

Qualitative Analysis of Organic Compounds

It helps identify & characterize unk. Org. compounds

Many org. compounds are usually a component of a mixture of several compounds that might be considered as impurities. These impurities may be : side products resulted during the preparation of the organic compound or may be decomposition products of the original pure organic compound and this occurs during storage under unsuitable conditions .

Some compounds may be obtained and stored pure because of their high degree of stability.

In most cases a good separation and purification should precede qualitative analysis of organic compounds so that identification will be successful.

The qualitative analysis of any organic compound (org. cpd.) should follow these steps:

1. Physical properties studying.

- State (solid, liquid, gas) • Determination of m.p. or b.p..
- Color , taste, & odor of the compound.
- Determination of the solubility group (solubility classification according to the general families).

2. Chemical properties studying.

- Effect of the compound or its solution on litmus paper.
- Determination of elements in the organic compound (nitrogen, sulfur, or halogens).
- Detection of the organic groups, i.e. group classification to get more specific families.
- Specific classification tests. •Preparation of derivatives.

Determination of Solubility Class

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Solubility Classification

- The solvents are:-
 - Distilled water.
 - Ether.
 - 5% sodium hydroxide solution.
 - 5% sodium bicarbonate solution.
 - 5% hydrochloric acid solution.
 - Cold conc. sulfuric acid.

Solubility Classification

The Aim

1. Type of functional group present in the cpd..

2. Polarity & the molecular weight of the cpd..

i.e.: Hydrocarbons are insoluble in water

non polar nature.

If an unknown cpd. is partially soluble in water,

a polar functional group is present .

As the M.wt. increase , the water solubility will decrease ($C \leq 5$ water soluble).

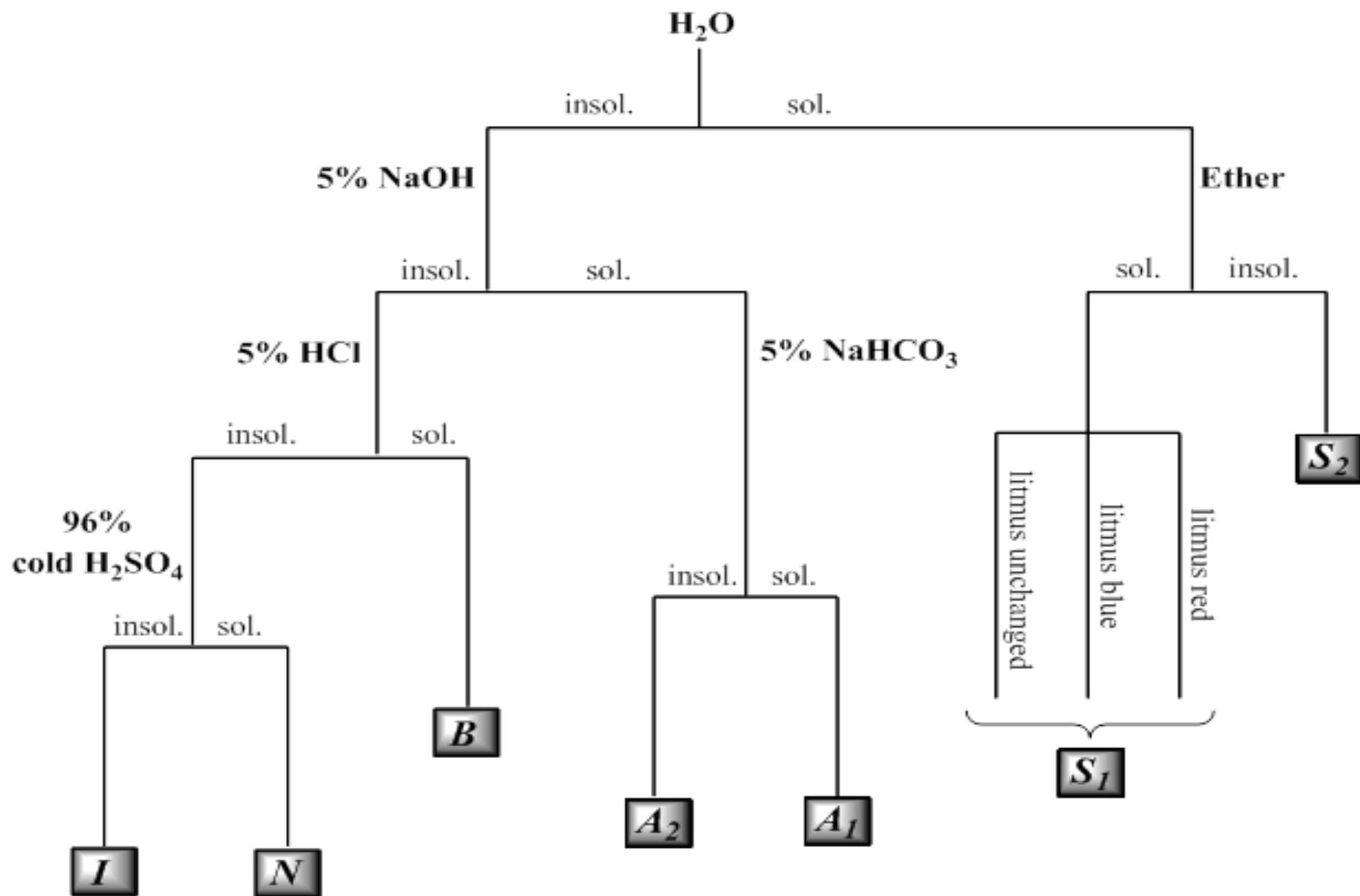
3. Nature of the compound (acidic, basic, neutral).

Solubility Classification

- 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 sol.** 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**.

Solubility Classification

- The compound (cpd.) is consider as soluble in any solvent if it dissolves to the extent of about **3% (0.1 gm/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 cpd. in **gradually increasing volumes** of the solvent up to **3 ml** (maximum allowed volume)**with shaking**.(How solid or liq. cpd. is soluble)
- When the cpd. is **more soluble in aqueous acid or aqueous base** than in **water**. Such increased solubility is the desired positive test (+ ve) for **acidic** or **basic** functional groups.



Solvents

Water:

- it is a polar solvent with dielectric constant equals to 80.
- it has the ability to form hydrogen bond
- can act either as an acid or a base (Amphoteric).

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 compounds with low molecular weight (less than 5 carbon atoms) such as alcohols, aldehydes, ketones, and carboxylic acids.

Solubility Classification

Ether:

- is a **non polar** solvent having a dielectric constant of 4.3.
- It cannot form hydrogen bond (**un associated liquid**).
- It is an organic solvent (like dissolve like)
- Therefore it **differs** from **water** in that it **cannot dissolve ionic compounds such as salts**.
- It dissolves most **water insoluble compounds**; therefore in the determination of solubility class, **the importance of ether** is for **water soluble compounds only** and **no further solubility tests using the remaining solvents are to be done**.

Solubility Classification

- Accordingly two probabilities are there:

1. Compounds soluble in both water and ether.

- 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 compounds is given **S1** class and includes, e.g., **aldehydes, ketones, and aliphatic acids.**

2. Compounds soluble in water only but not in ether.

- 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 and includes ionic salts such as salts of carboxylic acids and amines and compounds with more than one active group such as poly hydroxylated compounds and carbohydrates.

Solubility Classification

- Note: solubility in ether is tested only for water soluble compounds, for water insoluble compounds 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 compounds must be tested first in 5% sodium hydroxide solution which is a basic solvent. It reacts with water insoluble compounds that are capable of donating protons such as strong and weak acids.

Solubility Classification

- Water insoluble compounds that dissolve in 5% NaOH sol. must also be tested for solubility in 5% NaHCO₃ sol. Therefore, for water insoluble acidic compounds NaOH sol. Considered as a detecting solvent where as NaHCO₃ sol. Is called as a subclassifying 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 compound is insoluble in 5% NaOH sol., but rather, 5% HCl sol. should be used.

Solubility Classification

- Two probabilities are there:
 1. Compounds soluble in both bases: this group is given class **A1** which includes **strong acids** that have the ability to react with weak bases: **carboxylic acids** and **phenols with electron withdrawing groups (e.g. $-\text{NO}_2$)** protons are weakly attached and can be given easily.
 2. Compounds soluble in 5% NaOH sol. only : this group is given class **A2** and it includes **phenols, amides, and amino acids (weak acids)**.

Solubility Classification

- 5% HCl sol.

If the compound is insoluble in: **water** and **NaOH sol.** , this mean it is **not an acidic compound** but it is either a **basic or a neutral or inert compound**.

5% HCl sol. can dissolve **basic** compounds such as amines (RNH_2). If the compound is soluble in this solvent, then it is given class **B** which includes primary, secondary, and tertiary amines.

Solubility Classification

- Cold concentrated H₂SO₄

If the compound is insoluble in: water, 5% NaOH sol., and 5% HCl sol., solubility in cold conc. H₂SO₄ should be tested. If it is soluble in this acid it belongs to class **N** which includes neutral compounds such as high m.wt. alcohols, aldehydes, ketones, esters & ethers (more than four carbon atoms), & unsaturated hydrocarbons.

But if it is insoluble in cold conc. H₂SO₄ belong to class **I** which includes inert aliphatic (saturated) hydrocarbons, aromatic hydrocarbons, haloalkanes, and aryl halides.