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اسم المادة باللغة العربية: الكيمياء العضوية

اسم المادة باللغة الإنكليزية: **Organic Chemistry**

المرحلة: الثانية

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عنوان المحاضرة باللغة العربية: الكيمياء العضوية العملي التجربة الثانية

عنوان المحاضرة باللغة الإنكليزية: **Practical Organic Chemistry(2)**

Exp. 2

Qualitative Analysis of Organic Compounds

Qualitative analysis of organic compounds helps identify and characterize unknown organic compounds. Many organic 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. On the other hand, 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 should follow these steps:

A- Physical properties studying.
1- State of the organic compound (solid, liquid, gas).
2- Determination of the melting point or boiling point.
3- Color, taste, and odor of the compound.
4- Determination of the solubility group (solubility classification according to the general families).
B- Chemical properties studying.
1- Effect of the compound or its solution on litmus paper.
2- Determination of elements in the organic compound (nitrogen, sulfur, or halogens).
3- Detecting of the organic groups, i.e. group classification to get more specific families.
4- Specific classification tests.
5- Preparation of derivatives.

Determination of Solubility Class

Solubility class determination gives an idea about the type of the functional group present in the compound, the polarity and molecular weight of the compound, and the nature of the compound (acidic, basic, neutral). This is accomplished by testing the solubility of the compound in either of the following sets of solvents: distilled water, 5% sodium hydroxide solution, 5% sodium bicarbonate solution, 5% hydrochloric acid solution, and cold concentrated sulfuric acid, or distilled water and ether.

It is well known that the hydrocarbons are insoluble in water because of their non polar nature. If an unknown compound is partially soluble in water, then this indicates that a polar functional group is present. Additionally, solubility in certain solvents often leads to more specific information about the functional group. For example, benzoic acid is insoluble in water, but is converted by 5% sodium hydroxide solution to a salt, sodium benzoate, which is readily water soluble. In this case, then, the solubility in 5% sodium hydroxide solution of a water insoluble unknown is a strong indication of an acidic functional group. Prediction of the molecular weight and size may sometimes be obtained from the result of solubility tests. For example, in many homologous series of monofunctional compounds, the members with fewer than about five carbon atoms are water soluble, whereas the higher homologs are insoluble.

The first step to follow is to test the solubility of the compound in water. Generally and for solubility classification purposes, the compound is said to be 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 the solid compound or 3-4 drops of the liquid compound in gradually increasing volumes of the solvent up to 3 ml (max. allowed volume is 3 ml) with shaking. This technique is the one that should be followed in solubility classification to determine whether the compound is soluble or insoluble in that solvent.

When solubility in dilute acid or dilute base is being considered, the significant observation to be made is whether it is significantly 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. Below is very useful scheme for solubility classification.

Solvents

*** Water**

Water is a polar solvent with a dielectric constant equals to 80. It has the ability to form hydrogen bonding and can act either as an acid or a base. Therefore it can dissolve:

- Salts of ammonium ion (RNH_4^+) or organic acids salts with alkali metal cations ($RCOO^-$).
- Ionic compounds.
- Polar compounds "like dissolve like"
- Organic compounds with low molecular weight (less than 5 carbon atoms) such as alcohols, aldehydes, ketones, and carboxylic acids.

Water is useful to determine the degree of acidity of a compound, even if the compound is insoluble in water, using litmus paper (acidic, basic, or neutral).

Water is the first solvent used to determine the solubility class of a compound. If the compound is water soluble, the next step is to test its solubility in ether.

*** Ether**

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

Accordingly two probabilities are there:

1- *Compounds soluble in both water and ether.*

These compounds:

- non ionic.
- contain five or less carbon atoms.
- contain an active group that is polar and can form hydrogen bonding.
- contain only one strong polar group.

This division of compounds is given $\boldsymbol{S_1}$ class and includes, e.g., aldehydes, ketones, and aliphatic acids.

2- *Compounds soluble in water only* (but not in ether).

These compounds:

- ionic.
- contain two or more polar group with no more than four carbon atoms per each polar group.

This group is classified as $\boldsymbol{S_2}$ 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.

Not that solubility in ether is tested only for water-soluble compounds. For water insoluble compounds use the left side of the solubility classification scheme, i. e. test solubility in sodium hydroxide rather than ether.

*** 5% NaOH and 5% $NaHCO_3$**

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. The stronger the acid, the weaker the base it can react with. Water insoluble compounds that dissolve in 5% sodium hydroxide solution must also be tested for solubility in 5% sodium bicarbonate solution. Therefore, for water insoluble acidic compounds sodium hydroxide solution is considered as a *detecting solvent* whereas sodium bicarbonate solution is called as a *sub classifying solvent* since it can react with strong acidic only. That is, these two solvents gives an idea about the acidity degree of the compound. Note that testing solubility in 5% sodium bicarbonate solution is not needed if the compound is insoluble in 5% sodium hydroxide solution, but rather, 5% hydrochloric acid solution should be used.

Two probabilities are there:

1- *Compounds soluble in both bases.*

This group is given class $\boldsymbol{A_1}$. This class includes strong acids that have the ability to react with weak bases (carboxylic acids) and phenols with electron withdrawing groups (e. g., $-NO_2$). Protons are weakly attached and can be given easily.

2- *Compounds soluble in 5% sodium hydroxide solution only.*

This group is given class $\boldsymbol{A_2}$ and it includes phenols, amides, and amino acids.

*** 5% HCl**

If the compound is insoluble in water and sodium hydroxide solution (and, hence, insoluble in sodium bicarbonate solution too), this means that the compound is not an acid but, rather, is either a basic compound or a neutral compound. 5% hydrochloric acid solution, which can dissolve basic compounds such as amines (RNH_2), is used for such a compound. If the compound is soluble in this solvent, then it is given class ***B***. This class include primary, secondary, and tertiary amines.

*** Cold concentrated H_2SO_4**

If the compound is insoluble in water, 5% sodium hydroxide solution, and 5% hydrochloric acid solution, solubility in cold concentrated sulfuric acid should be tested. If the compound is soluble in this acid, it belongs to class ***N*** which include neutral compounds such as high molecular weight alcohols, aldehydes, ketones, esters, and ethers, (more than four carbon atoms), and unsaturated hydrocarbons. On the other hand, compounds that are insoluble in cold concentrated sulfuric acid belong to class ***I*** which includes inert aliphatic hydrocarbons.