DEPARTMENT OF CHEMISTRY
SURENDRANATH COLLEGE
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Paper: Lab - Syllabus

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Experiment: Organic Qualitative analysis

1) Detection of elements (Nitrogen, Sulphur and halogens).
2) Detection of functional groups of organic compounds by qualitative method.
3) Practical Note Book
4) MCQ

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(Writing model for laboratory note book)

Experiment No. 1: To identify the presence of elements and functional group present in the given organic sample.

1. **Preliminary test of organic sample:**

Experiment	Observation	Inference
i) **Colour and physical state**	Reddish brown liquid substance.	Compound may contain amine group.
ii) **Odour**	Aniline like smell.	Aromatic amine may be.
i) Solubility	Insoluble in water but soluble in dilute acids.	Amine may be.
ii) Litmus test:	Red litmus turned blue.	Amine may be.
iii)Flame test: Placed the compound (about 0.1 g) on a nickel spatula and heated over a burner flame.	Smoky flame.	Aromatic compound.
iv)Unsaturation test: a) 1 mL of organic substance or its	Precipitate appear but no	Saturated compound.

solution + bromine water.	change colour of bromine solution.	
b) 1 mL of organic substance or its solution + 1 mL dil. H_2SO_4 + 2 mL alkaline $KMnO_4$ solution.	No decolourisation of $KMnO_4$ solution.	Saturated compound.

2. Detection of elements present (Procedure for Lassaigne's test): Taken a small piece of (0. 04 g) freshly cut sodium metal and dried by pressing in between a filter paper. Taken a dry and clean fusion tube and placed inside the small piece of sodium metal. Heated the fusion tube until formation of sodium vapour (dark grey in colour) and poured the small amount of powdered organic sample portion directly on to the molten sodium. Heated the fusion tube to redness for about two minutes and carefully dipped the hot tube along with fusion residue in a porcelain basin having distilled cold water and crushed the tube. Boiled the resulting product and filtered the hot solution and washed the residue with distilled water. From the Lassaigne's filtrate the following tests are preformed.

Experiment	Observation	Inference
1. Taken 2-3 mL of Lassaigne's filtrate in a test tube and add 0.1-0.2 g of powdered iron(II) sulphate. Heated the mixture and at hot condition added dilute sulphuric acid to dissolve the iron hydroxide.	A Prussian blue precipitate is obtained.	Nitrogen is present.
2. (a) Added few drops of dilute acetic acid to 2-3 mL of Lassaigne's filtrate and added few drops of lead acetate solution.	No precipitate is obtained.	Sulphur is absent.
(b) Added 2-3 drops of freshly prepared dilute solution of di-sodium pentacyanonitrosyl ferrate (sodium nitroprusside) to 2-3 mL of Lassaigne's filtrate.	No purple coloured solution.	Sulphur is absent.
3. Added few drops of dilute nitric acid to 2-3 mL of Lassaigne's filtrate and added excess amount of sliver nitrate solution.	(a) No precipitated.	Chlorine is absent.
	(b) No precipitated.	Bromine is absent.
	(c) No precipitated.	Iodine is absent.

3. Tests for functional group present in given organic substance:

Experiment	Observation	Inference
1. Test for alcoholic hydroxyl group: **Ester test**: 0.1 g of organic substance + 1 mL of conc. H_2SO_4 + 1 mL glacial acetic acid. Heated the mixture for five minutes.	No ester smell appeared.	Alcoholic hydroxyl (-OH) group is absent.
2. Test for phenolic hydroxyl group: **Liebermann's test:** Organic substance + 2-3 crystals of sodium nitrite then heated. On cooling- a) Added 1 mL conc. H_2SO_4	No coloured precipitate appeared.	Phenolic (-OH) group is absent.
ii) Poured the above mixture in a beaker containing water	No change of colour of the solution.	Phenolic (-OH) group is absent.
iii) Made the above mixture alkaline by adding NaOH solution.	No change of colour of the solution.	Phenolic (-OH) group is absent.
3. Test for carbonyl group: **a) Sodium bisulphite test:** 2-3 crystals of organic substance + 2 mL sodium bisulphite solution. Allowed the mixture undisturbed.	No precipitate appeared.	Carbonyl (-CHO or >C=O) group is absent.
b) 2,4-Dinitrophenyl hydrazine (Brady's reagent) test: 1 mL alcoholic solution of organic substance + solution of 2,4-dinitrophenyl hydrazine. Kept the solution in hot water for 15 minutes	No yellow or red precipitate appeared.	Carbonyl (-CHO or >C=O) group is absent.

and then allowed cooling.		
4. Test for carboxyl group:		
a) Sodium bicarbonate test: 1 mL aqueous solution organic substance + solid $NaHCO_3$.	No evolution of gas.	Carboxyl(-COOH) group is absent.
b) Ferric chloride test: 1 mL aqueous solution organic substance + neutral solution of ferric chloride.	No change of the colour of the solution.	Carboxyl (-COOH) and phenolic hydroxyl group is absent.
5. Test for amine group:		
a) Azo dye test: Dissolved the organic substance in 1-2 mL dil. HCl and cool the solution by ice water. In cold condition added concentrated solution of $NaNO_3$. Again in cold condition added few drops of alkaline solution of β-naphthol.	Bright coloured dye appeared.	Amine ($-NH_2$) group is confirmed.
b) Carbylamine test: Organic substance + 2 mL alcoholic KOH solution + 2-3 drops of chloroform. Heated the mixture.	Offensive smelling gas evolved.	Primary amine ($-NH_2$) group is confirmed.
6. Test for amide group:		
a) Test with nitrous acid: Taken 0.5 g of organic substance in alcohol and added few volume of nitrous acid	No effervescences are detected.	Primary aliphatic amide group is absent.
b) Test with alkali: Taken 0.5 g of organic substance and added excess amount of concentrated sodium hydroxide solution and boiled the content.	No smell of ammonia.	Primary aliphatic amide group is absent.

c) **Test with hydroxylamine hydrochloride:** Taken 0.5 g of organic substance and added few amount of alcoholic solution of hydroxylamine hydrochloride. Boiled the content for five minutes and allowed cooling. In cold condition added 4-5 drops of dilute ferric chloride solution.	No bluish red coloured solution.	Aromatic amide group is absent.
d) **Biuret test:** Heated 0.5 g of organic substance in a test tube for elimination of vapour (if evolve) and added 1 mL of dilute alkali on cold condition and then added two drops of dilute copper sulphate solution.	No pink colour developed.	Compound is not urea.
7. Test for hydrocarbon:		
a) **Iodine test**: 0.2 g of organic substance (*2-3 drops if it is liquid*) + 5 mL benzene in a test tube. Above solution + 5 mL of very dilute solution of iodine (in benzene)	Violet colour is not persistent.	Hydrocarbon is absent.
b) **Friedel-Crafts test:** Heated 0.1 g of $AlCl_3$ in a dry test tube + 2-3 drops of mixture of equal amounts of organic substance and chloroform to $AlCl_3$.	No dye is produced.	Aromatic hydrocarbon is absent.

Conclusion: From the above systematic experiments it is found that the given organic substance contain nitrogen as element and it contains primary amine ($-NH_2$) group.

N.B: Mention the reactions for positive tests in the left hand side of the laboratory note book only by pencil.

Potassium can replace sodium in lassaigne's test.
a) True b) False
Answer: a
Explanation: Potassium, like sodium is electropositive in nature. In lassaigne's test, the elements present in the compound are converted from their covalent form to their ionic form by fusing the compound with sodium metal. Since, potassium has similar characteristics of electro positivity as sodium and since potassium is highly reactive, it can be used instead of sodium in lassaigne's test.
5. What is Lassaigne's test extract called as?
a) Fusion extract b) Sodium fusion extract c) Lassaigne extract d) Sodium extract
Answer: b
Explanation: Lassaigne's test extract is called as sodium fusion extract. The cyanides, sulphides and halides of sodium will be formed. These will be extracted from the fused mass by boiling it with distilled water. Hence, the name of the extract is sodium fusion extract.
6. In the test for nitrogen, the sodium fusion extract is acidified with which of the following?
a) Dilute sulphuric acid b) Dilute hydrochloric acid
c) Concentrated hydrochloric acid d) Concentrated sulphuric acid
Answer: d
Explanation: In the test for nitrogen, sodium cyanide first reacts with iron (III) sulphate and forms sodium hexacyanoferrate (II). On heating with concentrated sulphuric acid, some iron (II) ions are oxidized to iron (III)ions which react with sodium hexacyanoferrate (II) to produce iron (III) hexacyanoferrate (II), which is Prussian blue in color. Moreover, nitrogen atoms are soluble in concentrated sulphuric acid.
7. What is the color of the precipitate obtained in the test for sulphur?
a) White b) Black c) Violent d) Blue
Answer: b
Explanation: In the test for sulphur, the sodium fusion extract is acidified with acetic acid and lead acetate is added to it. Once this reaction takes place, a black precipitate is formed. This black precipitate is lead acetate, indicating the presence of sulphur.
8. In case of both nitrogen and sulphur existence, Prussian blue is still the color of the end product.
a) True b) False
Answer: b
Explanation: In case, both nitrogen and sulphur are present in an organic compound,

sodium thiocyanate is formed. The color formed is blood red and not Prussian blue. This is because, in this case, there are no free cyanide ions.

$Na + C + N + S \rightarrow NaSCN$

$Fe^{3+} + SCN^{-} \rightarrow [Fe(SCN)]^{2+}$ (blood red)

9. A X color precipitate, which is Y in ammonium hydroxide indicates presence of chlorine. Identify X and Y.

a) X = yellowish, Y = soluble b) X = yellow, Y = insoluble

c) X = white, Y = insoluble d) X = white, Y = soluble

Answer: d

Explanation: During the detection of chlorine, when the organic compound reacts with sodium, it forms sodium chloride. This sodium chloride gives the white precipitate of silver nitrate with silver nitrate solution. This white precipitate is also soluble in ammonium hydroxide.

In Lassaigne's test, the sulphur present in the organic compound, on fusion with sodium, is converted into:

a) Na_2S b) $Na_2S_2O_3$ c) CH_3SH Na_2S_2

1.Why is sodium metal kept under kerosene oil?

Ans. Sodium metal reacts with oxygen and moisture present in air. Kerosene oil prevents the contact of air and sodium and thus protects it from the action of moisture and oxygen.

2. Why an organic compound is fused with sodium metal for preparing Lassaigne's extract ?

Ans. When the organic compound is heated with sodium, the elements such as nitrogen, sulphur and halogens, if present in the compound, are converted into sodium salts which are soluble in water. The aqueous solution is then used to identify these elements.

3. Can we use potassium in place of sodium in Lassaigne's test ?

Ans. No. Potassium is too reactive metal and hence dangerous to use.

4. In the Lassaigne's test for nitrogen what is the bluish green colour due to?

Ans. It is due to the formation of ferric ferrocyanide, $Fe_4[(FeCN)_6]_3$.

5. During the detection of nitrogen, sometimes a blood red colour is obtained. What is this due to?

Ans. The formation of blood red colour with $FeCl_3$ solution shows the presence of both N and S in the organic compound. It is due to the formation of $Fe(CNS)_3$.

6.Why is fresh solution of $FeSO_4$ used in test for nitrogen ?

Ans. On keeping $FeSO_4$ solution for a long time, it gets oxidised to ferric sulphate by atmospheric oxygen. So, it will not give the desired reaction.

7. What is the function of the addition of HCl in the detection of nitrogen ?

Ans. The function of adding HCl is to dissolve green ppt. of Fe(OH)2 otherwise it may lead to wrong inferences.

8. Why is sodium metal dried up before fusion ?

Ans. It is done to avoid explosion due to of vapours of kerosene oil during heating.

(Don't write in laboratory note book)

Precautions

a) *Do not try to inhale organic compounds as most of organic compounds are poisonous.*
b) *Chloroform should use in the presence of laboratory attendants.*
c) *Do not try to inhale the gas evolved in carbylamine test.*
d) *Always use the minimum amount of available reagent. Do not mention the test which is not done practically in the laboratory.*
e) *Mention two positive tests for a functional group and one negative for each functional group.*