/100}{(100-x)/100ml}$$

① 1st Extraction

$$2 = \frac{x}{100-x}$$

✓ $\boxed{x = 67}$ particles in 100mL organic

Raffinale = 33 particles

② 2nd Extraction

$$2 = \frac{x|100}{33-x|100}$$

✓ $\boxed{x = 22}$ → Further go toward organic

∴ Raffinate = 11

③ 3rd Extraction

$$2 = \frac{x|100}{11-x|100}$$

✓ $\boxed{x = 7.3}$

(ii) Continuous Extraction:

- ★ Applicable when K_D value of Solute is Relatively Small.

- ★ Generally used For the separation of components of a ~~Solid~~ liquid mixtures. So, Soxhlet type appratus is used (special kind of extractor) ↓(For liq-Solid)

- ★ Efficient Extraction can be conducted with a minimum of Extracting solvent.

- ★ An Immiscible Extracting liquid is Kept Flowing continuously through the solution from which a solute is to be extracted.

- ★ very Fast Reaction/ performance, so no Equilibrium is achieved.

- ★ Two solvents are used, one is extracting solvent (non-Aquous) which is Lighter than sample solution (aqueous soln.). But in soxhlet appratus we used only one solvent.

Advantage
- uses a small amount of solvent
- can remove high amount of solute
- Excellen Efficiency than Batch extraction

(iii) Counter-current Extraction:

↳ Also a type of Liq-Liq Extraction.

- ★ In this organic solvent and Aqueous solvent move continueously in opposite direction & solute distributes in two oppositely moving phases.

- ★ in this Equilibrium is not achieved.

- ★ Craig Appratus is used For the counter current Extraction, containing series of glass tubes that are designed and arranged such that the lighter liquid phase is transferred from one tube to another tube.

- ★ Efficient process of extraction.

✓ Solid-Liquid Extraction:

↳ Soxhlet extraction appratus is used.

- ★ Required soxhlet, when desired compound has limited solubility in a solvent.
- ★ Extraction carried out by distillation & condensation of solvent.
- ★ Mutiple extractions to be done repeatedly using same volume of solvent.
- ★ Standard method of extraction of Solids From crude material.
- ★ Soxhlet extractor used For thermostable compounds.

Soxhlet Appratus:

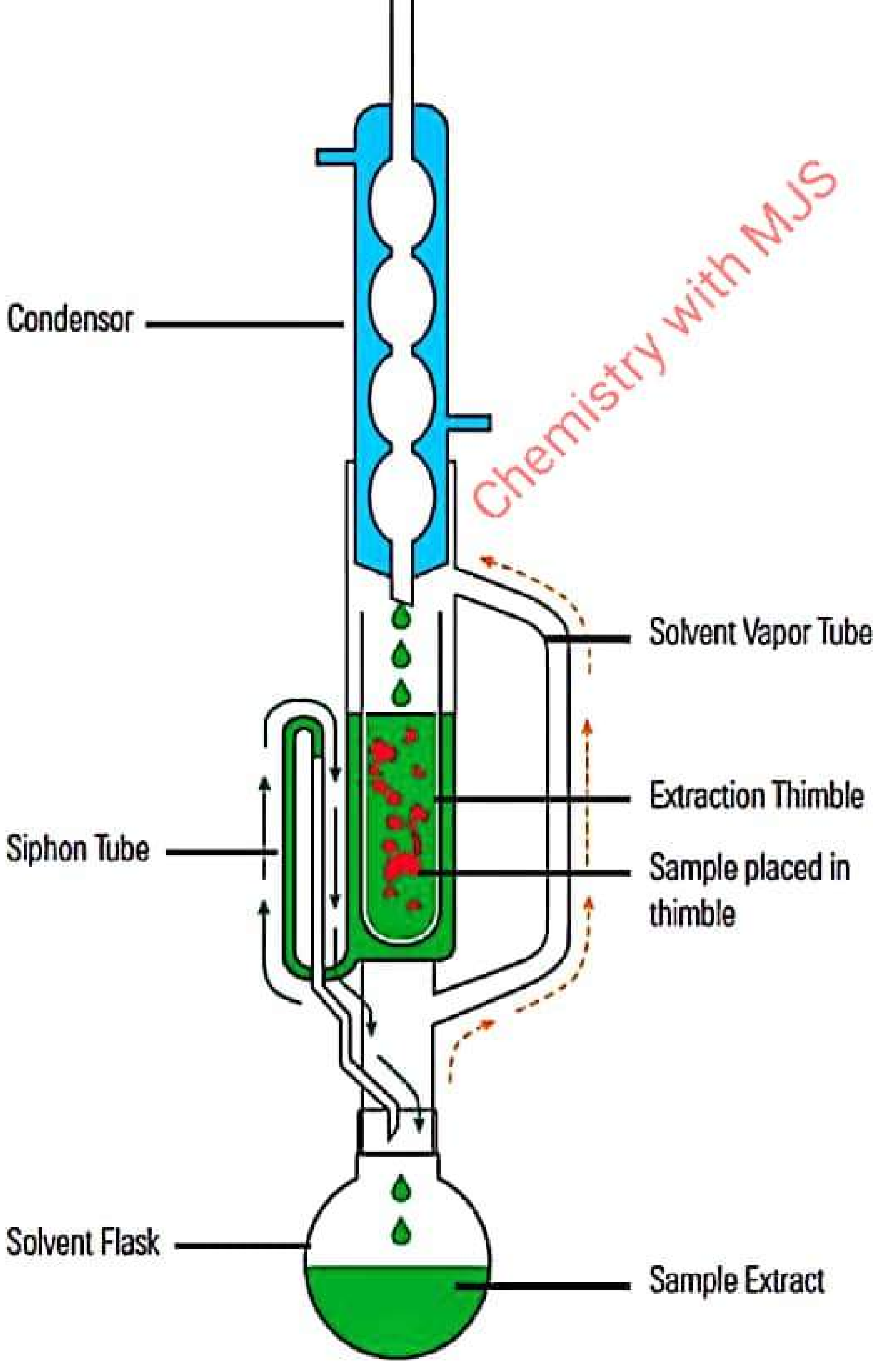
- Soxhlet Extractor
  - ★ Thimble Holder
    - Solid Sample
  - ★ Siphon tube
    - connects the thimble to distillation Flask
- Distillation flask
  - having solvent
  - placed on heating Source, to boil the solvent → vapours
- Condenser
  - connected to distillation Flask
  - cools down the solvent vapors to back to liquid form.

★ cyclic and continuous process, untill desired level of extraction is achieved.

USES:

⇒ Extraction of natural products.

⇒ Determinination of fat content in Food.


Condensor
Solvent Vapor Tube
Extraction Thimble
Siphon Tube
Sample placed in thimble
Solvent Flask
Sample Extract

4: Factors Affecting Solvent Extraction:

Effect of T:

★ temperature affect the solubility of solute toward Repective phase.

★ By increasing T → solubility/affinity increases extraction will become fast

Effect of innert solutes:

e.g $CaCl_2$, $MgCl_2$, sucrose etc.

Some organic solvents such as ★ethanol & ★methanol computely miscible with H_2O in the absence of salt. in the presence of $CaCl_2$, $MgCl_2$, sucrose → salting out

★ **Surface Area of Solid:**

- Large surface Area → increases the extraction Rate

Viscosity of Solvent: As η↓ Extraction↑

Effect of pH:

→ High & very low pH → ionization occurs

→ moderate pH → No ionization.

pH affects the Stability and charge on metal complex.

★ The Acidic pH pH at which metal ion can complex is most stable and ★neutral is the best pH for extraction of metal ions. $\underline{[H^+]} >> Ka$

Flow Rate of Solvent: ↑ extraction↑

Nature of solvent:

→ polarity

→ B.p → solvent should have low B.P → so that evaporate

Synergic★ agents: → enhance the extraction power of metal complexes with organic phase

These reagent when added to organic phase increase the efficiency of extraction.

They get associated with metal complexes, make it more soluble into organic phase

Applications:

1) Making tea is a good example of extraction.

② many organic Reagants formed coloured* chelates with metals, (* metal chelates through organt) but most of the chelates are insoluble in H_2O. they easily soluble in organic Solvents and can easily be extracted.

③ Also used in drug Analysis. → mostly these are organic → Separation of drugs.

④ Extraction of Gold & platinium e.g in Jewelry industry precious metals are extracted.
- Ni → extracted from steel industy.

⑤ In oil and Fat industry - → extraction by hexane
↓ to extract the oil & Fats from seeds.

⑥ Separation of metal cations-

⑦ Separation of organic compounds from Different plant sources.

⑧ Blood Samples → extraction of antibodies from blood, which are used for The treatment of diseases (viruses)

⑨ pharmaceutical industry → to separate the compounds & make it pure.

⑩ It is used to separate the components of Mixtures.

🌹 Good Luck 🌹