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Determination of Solubility Class

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Solubility classification of organic cpd. gives an idea about:

- 1- The type of the functional group present in the cpd..**
- 2. The polarity & the molecular weight of the cpd., (Hydrocarbons are insoluble in water because of their non polar nature , and if an unknown cpd. is partially soluble in water this indicates that a polar functional group is present).**

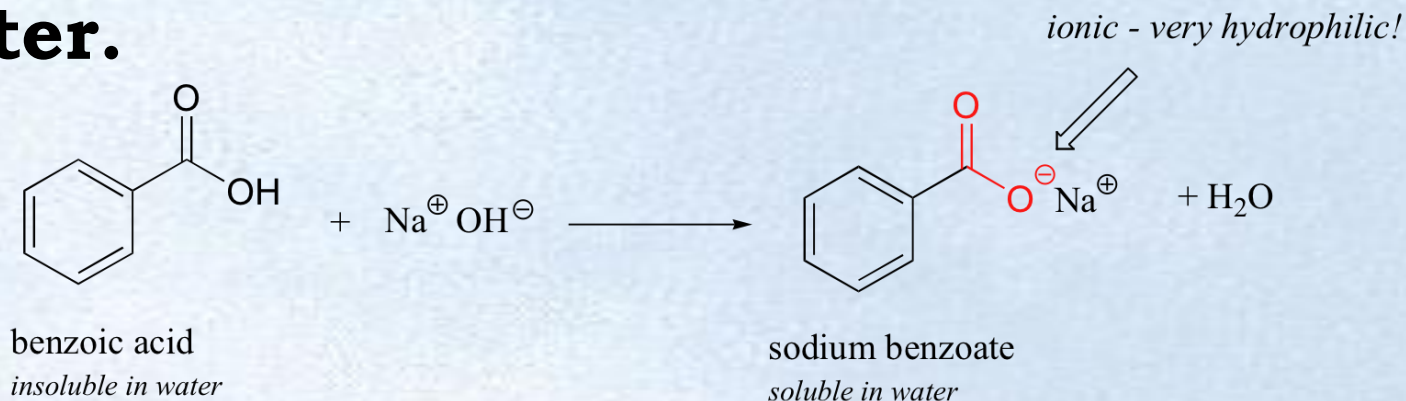
As the M.wt. increases, the water solubility decreases ($C \leq 5$ water soluble).
- 3. The nature of the compound (acidic, basic, neutral).**

Solubility Classification of organic compd.s is accomplished by testing the solubility of the compounds in the following solvents:

- Distilled water.**
- Ether.**
- 5% sodium hydroxide solution.**
- 5% sodium bicarbonate solution.**
- 5% hydrochloric acid solution.**
- Cold concentrated sulfuric acid.**

The solubility in certain solvents often leads to more specific information about the functional group.

For example: **Benzoic acid is insoluble in water , but it is converted by 5% NaOH solution to sodium benzoate salt which is readily soluble in water.**

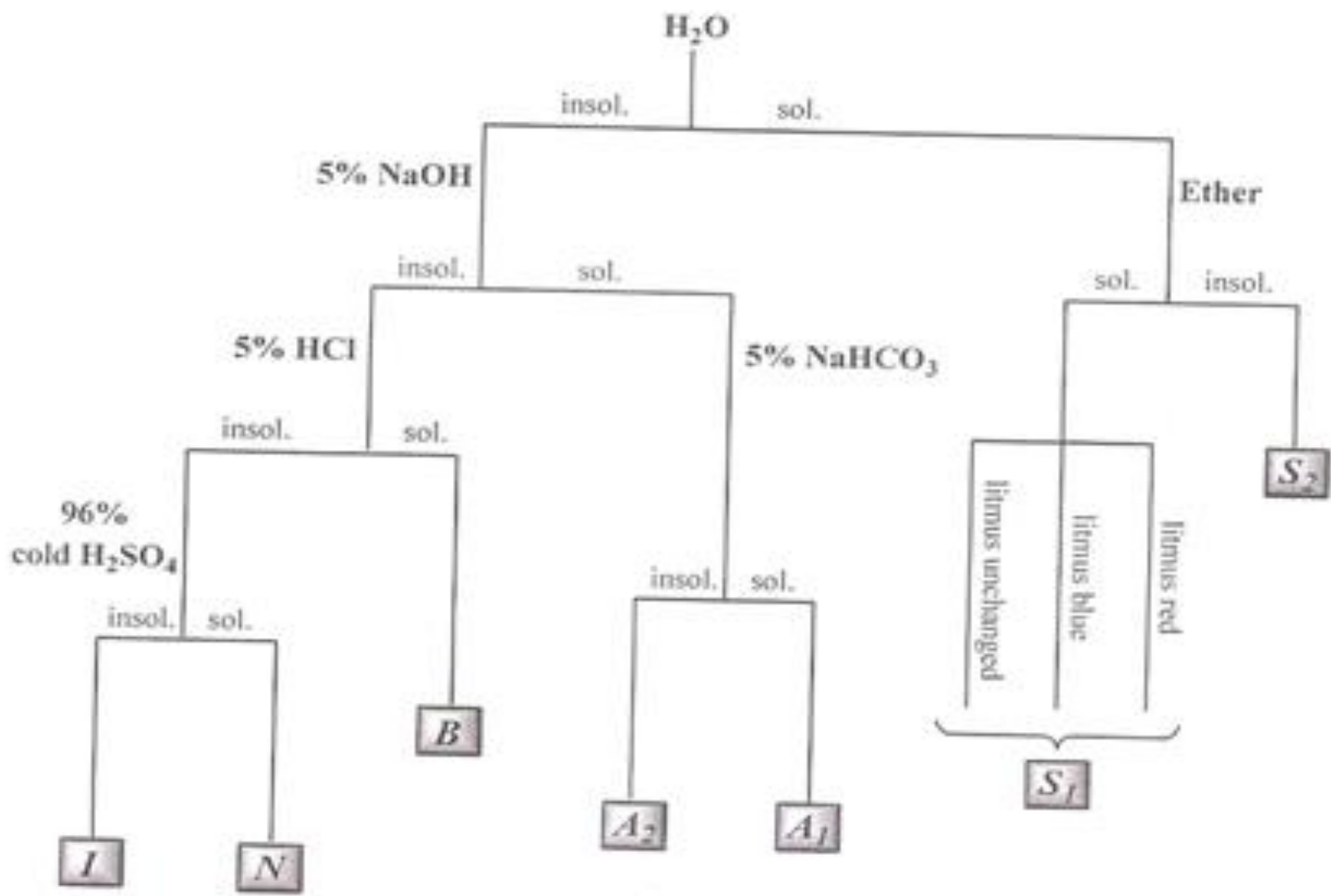


- When an unknown cpd. is insoluble in water and soluble in 5% NaOH sol. this indicates the presence of an acidic functional group.**

Generally, and for solubility classification purposes, the cpd. is said to be soluble in any solvent if it dissolves to the extent of about 3% (0.1gm/3 ml or 0.2 ml/3 ml).

- This is achieved by dissolving about 0.1 gm of solid or 3-4 drops of liquid organic cpd. in gradually increasing volumes of the solvent up to 3 ml (maximum allowed volume) with shaking.

- When the cpd. is more soluble in aqueous acid or aqueous base than in water, such increased solubility is the desired positive test for acidic or basic functional groups.



Determination of Solubility Class Flow Chart

Water

- **Water is a polar solvent with a dielectric constant equals to 80.**
- **It has the ability to form hydrogen bonding.**
- **It is amphoteric, it can act either as an acid or a base.**

Therefore it can dissolve:

- 1. Salts of ammonium ion (RNH_4^+) or organic acids salts with alkali metal cations (RCOO^-).**
- 2. Ionic compounds.**
- 3. Polar compounds “ like dissolves like ”.**
- 4. Organic cpd.s with low m.wt (carbon < 5) such as alcohols, carboxylic acids , aldehydes & ketones .**

Ether

- **Ether is a non polar solvent having a dielectric constant of 4.3 .**
- **It cannot form H-bond (unassociated liquid).**

It differs from water in that:

- **It can not dissolve ionic cpd.s such as salts.**
- **It is an organic solvent for non polar cpd.s (like dissolves like).**
- **It dissolves most water insoluble cpd.s ; therefore, in the determination of solubility class , the importance of ether is for water soluble cpd.s only & no further solubility tests using the remaining solvents are to be done.**

Accordingly, 2 probabilities are there:

1- Cpd.s soluble in both water & ether.

These cpd.s are :

- **Non ionic .**
- **Contain five or less carbon atoms.**
- **Contain an active group that is polar & can form H - bond.**
- **Contain only one strong polar group.**

This division of cpd.s is given **S1 class , it includes, e.g., aldehydes, ketones & aliphatic acids.**

2. Cpd.s soluble in water only (not in ether)

These cpd.s are:

- **Ionic.**
- **Contain two or more polar groups with no more than four carbon atoms per each polar group.**

This group is classified as **S2 class, it includes ionic salts such as salts of carboxylic acids & amines & cpd.s with more than one active group such as poly hydroxylated cpd.s & carbohydrates.**

Note: The solubility in ether is tested only for water soluble cpd.s. for water insoluble cpd.s use the left side of the scheme, i.e., test solubility in sodium hydroxide solution rather than ether.

5% NaOH and 5% NaHCO₃

- **Water insoluble cpd.s must be tested first in 5% NaOH solution which is a basic solvent.**

It reacts with water insoluble cpd.s that are capable of donating protons such as strong & weak acids.

The stronger the acid , the weaker the base it can react.

Water insoluble cpd.s that dissolve in 5% NaOH sol. must also be tested for solubility in 5% NaHCO₃ sol. Therefore, for water insoluble acidic cpd.s NaOH sol. considered as a **detecting solvent where as NaHCO₃ sol. is called as a **sub classifying** solvent since it can react with strong acids only.**

That is, these two solvents give an idea about the acidity degree of the compound.

• Note that testing solubility in 5% NaHCO₃ sol. is not needed if the cpd. is insoluble in 5% NaOH sol., but rather, 5% HCl sol. should be used.

There are two probabilities :

1. Cpd.s soluble in both bases.

This group is given class **A1 which includes , strong acids “ *which can react with weak bases* ”, carboxylic acids & phenols with electron withdrawing groups , e.g. -NO₂ “ protons are weakly attached & can be given easily ”.**

2. Cpd.s soluble in 5% NaOH sol. only.

This group is given class **A2** , it includes , phenols, amides & amino acids (weak acids).

5% HCl sol.

If the cpd. is insoluble in water & NaOH sol. , this mean it is not an acidic compound but rather it may be a basic, neutral or inert cpd.. 5% HCl sol. can dissolve basic cpd.s such as amines (RNH_2).

If the cpd. is soluble in this solvent, then it is given class **B**, it includes primary, secondary, & tertiary amines

Cold concentrated H_2SO_4

If the cpd. is insoluble in water, 5% NaOH sol. & 5% HCl sol. , solubility in cold conc. H_2SO_4 should be tested.

If the cpd. is soluble in this acid it belongs to class **N which includes , neutral cpd.s such as high m.wt. alcohols, aldehydes, ketones, esters & ethers (carbon atoms > 4) & unsaturated hydrocarbons.**

On the other hand, cpd.s that are insoluble in cold conc. H_2SO_4 belong to class **I , it includes inert aliphatic (saturated) hydrocarbons , aromatic hydrocarbons , haloalkanes & aryl halides.**



Questions & Exercises :

- 1- Water solubility test is the 1st test to run, explain why?**
- 2- Show by chemical equation , how can cold conc. H_2SO_4 dissolves oxygen containing neutral cpd.?**
- 3- Determine the solubility class & the nature of the following unknowns:**
 - (a) Unknown “ X ” is insoluble in water & gives bubbles with 5% NaHCO_3 .**
 - (b) Unknown “ Y ” is insoluble in water , insoluble in 5% NaOH , but soluble in 5% HCl .**



References

*Robert T. Morrison , Robert N. Boyd, **Organic Chemistry**, sixth edition.*

*Azhar M. Jasim , Duraid H. Mohammad ,
A Laboratory Manual on Practical Organic Chemistry for 2nd year students, University of Baghdad , College of Pharmacy, Department of Pharmaceutical Chemistry, 2012.*