جامعة الانبار

كلية : الصيدلة

قسم : الكيمياء الصيدلانية

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 الاولى

عنوان المحاضرة باللغة الإنكليزية: **Practical Organic Chemistry (1)**

محتوى المحاضرة
Elemental Analysis (1)

Elemental analysis is considered as an important step in the identification of organic compounds. The chief element in the organic compounds is carbon, hydrogen and oxygen for which we do not employ chemical tests. Next to them in order of importance are nitrogen, halogens (chlorine, bromine, fluorine and iodine), and sulfur that can be detected by reaction with sodium metal. The method is known as sodium fusion method.

The nonpolar nature of organic compounds makes the detection of N, S, and X difficult because organic compounds do not ionize in solution to give ions of these elements. For this reason it is necessary to convert these elements into inorganic ions before doing the tests. The conversion is accomplished by heating a small quantity of the organic compound with an equal quantity of metallic sodium. The organic compound will decompose and soluble sodium salts of the elements will be formed.

$$\text{C, H, O, N, X, S} + \text{Na} \xrightarrow{\Delta} \text{NaCN, } Na_2S\text{, NaOH, NaX, NaSCN}$$

Sodium thiocyanide, NaSCN is produced when both sulfur and nitrogen are present in the same organic compound only when the quantity of sodium element is small.

Sodium element is very reactive and react with water vigorously resulting in explosion. So extreme care should be taken during the handling the metal.

$$\text{Na} + H_2O \rightarrow \text{NaOH}$$

Therefore sodium element is kept dipped in liquid paraffin to prevent exposure to moisture. The paraffin should be wiped off before using the sodium. It is also advised not to touch it directly by hands since hands are usually moist, resulting in burning sensation. Not that sodium is a shiny element and when it is exposed to air and moisture it is oxidized and become non shiny.

Procedure

A small quantity of the unknown is placed in a clean dry test tube together with a small piece of sodium metal. The test tube is held vertically by a clamp. The lower part of the test tube is heated gradually until the sodium melts and its vapors fill the lower part of the tube. This gradual heating is to prevent the loss of the products as vapors. Heating is then continued for additional five minutes until the bottom of the test tube becomes red. Cautiously drop the still hot test tube into a beaker containing about 20 ml of distilled water. The tube will break down and, if not, use a glass rode to break it. The resulting solution is heated almost to boiling and filtered. The filtrate, which should be colorless, is used for the specific tests.

To remove the excess unreacted sodium add a small quantity of alcohol (ethanol or methanol) to the test tube before breaking it with heating so that the alcohol will react with the excess sodium metals to give sodium alkoxide.

$$\underset{\text{sodium}}{\text{Na}} + \underset{\text{ethanol}}{CH_3CH_2OH} \rightarrow \underset{\text{sodium ethoxide}}{NaOCH_2CH_3}$$

1- Detection of Nitrogen

To 3 ml of the filtrate add 4 drops (0.2 gm) of ferrous sulfate $FeSO_4$ solution. Check the basicity of the solution and make it basic by the addition of enough sodium or potassium hydroxide solution (10%). Heat for boiling (30 second). Now add drops of dilute sulfuric acid enough to make the solution acidic. A Prussian blue precipitate indicates a positive test of nitrogen.

basic

$$2NaCN + FeSO_4 \xrightarrow[NaOH]{} Fe(CN)_2 + Na_2SO_4$$

$$Fe(CN)_2 + 4NaCN \rightarrow \underset{\text{sodium ferrocyanide}}{Na_4Fe(CN)_2}$$

2- Detection of Sulfur

Acidify 2 ml of the filtrate with dilute acetic acid. Then add 5 drops of lead acetate solution. A black precipitate of lead sulfide indicates the presence of sulfer.

$$Na_2S + (CH_3COO)_2Pb \xrightarrow{\text{dil. } CH_3COOH} \underset{\text{black ppt.}}{PbS\downarrow} + 2CH_3COONa$$

Acetic acid is used in the acidification and not other acids (H_2SO_4, HCl, HNO_3) since they give insoluble white precipitate through reaction with lead acetate.

$$H_2SO_4 \rightarrow PbSO_4$$

$$HCl \text{ or } HNO_3 + (CH_3COO)_2Pb \rightarrow PbCl_2 \text{ or } Pb(NO_3)_2 \downarrow$$

3- Detection of Halogens

In case of presence of nitrogen and sulfur in the compound, acidify 3 ml of the filtrate with dilute nitric acid (add drop by drop until the solution becomes acidic). Boil for 5 minutes and then add drops of silver nitrate. White or yellow precipitate indicates the presence of halogens.

$$NaX + AgNO_3 \xrightarrow{HNO_3} AgX$$

AgCl white ppt.
AgBr white ppt.
AgI yellow ppt ↓

Boiling for 5 minutes is done to remove nitrogen and sulfur present in the filtrate as hydrogen cyanide and hydrogen sulfide gases.