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Separation & Purification of Organic Compounds

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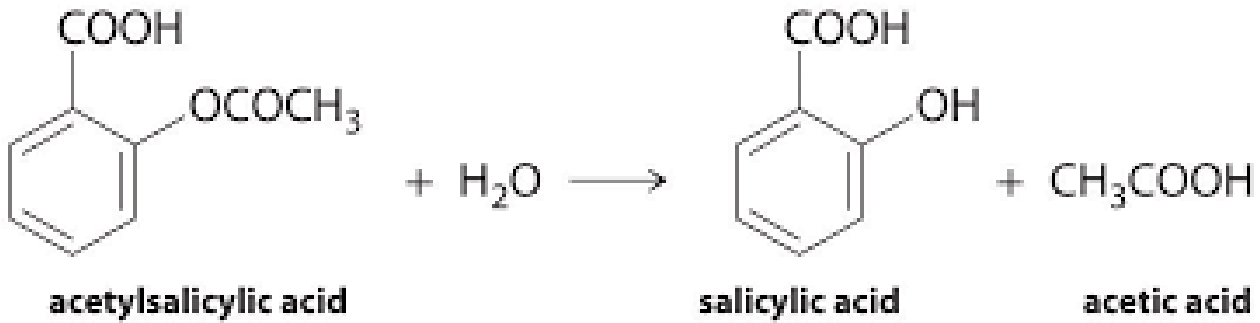
Lab: 4

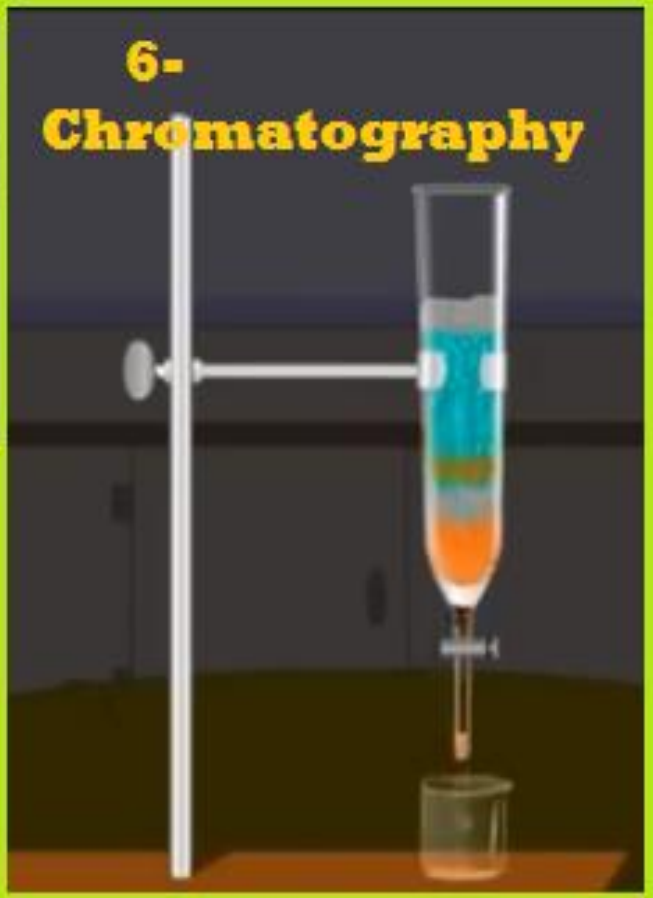
Separation & purification of organic compound is important:

1- Products of organic reactions are seldom pure products as a result of side reactions.

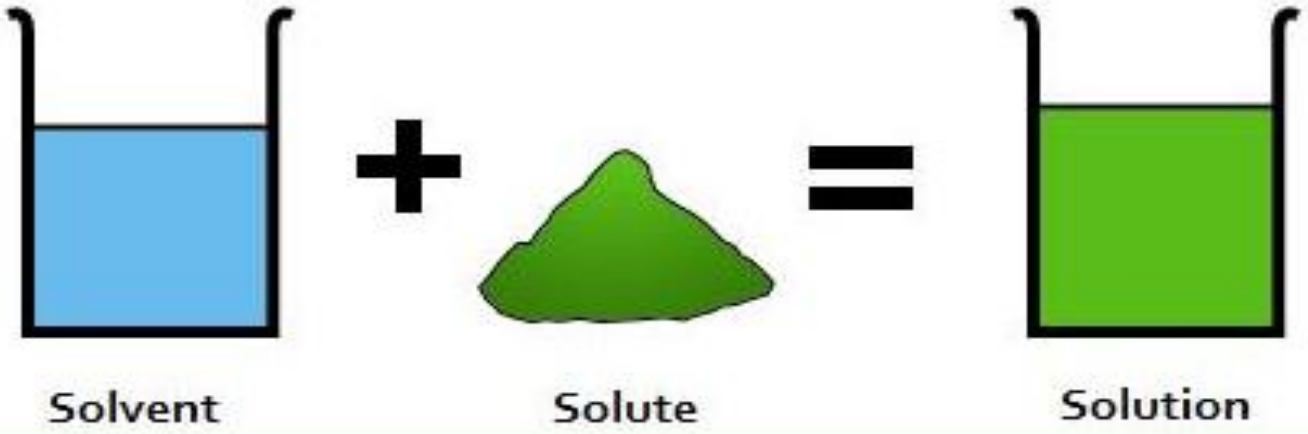
2- Pure compounds are also subject to partial decomposition on standing for some time or on exposure to light, air, heat, moisture, etc., ex. **acetyl salicylic acid, aspirin®**, decomposes to salicylic acid & acetic acid

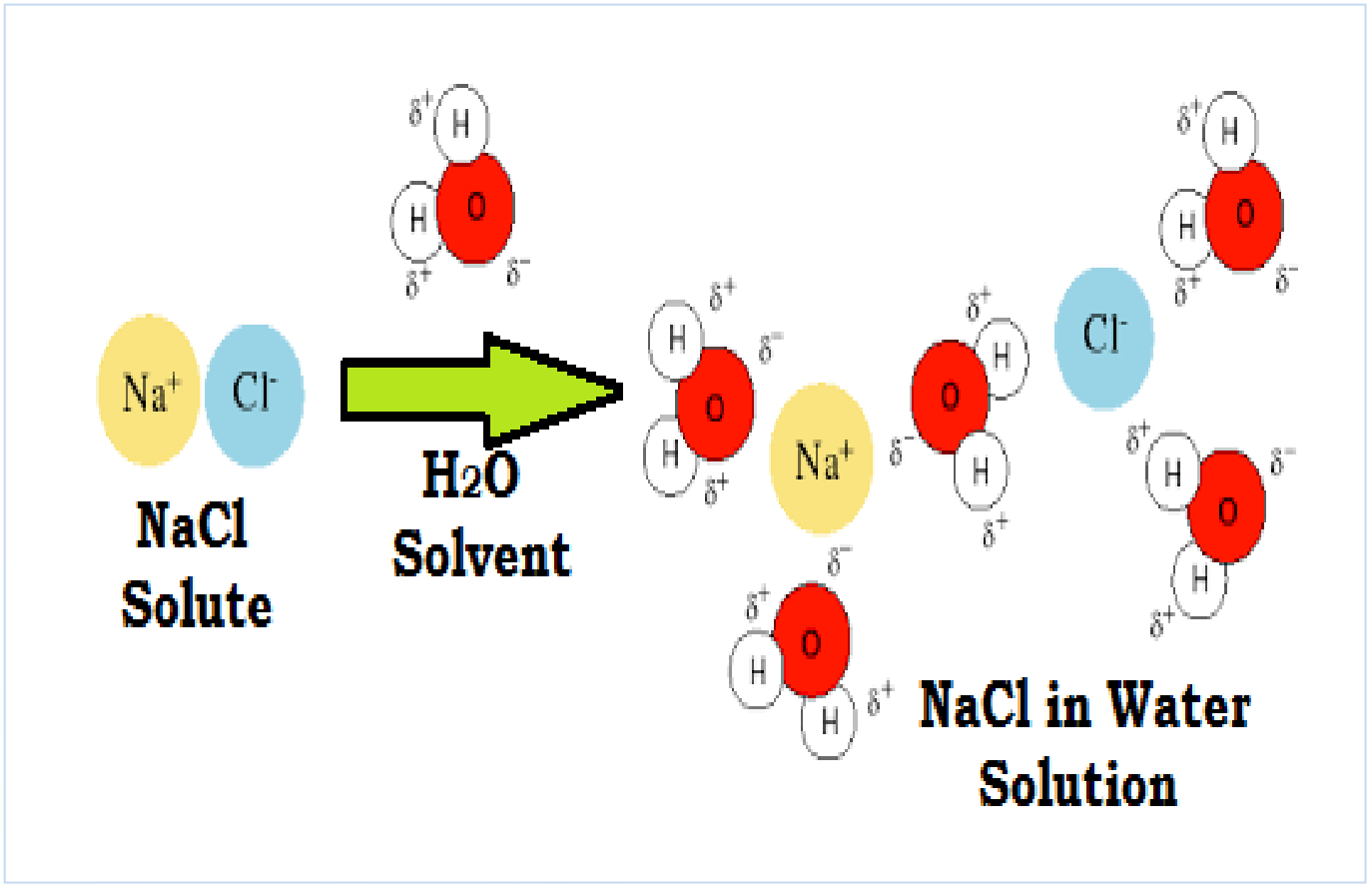


Methods of Separation & Purification of Organic Cpd

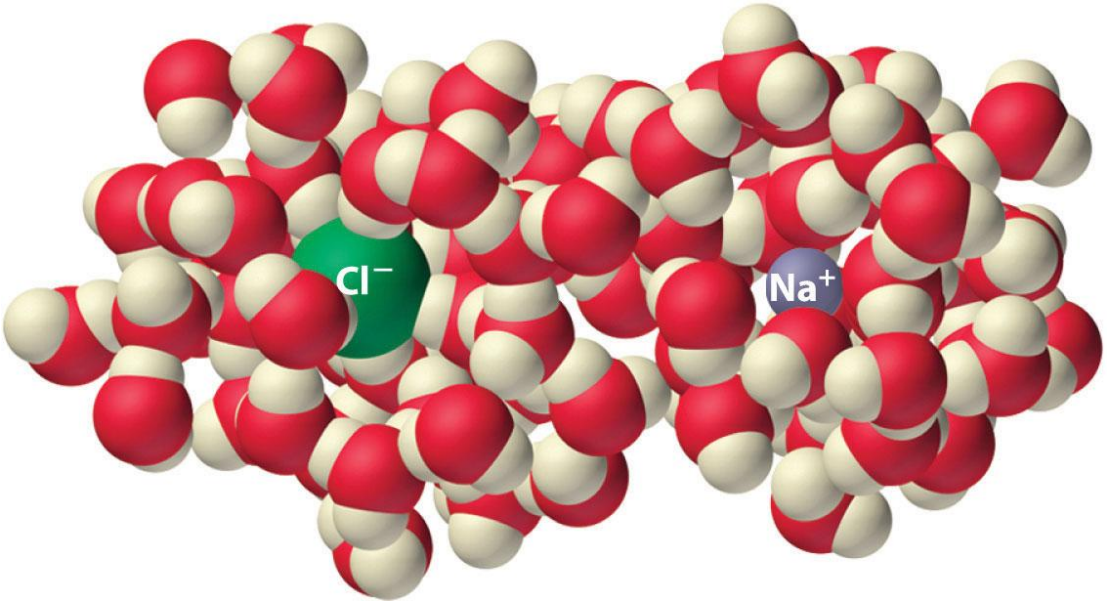


Solutions & Solubility



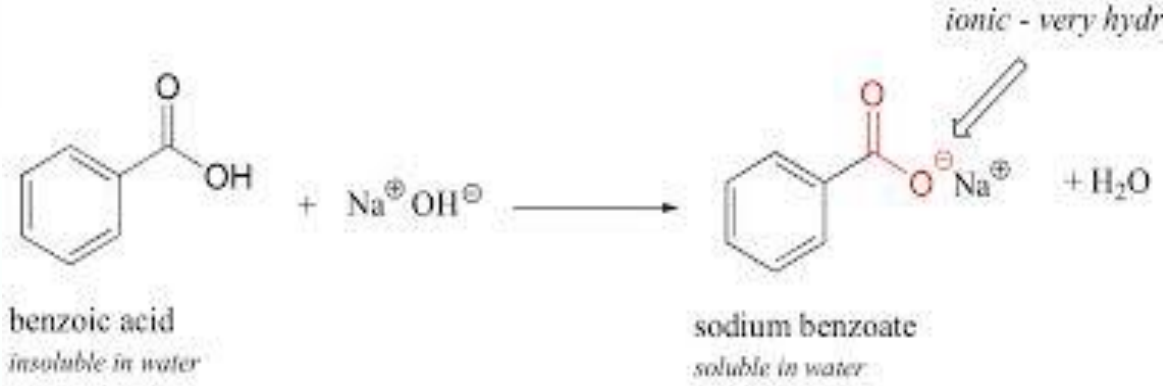


When a solid or a liquid dissolves, the structural units (ions or molecules) become separated from each other and the solvent molecules occupy the space between them.

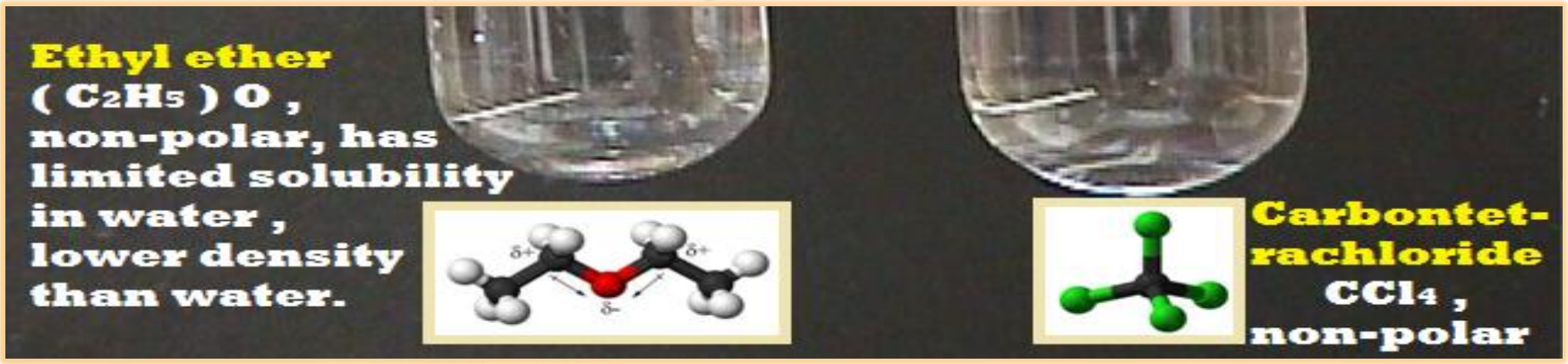

Cl⁻
Na⁺

The solubility of organic cpd. can be divided into two major types :

1- Solubility in which the chemical reaction is the driving force, *ex*. Acid-base reaction



2- Solubility in which only simple miscibility is involved, *ex*. ethyl ether in CCl_4



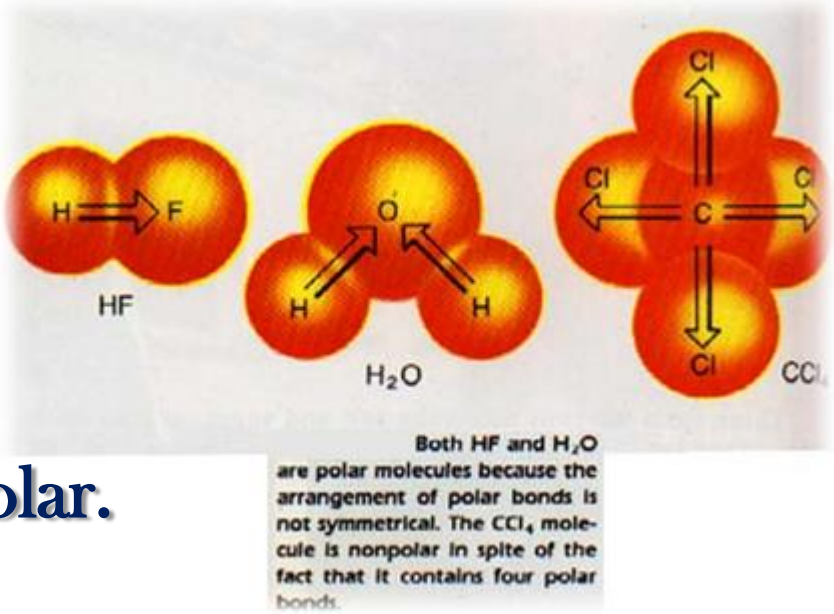
Theory of Solubility:

1- Polarity effect:

" like dissolves like ".

F > O > Cl , N > Br > C , H

Decreasing electronegativity



Both HF and H_2O are polar molecules because the arrangement of polar bonds is not symmetrical. The CCl_4 molecule is nonpolar in spite of the fact that it contains four polar bonds.

H_2, O_2, Cl_2, Br_2 & N_2 has zero dipole moment, i.e. non polar.

2- Dielectric constant:

D.C., is the ability of the solvent to separate ionic charges.
The D.C. of the solvent is related to it's polarity.

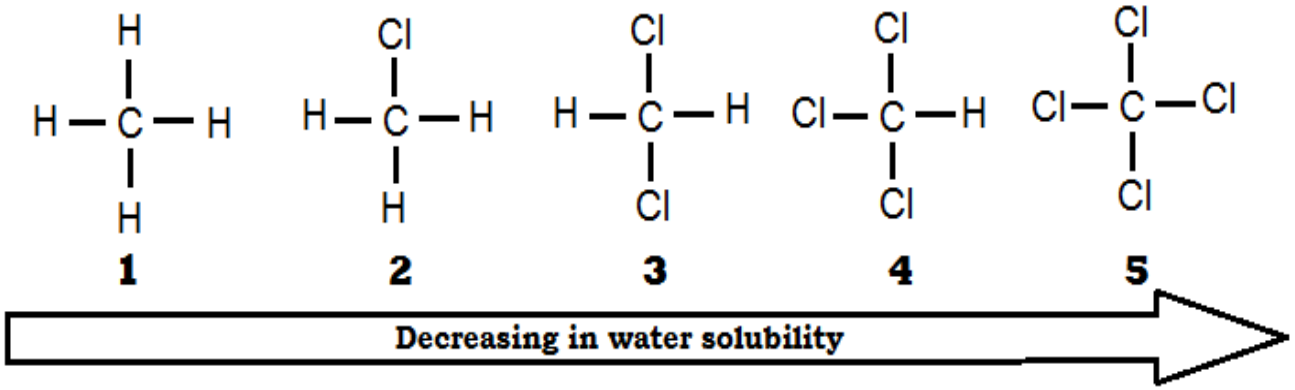
Water D.C. of 80 dissolves NaCl readily,
Hexane (D.C. of 1.9)
Diethyl ether (D.C. of 4.4) → Poor solvents for ionic salts.

The effect of cpd's structure on it's water solubility :

1- Number of carbon atoms : Solubility ↑ as the no. of C atoms ⩽ 5

2- The presence of = , ≡ , Ar. ring ~~affect~~ solubility

3- As a halogen is substituted for a H, the water solubility ↓



4- Acids & amines are more soluble than non polar cpds.

5- In a homologous series as ↑ m.wt. ⟹ Solubility ↓

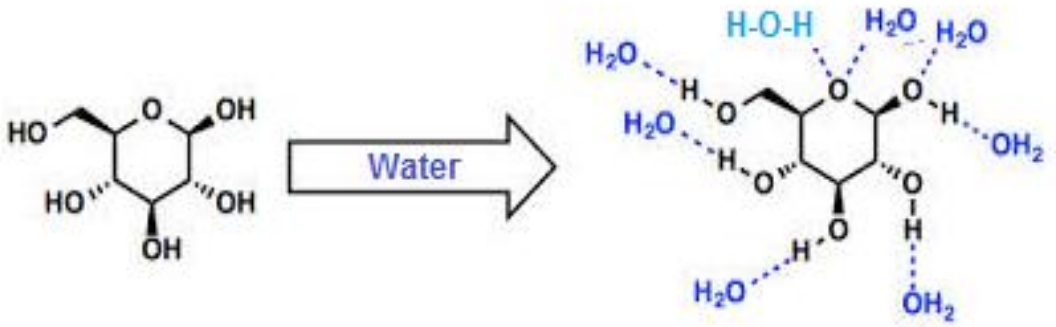
6- Saccharides,

ex. Glucose,

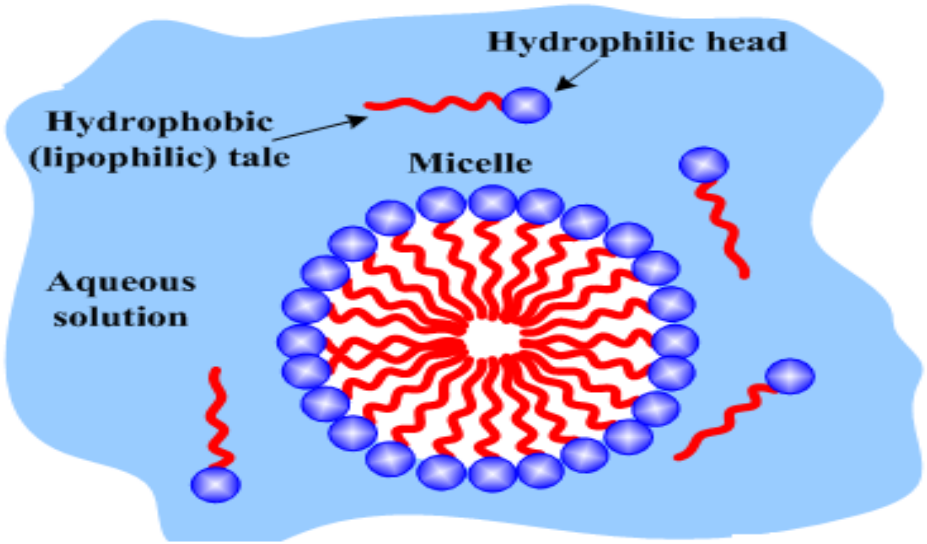
have many polar

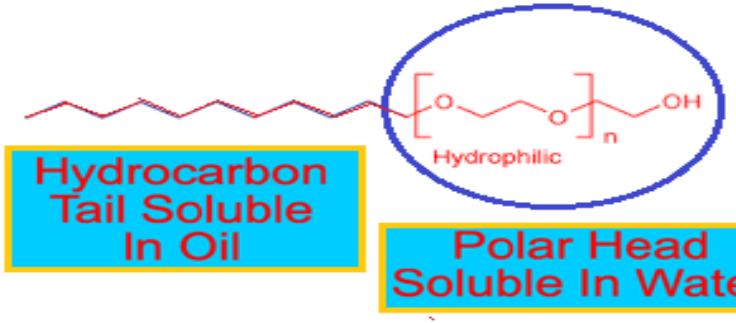
- OH groups , **so**

they are water soluble.



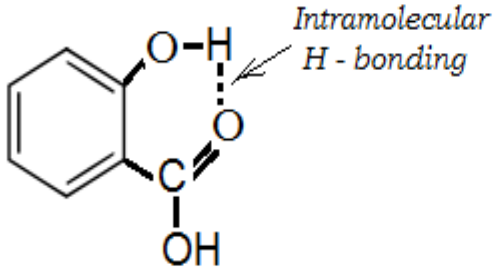
7- Compounds having both **polar & non polar** parts are soluble in water.





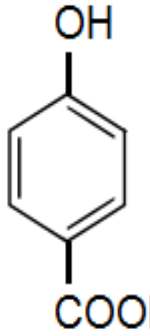
7- Intramolecular **H- bonding**

↓ water solubility.



Salicylic acid

Less water solubility

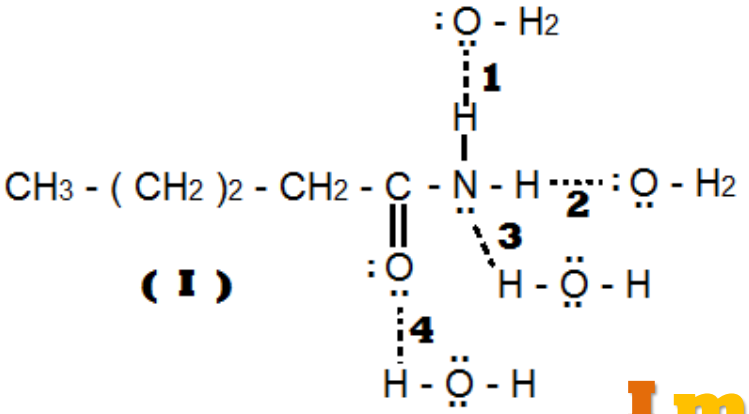


p- Hydroxybenzoic acid

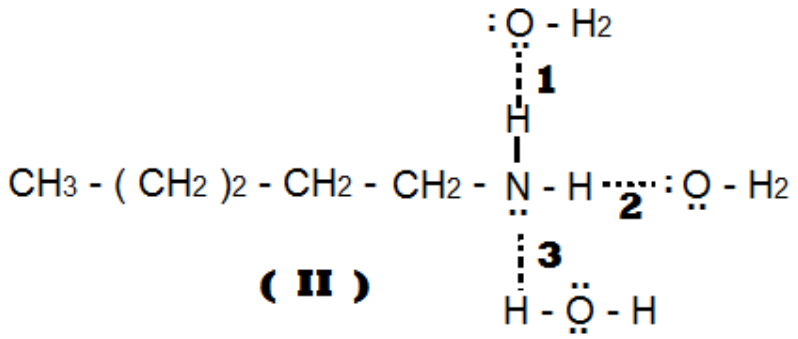
More water solubility

8- Intermolecular **H – bonding** between cpd & water

↑ water solubility.



(I)



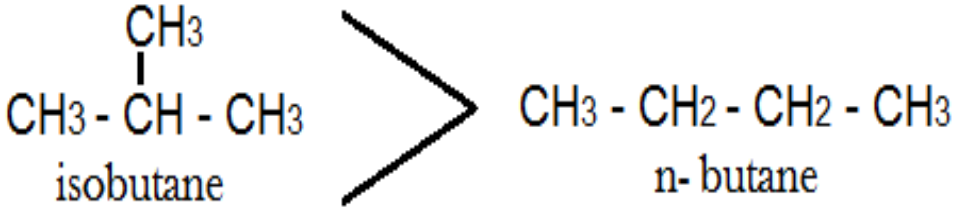
(II)

I more water soluble than II.

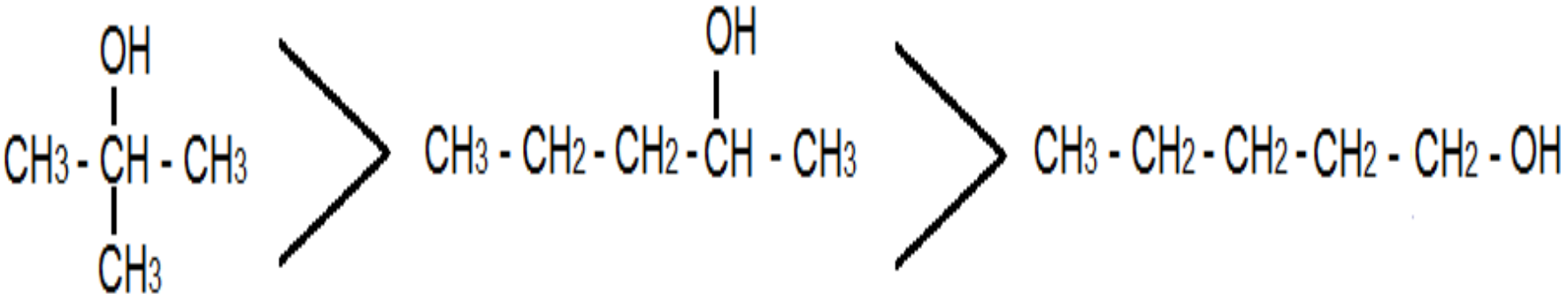
10- Molecular structure:

a- Branching

water solubility.



b- Position of the functional group.




FILTRATION

Filtration:

It is an important procedure after completing the reaction either to:

1- Separate the solid product (precipitate), or,

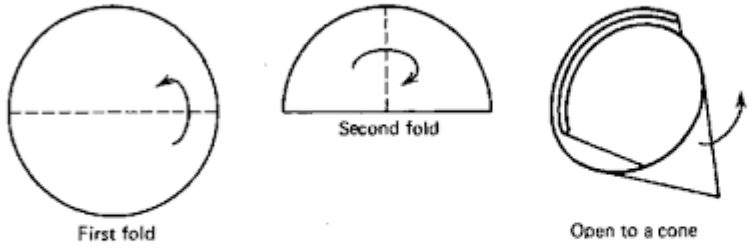
2- To get rid of insoluble impurities or reactant materials.

The desired soluble cpd. is recovered from the filtrate by evaporating the solvent.

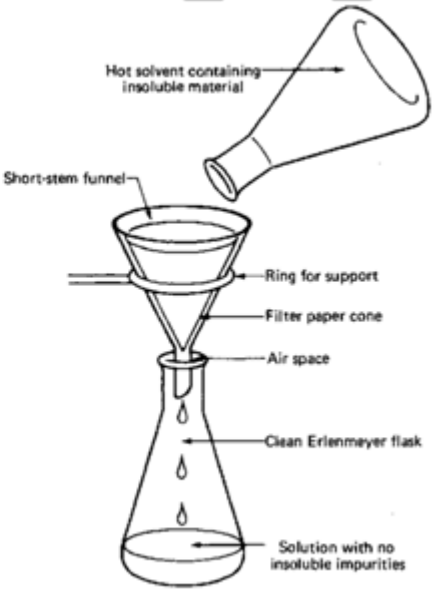
So the liquid is poured into a filter paper fitted in a funnel and either the precipitate or the filtrate that contains the desired soluble compound is collected.

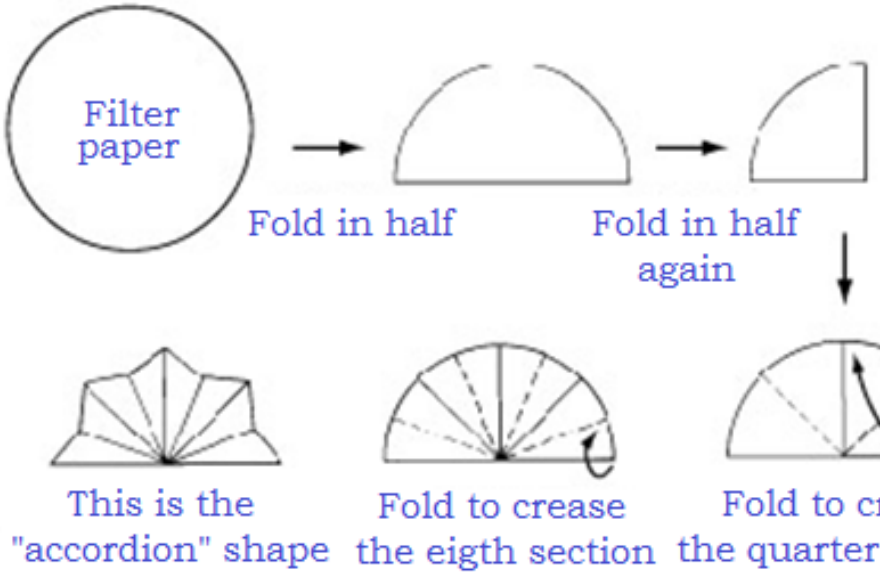
In this method we take the advantage that one compound in the mixture is readily soluble in a given solvent , whereas the remainder of the mixture may be relatively insoluble.

Preparation of filter paper



Filter paper cone





Name of experiment: **Solution and Filtration**

Aim of experiment: Separation of **Benzoic acid** from a mixture of benzoic acid & glucose

Procedure :

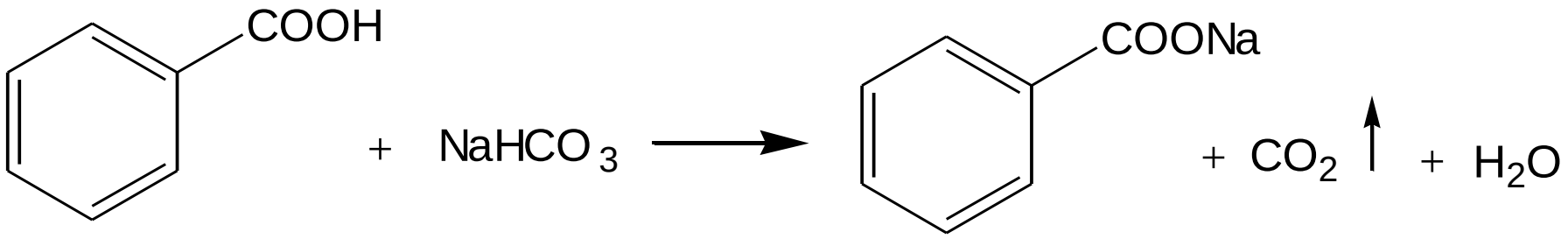
1- The impure mixture is to be dissolved in about 10 ml of distilled water. (*The sugar will dissolve in water , while benzoic acid remains precipitated*)

2- Then perform filtration , benzoic acid remains as the precipitate on the filter paper , while sugar goes with the filtrate as a solution.

Notes :

► **The sugar can be recovered from the filtrate by evaporating the solvent (water).**

► **To test that the precipitate (on the filter paper) is the benzoic acid, sodium bicarbonate solution is to be added on this precipitate. The benzoic acid will be dissolved due to the formation of soluble sodium benzoate and bubbles of the evolved CO_2 gas will be seen.**



Benzoic acid Sodium bicarbonate Sodium benzoate