Element analysis

element analysis is considered as an important step in the identification of organic compounds. the chief elements making up in organic compounds are 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.

Sodium Fusion Method

This method is used for detection of nitrogen, sulfur and halogens (N, S and X). 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 element 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.

$$\mathbf{C, H, O, N, X, S + Na \xrightarrow[\text{fusion}]{\triangle} NaCN, Na_2S, NaOH, NaX, NaSCN}$$

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.

$$\mathbf{NaSCN + 2Na \longrightarrow NaCN + Na_2S}$$

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 will a small piece of sodium metal. The test tube is held vertically by a clamp. The lower part of the test tube is heated gradual until the sodium melts and its vapours fill the lower part of the tube .this gradual heating is to prevent the loss of the products as vapours .heating is then continued for additional five minutes until the bottom of test tube becomes red . cautiously drop the sill hot test tube in to a beaker containing about 20 ml of distilled water. The tube will break down and ,if not , use a glass rode to break ik . the resulting solution is heated almost to boiling and filtrate , which should be colourless ,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 the excess sodium metal to give sodium alkoxide.

$$Na + CH_3CH_2OH \longrightarrow NaOCH_2CH_3$$

ethanol **sodium ethoxide**

1- Detection of Nitrogen.

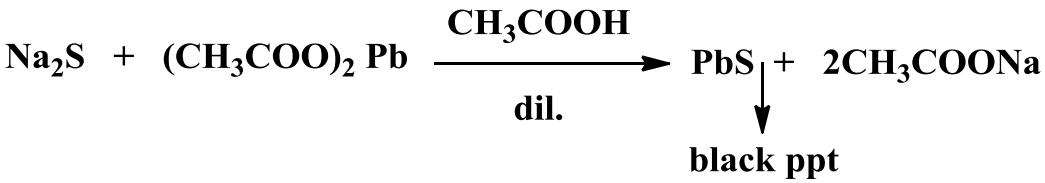
To 3 ml of the filtrate add 4 drops (0.2 gm) of ferrous sulfate solution. Check the basicity of the solution and make it basic by the addition of enough sodium or potassium hydroxide solution (10%). Heat for boiling (30sec.). New add drops of dilute sulfuric acid enough to make the solution acidic. A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

$$FeSO_4 + 6NaCN \longrightarrow Na_4[Fe(CN)_6] + Na_2SO_4$$

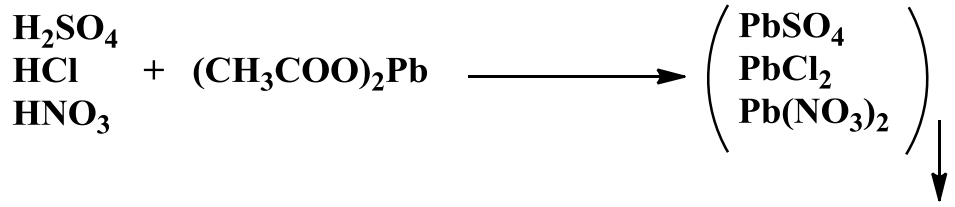
$$3Na_4[Fe(CN)_6] + 2Fe_2(SO_4)_3 \longrightarrow Fe_4[Fe(CN)_6]_3 + 6Na_2SO_4$$

2- Detection of Sulfur.

A- 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 sulfur.

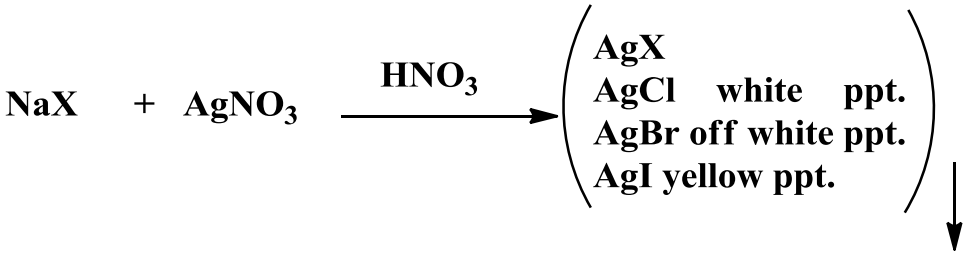


B- Acetic acid is used in the acidification and not other acids (sulfuric, hydrochloric, nitric) since they give insoluble white precipitate through reaction with lead acetate.

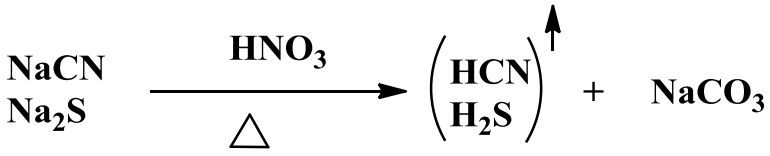


3- Detection of Halogens.

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.



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



Question

1- Explain how could you prove specifically the presence of chloride, bromide, or iodide ions in a solution containing halide ions.
2- Why should not you use tap water in elemental analysis?
3- For any reason the sodium fusion method used and why?
4- Do sodium element is kept in liquid paraffin?
5- How you can determine the nitrogen