Chemistry Study Materials for Class 11
(NCERT Based Notes of Chapter- 12)
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SOME BASIC PRINCIPLES AND TECHNIQUES

Detection of Nitrogen, Sulphur and Halogens

Nitrogen, sulphur and halogens present in an organic compound are detected by "***Lassaigne's test***". Here the organic compound is fused with metallic sodium in a fusion tube. It is then plunged into distilled water taken in a china dish. The solution is boiled and filtered. The filtrate is known as sodium fusion extract.

Principle: In an organic compound, nitrogen, sulphur and halogen atoms are present in covalent form. By heating with metallic sodium, these elements are converted to ionic form as follows:

$Na + C + N_2 \xrightarrow{\Delta} NaCN$

$2Na + S \xrightarrow{\Delta} Na_2S$

$Na + X_2 \xrightarrow{\Delta} NaX$ (X = Cl, Br or I)

For the detection of the elements, the following tests are done:

No.	Experiment	Observation	Inference
1.	To one part of sodium fusion extract add freshly prepared ferrous sulphate ($FeSO_4$) solution. Heated to boiling, cooled and acidified with dil. H_2SO_4.	Blue or green coloration or precipitate (ppt)	Presence of nitrogen
2.	A little of the sodium fusion extract is acidified with dil. HNO_3 and then silver nitrate ($AgNO_3$) is added.	White ppt soluble in ammonium hydroxide (NH_4OH)	Presence of Chlorine
		Pale yellow ppt slightly soluble in NH_4OH	Presence of Bromine
		Yellow ppt insoluble in NH_4OH	Presence of Iodine
3.	To a little of the sodium fusion extract, add sodium nitroprusside solution	Violet colouration	Presence of sulphur

Test for Phosphorus

The organic compound is heated with an oxidising agent like sodium peroxide. The phosphorus present in the compound is oxidised to phosphate. The solution is boiled with nitric acid and then treated with ammonium molybdate. A yellow colouration or precipitate indicates the presence of phosphorus.

QUANTITATIVE ANALYSIS OF ORGANIC COMPOUNDS

The percentage composition of elements present in an organic compound is determined by the following methods:

1. Estimation of Carbon and Hydrogen

Carbon and hydrogen are estimated by ***Liebig's combustion method***. In this method, a known mass of an organic compound is burnt in the presence of excess of oxygen and copper (II) oxide. Then carbon is oxidised to CO_2 and hydrogen is oxidised to H_2O.

$$C_xH_y + (x + y/4)\ O_2 \longrightarrow x\ CO_2 + (y/2)\ H_2O$$

The water so produced is absorbed in a weighed U-tube containing anhydrous calcium chloride and carbon dioxide is absorbed in another U-tube containing concentrated solution of potassium hydroxide. These tubes are connected in series. The increase in masses of calcium chloride and potassium hydroxide gives the amounts of water and carbon dioxide from which the percentages of carbon and hydrogen are calculated.

Calculations:

Let the mass of organic compound be m g, mass of water and carbon dioxide produced be m_1 and m_2 g respectively.

$$\text{Percentage of hydrogen} = \frac{2 \times m_1 \times 100}{18 \times m}$$

$$\text{Percentage of carbon} = \frac{12 \times m_2 \times 100}{44 \times m}\ \%$$

2. Estimation of Nitrogen

There are two methods for estimation of nitrogen:

(i) Dumas method and

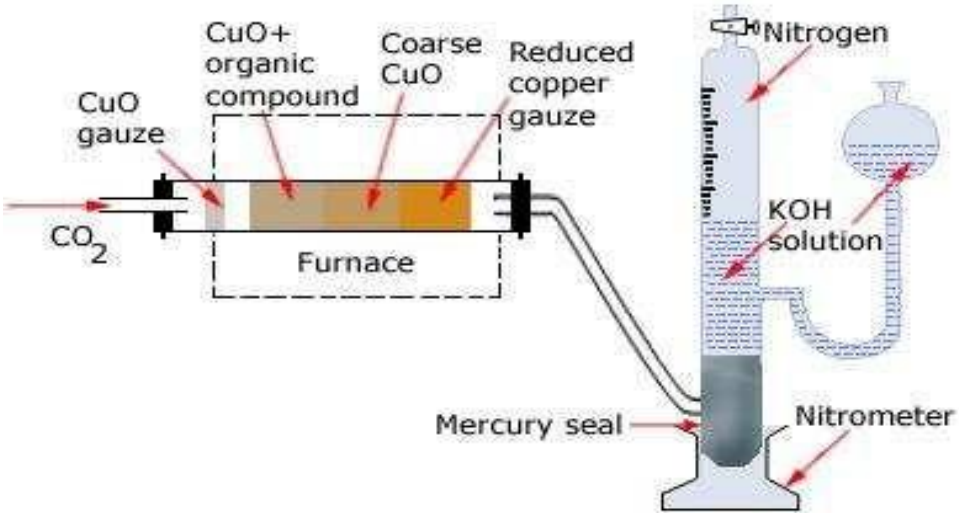
(ii) Kjeldahl's method.

i) Dumas method:

Here the organic compound is heated with copper oxide in an atmosphere of carbon dioxide so that free nitrogen, carbon dioxide and water are produced.

$$C_xH_yN_z + (2x + y/_2)\ CuO \longrightarrow x\ CO_2 + y/_2\ H_2O + z/_2\ N_2 + (2x + y/_2)\ Cu$$

This mixture of gases is collected over an aqueous solution of potassium hydroxide which absorbs carbon dioxide. Nitrogen is collected in the upper part of the graduated tube



Calculations:

Let the mass of organic compound = m g

Volume of nitrogen collected = V_1 ml

Room temperature = T_1 K

Volume of nitrogen at STP = $\frac{P_1V_1 \times 273}{760 \times T_1}$ = V ml

Where P_1 and V_1 are the pressure and volume of nitrogen gas

P_1 = Atmospheric pressure – Aqueous tension

We know that 22400 ml N_2 at STP weighs 28 g.

Therefore, V ml N_2 at STP weighs = $\frac{28 \times V}{22400}$ g

Percentage of nitrogen = $\frac{28 \times V \times 100}{22400 \times m}$ %

3. Estimation of halogens (Carius method):

Here a known mass of an organic compound is heated with fuming nitric acid in the presence of silver nitrate contained in a hard glass tube known as Carius tube, in a furnace.

Carbon and hydrogen present in the compound are oxidised to carbon dioxide and water. The halogen present forms the corresponding silver halide (AgX). It is filtered, washed, dried and weighed.

Calculations:

Let the mass of organic compound taken = m g Mass of AgX formed = m_1 g

1 mol of AgX contains 1 mol of halogen

$$\text{Mass of halogen in } m_1 \text{ g of AgX} = \frac{\text{Atomic mass of halogen} \times m_1 \text{ g}}{\text{Molecular mass of AgX}}$$

$$\text{Percentage of halogen} = \frac{\text{Atomic mass of halogen} \times m_1 \times 100}{\text{Molecular mass of AgX} \times m} \%$$

4. Estimation of Sulphur (Carius method):

A known mass of an organic compound is heated in a Carius tube with sodium peroxide or fuming nitric acid. Sulphur present in the compound is oxidised to sulphuric acid. It is precipitated as barium sulphate by adding excess of barium chloride solution. The precipitate is filtered, washed, dried and weighed. The percentage of sulphur can be calculated from the mass of barium sulphate ($BaSO_4$).

Calculations:

Let the mass of organic compound taken = m g and the mass of barium sulphate formed

= m_1 g 1 mol of $BaSO_4$

= 233 g $BaSO_4$

= 32 g sulphur

m_1 g $BaSO_4$ contains $\frac{32 \times m_1}{233}$ g sulphur

$$\text{Percentage of sulphur} = \frac{32 \times m_1 \times 100}{233 \times m} \%$$

5. Estimation of Phosphorus

A known mass of an organic compound is heated with fuming nitric acid. Phosphorus present in the compound is oxidised to phosphoric acid. It is precipitated as ammonium phosphomolybdate [$(NH_4)_3PO_4.12MoO_3$] by adding ammonia and ammonium molybdate.

Calculations:

Let the mass of organic compound taken = m g and mass of ammonium phosphomolybdate = m_1g Molar mass of $(NH_4)_3PO_4.12MoO_3$ = 1877 g

Percentage of phosphorus = $\frac{31 \times m_1 \times 100}{1877 \times m}$ %

6. **Estimation of Oxygen**

The percentage of oxygen in an organic compound is usually found by difference between the total percentage composition (100) and the sum of the percentages of all other elements.

i.e. percentage of oxygen = 100 – sum of the percentage of all the other elements.

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