EXPERIMENT-10

To detect the presence of extra elements (nitrogen, sulphur and halogens) in an organic compound.

10.1 OBJECTIVES

After performing this experiment, you should be able to

- *detect the presence of nitrogen, sulphur and halogens (extra elements) present in a given organic compound.*
- *explain the reason for the necessity of fusing an organic compound with sodium metal in the detection of extra elements, and*
- *write chemical equations of various reactions taking place in this experiment.*

10.2 WHAT YOU SHOULD KNOW

The determination of structure of an unknown organic compound requires the detection of the elements present in it. Generally all organic compounds contain carbon and hydrogen. In addition to these elements, they may also contain oxygen, nitrogen, sulphur and helium. The elements nitrogen, sulphur and halogens are known as extra elements.

For detecting the presence of elements in a compound, these have to be converted into ionic forms. This is done by fusing the compound with sodium metal. The elements, nitrogen, sulphur and halogens present in the compound are converted into soluble sodium salts according to the following equations.

$$Na + C + N \longrightarrow NaCN$$

$$2\,Na + S \longrightarrow Na_2S$$

$$Na + X \longrightarrow NaX$$

(where X is Cl, Br or I)

If a compound contains both nitrogen and sulphur, sodium thiocyanate is formed on fusing with sodium. The soluble sodium salts are extracted with water. The extract is usually known as sodium fusion extract.

10.2.1 Test for Nitrogen

Nitrogen is present as sodium cyanide in sodium fusion extract. Sodium cyanide is converted to sodium ferrocyanide on treating with ferrous sulphate. On further treating it with ferric chloride, a prussian blue complex, ferricferrocyanide is formed.

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

$$Na_4[Fe(CN)_6] + 4FeCl_3 \longrightarrow Fe_4[Fe(CN)_6]_3 + 12\,NaCl$$

Ferricferrocyanide
(Prussian blue)

20.2.2 Test for Sulphur

(i) Sulphur is present in sodium extract as sodium sulphide. It gives a purple colour with sodium nitroprusside due to the formation of sodium thionitroprusside.

$$Na_2S + Na_2[Fe(CN)_5NO] \longrightarrow Na_4[Fe(CN)_5NOS]$$

Sodium nitroprusside — Sodium thionitroprusside (Purple)

(ii) A sulphide reacts with lead acetate to yield lead sulphide as a black precipitate.

$$Na_2S + Pb(CH_3COO)_2 \longrightarrow PbS + 2CH_3COONa$$

Lead sulphide (black)

(iii) A sulphide reacts with silver nitrate to yield silver sulphide which is black in colour.

$$Na_2S + 2\,AgNO_3 \longrightarrow Ag_2S + 2NaNO_3$$

Silver Sulphide (black)

10.2.3 Test for Nitrogen and Sulphur when present together

When both nitrogen and sulphur are present together in an organic compound, sodium thiocyanate is produced during sodium fusion. Sodium thiocyanate gives blood red colour with ferric chloride.

$$NaCNS + FeCl_3 \longrightarrow [Fe(CNS)]^{2+} + Na^+ + 3Cl^-$$

(Blood red)

10.2.4 Test for Halogens

Halogens, present as halides in sodium extract, are tested as follows;

(i) **Silver Nitrate test :** Chlorides give white precipitate of silver chloride with silver nitrate. The precipitate is soluble in ammonium hydroxide.

$$NaCl + AgNO_3 \longrightarrow AgCl + NaNO_3$$

$$AgCl + 2NH_4OH \longrightarrow [Ag(NH_3)_2]Cl + 2H_2O$$

Bromides yield yellow precipitate, sparingly soluble in ammonium hydroxide. Iodides yield dark yellow precipitate, insoluble in ammonium hydroxide.

(ii) When a solution containing bromide or iodide is treated with chlorine water, the bromide and iodide are oxidised to the corresponding halogens. Being covalent, these halogens get dissolved in carbon tetrachloride CCl_4. Bromine imparts yellow to brown colour and iodine imparts purple colour in CCl_4 layer.

$$2NaBr + Cl_2 \longrightarrow 2NaCl + Br_2$$

$$2NaI + Cl_2 \longrightarrow 2NaCl + I_2$$

Chlorine being more electronegative, displaces bromides and iodides from solution.

10.3 MATERIALS REQUIRED

(1) Apparatus	(2) Chemicals
Test tubes - 6, Test-tube stand, Test tube holder, Ignition tubes - 3, Spatula, Watch-glass, Filter paper, Pair of tongs, Porcelain dish, Funnel, Glass rod, Dropper, Wire gauze.	Sodium metal, Ferrous sulphate, Ferric chloride, Silver nitrate, Ammonium hydroxide, Chlorine water, Carbon tetrachloride, Nitric acid, Sodium thiocyanate

10.4 HOW TO PERFORM THE EXPERIMENT

The experiment consists of two stages, (i) preparation of sodium fusion extract and (ii) to test for individual elements.

(i) Preparation of Sodium Fusion Extract.

Take out a small piece of sodium metal from a petri dish with the help of pair of forcepts. Dry the sodium piece between the folds of filter papers. Put a small piece of sodium metal into a clean and dry ignition tube. Hold the tube with a pair of tongs and heat it gently over the flame so that sodium melts and makes a stirring ball. Remove the ignition tube away from the flame and immediately add a small amount of solid organic compound into it. Heat the tube to red hot. Again add a little of the compound into the tube and heat it again to red hot. Plunge the red hot ignition tube in about 10 ml of distilled water taken in a porcelain dish. Immediately cover the porcelain dish with a wire gauze to avoid bumping of any unreacted sodium piece. Repeat the process of sodium fusion using two more ignition tubes. Break the tubes into small pieces with a clean glass rod. Boil the mixture for about 2 -3 minutes and filter.

(In case of liquid compounds, cover the molten sodium with solid sodium carbonate and then add the compound with a dropper).

Now proceed systematically and perform the following tests with the given sample (s), of organic compounds and identify the extra elements present in them. Record your observations and inferences in the table 10.1 in the following manner.

S.No.	Experiment	Observations	Inference
1.	**Test for nitrogen** Take about 1 ml of sodium fusion extract in a test tube and add a pinch of ferrous sulphate. Shake the contents and acidify with dilute hydrochloric acid. Boil the contents add a few drops of ferric chloride solution.	Prussian blue or green precipitate or colour	Nitrogen present
2.	**Test for sulphur**		
	(i) Take 1 ml of sodium fusion extract in a test tube and add a few drops of sodium nitropruside to it and shake.	Purple colour	Sulphur present
	(ii) Take 1 ml of sodium fusion extract in a test tube and acidify it with acetic acid. Add a few drops (4-5) of lead acetate solution to it.	Black precipitate	Sulphur present
3.	**Nitrogen and Sulphur present together**		
	Take about 1 ml of sodium fusion extract in a test tube and acidify it with dilute hydrochloric acid. Add a few drops of ferric chloride solution.	Blood red Colour	Nitrogen and Sulphur present
4.	**Test for Halogens**		
	(i) Take about 1 ml of sodium fusion extract in a test tube and acidify it with dilute nitric acid. Add to it about 2 ml of silver nitrate solution. Observe for the appearance for any precipitate and its colour.	(a) A curdy white precipitate soluble in NH_4OH	Chlorine present
	Add 2-3 ml of ammonium hydroxide to the precipitate and shake the contents. Observe the solubility of precipitate	(b) A light yellow precipitate partially soluble in NH_4OH	Bromine present

		(c) A pale yellow precipitate insoluble in NH_4OH	Iodine present
(ii)	If chloride is absent, perform the following test for bromide and iodide. Take 2 ml of sodium fusion extract in a test tube and acidify it with dilute hydrochloric acid. Add 1 ml of carbon tetrachloride. Being denser, CCl_4 forms the lower layer. Add excess of chlorine water and shake the mixture vigrously. Observe for appearance of any colour in CCl_4 layer.	(a) Orange Colour (b) Violet Colour	Bromine presence Iodine presence

10.5 PRECAUTIONS

1. Sodium metal is highly reactive. When exposed to air, it reacts even with the moisture present in the atmosphere. It also reacts with the sweat of hands. Hence, do not hold it with hands. Always use forcepts while handling sodium.
2. Before using sodium metal, press it within the folds of filter paper to remove oil.
3. Use dry ignition tubes for sodium fusion. Sodium reacts with water violently.
4. Put the unused sodium piece back in bottle. Do not throw it in to the sink.
5. Repeat the process of sodium fusion with three ignition tubes. This is to ensure that the fusion has taken place.
6. After immersing the red hot ignition tube in water, break it with a glass rodgently. Boil the contents for 2-3 minutes so as to extract the soluble sodium salts in water.

10.6 OBSERVATIONS

Table 10.1

S.No.	Experiment	Observations	Inference

The given sample responded postively to _____ _____ and _____ tests. Therefore, the extra elements present in the sample is _____

10.7 CONCLUSION

The given organic compound contains..... as extra elements.

10.8 CHECK YOUR UNDERSTANDING

1. *Why is only freshly cut sodium metal used for fusion of an organic compound ?*

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2. *Why is it necessary to carry out the fusion of an organic compound with sodium, more then once using different ignition tubes ?*

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3. *While testing for nitrogen why is it necessary to neutralize the sodium extract completely with an acid ?*

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4. *Why is it necessary to fuse an organic compound with sodium for carrying out the tests for extra elements present in it ?*

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5. *Why are bromide and iodide displaced from their salts in solution by chlorine water?*

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6. *What will happen if sodium extract is not completely neutralized before performing silver nitrate test for halogens ?*

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7. *Why is it essential to use distilled water in the preparation of sodium extract ?*

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10.9 NOTE FOR TEACHER

Sodium metal is highly reactive. Ensure that students handle it carefully and take all precautions.

In preparing sodium fusion extract, the low concentrations of ions in solution give only a faint colour or precipitate. It is advisable to use 2-3 ignition tubes during sodium fusion. In case of liquid compounds, the molten sodium metal is first covered with sodium carbonate. The liquid compound is added over it so that it is absorbed in sodium carbonate. The low boiling liquids tend to escape without undergoing reaction with sodium.

After the students have performed the tests of all the extra elements, they may be asked to identify the extra elements in given compounds and write full details in the experiments sheet.

10.10 CHECK YOUR ANSWERS

Ans1. Sodium is a highly reactive metal. Its surface gets a coating of its oxide due to its reaction with atmospheric oxygen. By taking a freshly cut Sodium, the expressed surface is free from Oxide.

Ans2. A majority of organic compounds are low boiling and volatile. They tend to escape out of the ignition tube during heating. To ensure an adequate concentration of ions containing extra elements in test solution, it is preferable to carry out the sodium fusion 2-3 times.

Ans3. On adding ferrous sulphate to sodium extract, ferrous hydroxide forms a dirty green precipitate. To avoid this, the test solution is neutralized completely.

Ans4. The extra elements are present as covalently bonded atoms in organic compounds. To test them in solution, these are converted into ionic form by fusing the compound with sodium.

Ans5. Chlorine, being more electronegative displaces bromide and iodide in solution.

Ans6. A grey black precipitate of silver oxide is formed. It becomes difficult to identify the colour of silver halides.

Ans7. Ordinary water contains chloride ions. These interefere with the halide ions formed from halogen of the organic compound.