ORGANIC PRACTICAL:

INTRODUCTION:

Simple organic compounds are classified according to their functional groups.
These functional groups are the reaction sites where specific reactions particular to a certain group of compounds take place.
Organic compounds may also be classified as **ALIPHATIC** and **AROMATIC** Compounds.
Aliphatic compounds are the straight chain compounds while aromatic compounds are those that contain a benzene ring.

NOTE:
For aromatic compounds when the functional group is attached to the side chain, the reactions are similar to those of the aliphatic compounds.
But if the functional group is attached to the benzene ring directly, then the properties are different.

In most cases, organic qualitative analysis what is needed is to determine the functional Group present in the sample, but there are times when the group attached to the functional group is also tested for. e.g. the presence of a methyl group (-C H_3) adjacent to the functional group.

The analysis involves a number of stages that include;

1. **appearance**

   The molecules of organic compounds are held together by weak Vander Waal's forces
   and or H- bonds. This often makes them to exist as liquids or gases at room temperature.
   Where they exist as solids, they either easily melt or sublime on heating usually the liquids and or solids are colourless while solids are often salts of carboxylic acids and
   carbonyl compounds.

   (a) Liquids;
   These include alcohol, aldehydes, ketones, carboxylic acids, esters, aliphatic
   and aromatic hydrocarbons.

   (b) Solids;
   These include salts of carboxylic acids, phenol, dicarboxylic acids and carbohydrates.

2. **Burning the substance** (The flame test)

   Unlike inorganic compounds, organic compounds burn and the type of flame is a useful guide;

   (a) Saturated aliphatic compounds with short carbon chains burn with a blue, non- sooty flame.
   (b) Aromatic compounds burn with a yellow heavily sooty flame.

NOTE;
Unsaturated aliphatic compounds burn with a yellow flame, which is
slightly sooty
- Compounds with high oxygen content e.g. carbohydrates and carboxylic acids don't easily burn.

3. Smell;

The smell of the compound can be a guide but not a sure test –

Aldehydes, ketones and esters have a fruity smell.
Methanoic and Ethanoic acids have an irritating smell.

4. Solubility in Water;

The solubility of organic compounds decreases with increasing molecular mass of the compounds in a given series.
Therefore, if the substance readily dissolves in water, then the sample is likely to be a low molecular compound.

NOTE: Aromatic compounds are less soluble compared to their corresponding aliphatic compounds.
Usually the solution formed with water is tested with litmus (blue and red) or universal indicator to ascertain whether it is neutral, basic or acidic;

(i) Dissolves and solution has no effect on litmus paper-
saturated short carbon chain aliphatic, alcohol, carbonyl compound (ketone or aldehyde), ester or carbohydrate.

(ii) Dissolves and solution turns litmus red-
Aliphatic short carbon chain, carboxylic acid or a salt of an amine with a strong acid (due to hydrolysis)

NOTE;

1. Phenol and aromatic carboxylic acids do not easily dissolve in cold water, but soluble in hot water, and their solutions turn litmus red.

2. To distinguish between the carboxylic acid and phenol, add a little sodium carbonate or sodium hydrogen carbonate to the original sample or portion of the solution – carboxylic acids liberate carbon dioxide while phenol does not.

(iii) Dissolves and solution turns litmus blue –
Amine or a salt of a carboxylic acid with sodium, potassium or calcium (these substances under go hydrolysis, producing alkaline solutions)

5. Action of dilute sodium hydroxide

Add 2 cm^3 of sodium hydroxide solution to 1 cm^3 of the unknown sample and shake – if no reaction appears to take place, warm.

(a) If the compound did not dissolve in water but dissolves in sodium hydroxide solution –This indicates an aromatic carboxylic acid or phenol.

(b) If the compound is a liquid with fruity smell and slightly soluble in water – it dissolves in aqueous sodium hydroxide, on boiling the smell goes and another layer formed – this indicates an ester.
(c) If a brown, oily strong smelling resin formed – a lower aliphatic alcohol.

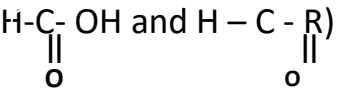
6. **Brady's reagent** (2,4 – dinitrophenyl hydrazine)
Add a few drops of Brady's reagent – a yellow ppt indicates the presence of a carbohyl Compound (ketone or aldehyde)

OR; To 1 cm^3 of the unknown add 2-3 drops of 2,4- dinitrophenyl hydrazine in the presence of a few drops of conc. HCl as a catalyst warm the mixture and finally dilute with water – ketones and aldehydes give a yellow ppt.

Note; Brady's reagent is used to identify carbonyl compounds which give a yellow ppt.
To distinguish between ketones and aldehydes, this is followed by the use of Fehling's
Solution, Tollen's reagent (silver nitrate in ammonia solution) or acidified potassium dichromate solution.

7. Fehling's solution.
To 1 cm^3 of the unknown, add 0.5 cm^3 of Fehling's solution I and 0.5 cm^3 of Fehling's solution II. A red ppt indicates the presence of an aldehyde.

NOTE;
Sometimes the precipitate may be brown or yellow- still that indicates an aldehyde.
If the solution mixture remains blue, that shows that an aldehyde is absent (ketone present)
In case the sample initially gave yellow/orange ppt with Brady's reagent).
HCOOH also gives a red – brown ppt (behaves like aldehydes, compare the structures



8. **Tollen's reagent** (Ammonical silver nitrate/ silver mirror test)

To 5 drops of the unknown, add about 5 cm^3 of the Ammonical silver nitrate.
OR; To 3 cm^3 of silver nitrate solution add 2 drops of dilute sodium hydroxide, then add aqueous ammonia dropwise until the precipitate just dissolves. Add 2 cm^3 of the unknown and warm,

- A silver mirror on the wall of the test tube confirms the presence of an aldehyde. Also HCOOH gives a silver mirror (it is a reducing agent like aldehydes)

9. Acidified potassium dichromate solution.

To 2 cm^3 of the unknown add 1 cm^3 of acidified potassium dichromate solution and heat the mixture – Orange solution turns green. This indicates the presence of an aldehyde or primary/ secondary alcohol (Also HCOOH, it is a reducing agent- so it gives result similar to aldehydes)

NOTE;

(i) $K_2Cr_2O_7/ H^+$ is a strong oxidising agent which oxidises aldehydes to carboxylic acids and itself reduced, from Cr^{6+} to Cr^{3+}

(ii) $K_2Cr_2O_7 /H^+$ oxidises primary and secondary alcohols to carbohyl compounds, aldehydes and ketones respectively.

(iii) Sometimes, you may add $Cr_2O_7^{2-}/H^+$ on warming the solution turns from orange to green indicating aldehyde, 1^o or 2^o alcohol – when asked to add Brady's reagent to this mixture, formation of an orange / yellow precipitate indicates that a carbonyl compound has been formed, therefore the unknown substance should be a primary or secondary alcohol

10. Luca's reagent;

Made by mixing 1:1 mole ratio of anhydrous zinc chloride with concentrated hydrochloric acid.

To 1 cm^3 of the unknown (alcohol) add 2 cm^3 of Luca's reagent, shake well and allow to stand (preferably in ice –water mixture)

(i) If solution remains clear (No observable change), primary (1^o) alcohol is present.

(ii) If the solution becomes cloudy within 5-10 minutes, a secondary (2^o) alcohol

(iii) If the solution immediately turns cloudy (an insoluble layer forms at the surface), a tertiary alcohol is present.

NOTE;

Phenol doesn't show any such result.

11. **Ethanoic acid** (Esterification)

To 1 cm^3 of the unknown (alcohol) add 1 cm^3 of glacial Ethanoic acid followed by a few drops of concentrated sulphuric acid. Boil the mixture for about 2 minutes.

Pour this mixture in a small beaker of cold water - sweet fruity smell indicates an alcohol.

NOTE;
Esterification is also used to confirm the presence of a carboxylic acid;
i.e to 1 cm^3 of the unknown, add 1 cm^3 of pure Ethanoic acid followed by a few drops of concentrated sulphuric acid; boil the mixture for about 2 minutes. Dilute the mixture with cold water, a sweet fruity smell indicates a carboxylic acid;

12. Concentrated Sulphuric acid or Aluminium oxide;
At high temperatures these are dehydrating agents-
Heat a mixture of concentrated sulphuric acid or aluminium oxide with the unknown (alcohol) and bubble the gas evolved through acidified potassium permanganate solution
The gas evolved decolourises the solution, this indicates an alcohol.

13. Acidified potassium permanganate solution ;
To 1 or 2 cm^3 of the unknown (alcohol) add 2-5 drops of H^+/ MnO_4^-The permanganate solution is decolourised, indicating the presence of an alcohol (primary or secondary) or aldehyde.
$H^+/KMnO_4$ rapidly oxidises 1^o and 2^o alcohols to aldehydes and ketones respectively and aldehydes are oxidised to carboxylic acids.
NOTE;
Methanoic acid also decolourises MnO^-_4/ H^+ (it is a reducing agent)

14. Iron(III)chloride solution
To 1 cm^3 of the unknown, add 2 drops of iron (III) chloride solution.
- A reddish brown solution (or precipitate) indicates the presence of an aliphatic carboxylic acid.
- A pale yellow – brown ppt indicates the presence of an aromatic carboxylic acid
- Violet /purple solution indicates presence of phenol.

15. Soda lime (Mixture of sodium hydroxide and calcium oxide)
To a small portion (1 cm^3 or a spatula endful) of the unknown add two spatula endfuls of soda lime and heat the mixture.

- A colourless gas that turns limewater milky (carbondioxide) indicates presence of a carboxylic acid.
  NOTE; The carbon dioxide evolved is due to the decarboxylation of the carboxylic acid.

16. Phosphorus (v) chloride or phosphorus (III) chloride,

To 1 cm^3 of the unknown add a little PCl_5, PCl_3 or $SOCl_2$
White fumes (of HCl) indicates the presence of a compound containing an –OH group,i.e alcohol or phenol.

17. Iodoform test

Add 2 cm^3 of iodine solution to 1 cm^3 of the unknown followed by dropwise addition of dilute sodium hydroxide solution until the brown colour of iodine is just discharged. Heat the mixture if necessary.

- A yellow ppt indicates the presence of a methly group ($-CH_3$) attached to the carbon atom with the functional group. ie

$$CH_3-\overset{O}{\overset{\|}{C}}-R \text{ where R = H or alkyl group}$$

$$CH_3-\overset{OH}{\overset{|}{C}}-R$$

Iodine in alkaline solution reacts with these compounds to form triodomethane, CHI_3, the yellow ppt.

Note;
This test is negative for ethanoic acid despite the presence of the methyl group

$$CH_3-\overset{O}{\overset{\|}{C}}-OH$$

Due to the reaction between the acid and sodium hydroxide.

18. Nitrous acid;

Dissolve 1 cm^3 of the unknown in concentrated sulphuric or hydrochloric acid; cool the mixture and then add ice-cold sodium nitrite solution dropwise and keep the solution mixture in an ice- bath for 5- 10 minutes.

- Clear colourless solution with evolution of nitrogen gas, indicates presence of primary aliphatic amine.
- Clear colourless solution with <u>NO</u> evolution of nitrogen gas, indicates presence of primary aromatic amine.

Here the compound formed reacts with phenol in alkali to give brightly coloured azo dyes.

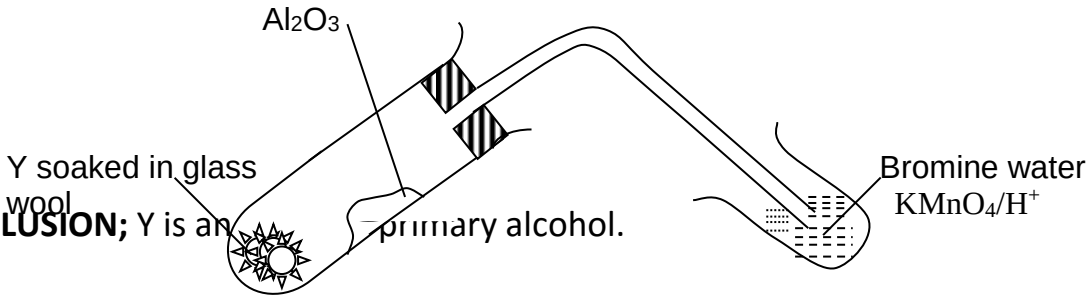
- Secondary amines give yellow oily solutions, the nitroso amine.
- Tertiary amines give clear solutions with NO evolution of nitrogen gas.

EXAMPLES:

1. You are provided with an organic substance Y. Carry out the following tests and record your observations and deductions in the table below;

Test	**Observation**	**Deduction**
Burn a little of Y on a spatula.	Colourless liquid burns with a non- sooty flame	Saturated aliphatic compound, alcohol ketone, aldehyde or carboxylic acid.
Add 1 cm^3 of Y to 1cm^3 of water in a test tube and shake. Test the mixture with litmus, Add 2- 3 drops of sodium carbonate soln	Dissolves forming a colourless solution that has no effect on litmus,No observable change.	Low molecular aliphatic compound. Alcohol, ketone or aldehyde.
Add 2 –3 drops of Y to 2 cm^3 of Brady's reagent	No observable change (solution remains orange)	Carbonyl compound absent, alcohol present.
Add 3 drops of Y to 2cm^3 of iodine solution .Add NaOH(aq) dropwise until the colour of iodine is discharged.	Yellow ppt	A methyl group next to the carbon with the functional group CH_3- C – OH
Add 2 cm^3 of $K_2Cr_2O_7/H^+$ to 3 cm^3 of Y in a boiling tube heat the mixture. Divide the solution into two parts	The orange solution turns green.	Primary or secondary alcohol oxidised.
To the first part add 3 drops of Brady's reagent	Yellow ppt	Carbonyl compound from oxidation of primary or

		secondary alcohol
To the second part add 3 drops of sodium carbonate solution	Bubbles of colourless gas, turns lime water milky.	Carboxylic acid from oxidation of primary alcohol
Soak glass wool in Y and place it in a boiling tube, Put 1g of Al_2O_3 mid way the boiling tube and set up the apparatus as below	White fumes decolourise the solution.	Alkene from dehydration of primary alcohol



CONCLUSION; Y is an primary alcohol.

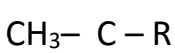
NOTE: Primary alcohols are oxidised to aldehydes; in the presence of a strong oxidising agent with strong heating, the aldehyde is further oxidised to a carboxylic acid.

2. You are provided with an organic substance N carry out the following tests and record your observations and deduction in the table bellow.

Test	**Observation**	**Deduction**
Burns a little of N on a spatula	Colourless liquid burns with a non sooty flame	Aliphatic compound alcohol, ketone, aldehyde or carboxylic acid.
Add 1 cm^3 of water to 1 cm^3 of N and test the mixture with litmus.	Dissolves to form colourless solution No effect on litmus	Low molecular aliphatic compound, alcohol, ketone or aldehyde
Add 2- 3 drops of N to 2 cm^3 of Brady's reagent.	Yellow ppt	Ketone or aldehyde
Add 2 cm^3 of acidified potassium dichromate solution and heat.	No observable change	Ketone present, aldehyde absent.
Add 5 drops of Fehlings solution and heat the mixture over a water bath.	No observable change.	Aldehyde absent ketone present

To 1 cm^3 of silver nitrate 2 drops of NaOH, add NH_3(aq) dropwise the ppt just dissolves. Add 1 cm^3 of N and warm the mixture over hot water.	No observable change.	Ketone confirmed.
To 1 cm^3 of N add 2 cm^3 of iodine solution followed by NaOH(aq) until the colour of iodine is discharged	Yellow ppt	Ketone with a methyl group next to the carbon bearing the functional group.

The nature of N is; Aliphatic ketone with a methyl group attached on the functional group



3. You are provided with an organic substance W. You are required to determine the nature of the substance. Carry out the following tests to identify W. Record your observations in the table below.

TEST	**OBSERVATION**	**DEDUCTION**
Burn a small about of W on a spatula.	Colourless liquid burns with a non- sooty flame.	Saturated aliphatic compound.
To 1 cm^3 of W add 2 cm^3 of water and shake. Test with litmus, add a little sodium carbonate powder to the solution.	Dissolve forming a colourless solution turns litmus red. Effervescence of a colourless gas turns lime water milky.	Low molecular compound. - COOH present. (carboxylic cpd)
Mix 1 cm^3 of W with 1 cm^3 of ethanol and add 5 drops of conc sulphuric acid and warm, pour the mixture into 10 cm^3 of water.	Sweet fruity smell.	Esterification - COOH Present.
To 1 cm^3 of W, add 5 drops of Iron (III) chloride solution.	Brown solution.	Phenol absent - COOH Present
To 1 cm^3 of W, add 3 drops of acidified potassium manganate (VII) solution.	Purple solution turns colourless.	HCOOH Confirmed.

(f) State the identity of W.

Formic acid – HCOOH (or methanoic acid)

4. You are provided with substance Z which is organic. You are required to determine the nature of Z. Carry out the following tests and identify any gases evolved. Record your observations and deduction in the table below;

TEST	OBSERVATIONS	DEDUCTIONS
Burn a small amount of Z on a crucible	Burns with a blue non-sooty flame.	Saturated Aliphatic compound
Add $2cm^3$ of water to $1cm^3$ of Z, test the mixture with litmus.	Colourless liquid, dissolves to form colourless solution neutral to litmus.	Alcohol, Ketone or Aldehyde
To $2cm^3$ of Brady's reagent, add 2 drops of Z	Yellow ppt	Aldehyde or Ketone
To $4cm^3$ of saturated sodium hydrogen sulphite solution, add $2cm^3$ of Z.	Colourless crystals	Aldehyde or Ketone
To 5 drops of Z, add 4 cm^3 of iodine solution followed by sodium hydroxide solution added dropwise until the colour of iodine is discharged. Warm the mixture gently.	Yellow ppt	Methyl carbonyl conpound O CH3–C– R
To 3 cm^3 of Z add $1cm^3$ of acidified potassium dichromate solution, warm the mixture.	No observable change	Ketone present
To 3 cm^3 of silver nitrate	No observable change	Methyl ketone comfirmed

solution add 2 drops of sodium hydroxide solution. Then add ammonia solution dropwise until the precipitate dissolves, add 5drops of Z and warm.		present.

Deduce the nature of Z ; Methyl carbonyl compound

$$CH_3 - \overset{O}{\overset{\|}{C}} - R$$

EXERCISES

1. You are provided with an organic substance K carry out the following tests to identify the nature of K. Record your observations and deductions in the table below.

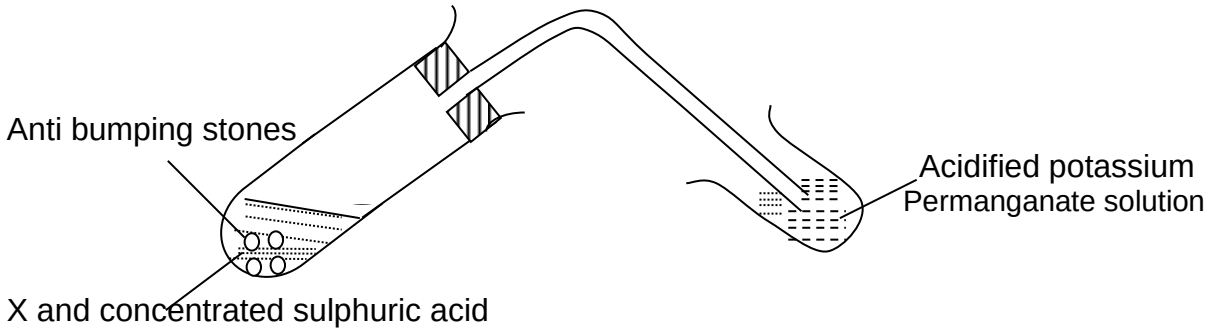
TEST	**OBSERVATION**	**DEDUCTION**
Burn a small amount of K on a crucible or spatula		
To 1 cm^3 of K add 2 cm^3 of warm and shake. Test the mixture with litmus paper.		
To 1 cm^3 of Brady's reagent add a few drops of K		
To 2 cm^3 of K add 2 cm^3 of acidified $K_2Cr_2O_7$ and heat. Add 5 drops of Brady's reagent.		
To 2 cm^3 of K add 1 cm^3 of Zinc chloride solution made by dissolving anhydrous Zinc chloride in concentrated hydrochloric acid. Leave to		

stand for 10 minutes.		
To 1 cm^3 of K add 4 cm^3 of iodine solution followed by dilute sodium hydroxide solution dropwise until the iodine is discharged warm.		

(g) Identify;
(i) the functional group in Y. ……
(ii) the class of compounds to which Y belongs. ……

2. You are provided with an organic compound J. You are required to determine the nature of J. Carry out the following tests and record your observations and deduction in the table below.

TEST	**OBSERVATIONS**	**DEDUCTIONS**
(a) Burn a small amount of J on a spatula or porcelain		
(b) Divine the remaining J into five portions (i) To the first portion add water and test with litmus.		
(ii) To the second add 2 drops of Iron (III) chloride solution		
(iii) To the third portion add acidified potassium dichromate solution.		
(iv)To the forth portion add Brady's reagent.		
(iv) To the fifth add 2 cm^3 of conc sulphuric acid and set the apparatus as shown below heat the mixture and bubble any vapour evolved through acidified $KMnO_4$ (aq)		



Explain the nature of J.

3. You are provided with an organic substance B. You are required to determine the nature of B
Carry out the following tests on B, record your observations and deductions in the table below.

TEST	OBSERVATIONS	DEDUCTIONS
(a) Burn a small amount of B on a spatula end or porcelain dish		
(b) Shake 3 drops of B with 2cm^3 of water and allow to stand. Test with litmus.		
(c) Add 2-3 drops of acidified potassium dichromate solution to a few drops of B. Heat the mixture.		
(d) Add 2 – 3 drops of Brady's reagent to 0.5 cm^3 of B.		

(e) Dissolve 4- 5 drops of B in 1 cm^3 of methanol then add about 1 cm^3 of dilute sodium hydroxide followed by iodine solution until the colour of iodine persists then warm and allow to stand.		

Comment on the nature of B,

……

……

4. You are provided with W, a single organic compound, you are required to determine the nature of W. Carry out the following tests on W and record your observations and deductions in the table below;

TEST	**OBSERVATION**	**DEDUCTION**
(a) Burn a small amount of W at the end of a spatula		
(b) Dissolve 2 cm^3 of W in 5cm^3 of water and divide the solution into five parts.		
(i) Test the first part with litmus paper, then add 3 drops of sodium hydrogen carbonate solution.		
(ii) To the second part add 3 drops of acidified potassium dichromate solution heat the mixture.		
(iii)To the third part add 3 drops of Brady's reagent.		
(iv)To the forth part add 3		

drops of fehling's solution and heat.		
(v) To the fifth part add 1cm^3 of iodine solution then sodium hydroxide dropwise until the colour of the mixture is discharged, then heat.		

(d) State the nature of W

……
……

5. You are provided with sample R, an organic compound. You are required to identify the nature of R. Carry out the following tests on R and record your observations and deductions in the table below;

TEST	OBSERVATIONS	DEDUCTIONS
a) Burn a small amount of R at the end of a spatula.		
b) Add 2 drops of R to 1cm^3 of water, shake and test the solution with litmus		
c) To 1cm^3 of R add 3cm^3 of acidified $K_2Cr_2O_7$ solution and boil the mixture. Divide the resultant solution into two(2) parts.		
(i) To the first part add 5 drops of Sodium hydrogen carbonate solution.		
(ii) To the second part add 5 drops of Brady's reagent.		

d) To 2cm^3 of R, add 2cm3 of Tollen's reagent. Heat the mixture.		
e) To 1cm^3 of R add 2cm3 of iodine solution followed by sodium hydroxide solution until the colour of iodine is discharged.		

Conclusion;
Identify the nature of R ……

6. You are provided with a substance L which is a single organic compound. You are required
To determine the nature of L. Carry out the following tests on L and record your observations and deductions in the table below. Identify any gases evolved.

TEST	**OBSERVATIONS**	**DEDUCTIONS**
(a) Burn a small amount of L at the end of a spatula		
(b) Add two drops of L to 1cm^3 of water and test the solution with litmus then add aqueous sodium carbonate dropwise to the same solution		
(c) Add 4 drops of L to 1cm^3 of ethanol followed by a few drops of concentrated sulphuric acid. Heat the mixture and pour the product in a beaker of cold water.		

(d)To 1 cm^3 of L add a little of potassium permanganate solution and warm		
(e)To 1 cm^3 of L add 3 drops of neutral Iron (III) chloride solution.		

Identify the nature of L

……..

7. You are provided with an organic compound A. You are required to determine the nature of A
   Carry out the following tests and record your observations and deductions in the table below. Identify any gases evolved.

TEST	OBSERVATIONS	DEDUCTIONS
a) Burn a little A directly on a spatula		
b) To a little of A add an equal volume of water followed by 2 drops of universal indicator.		
c) To 1cm^3 of A add a little solid sodium carbonate. (do not shake)		
d) Mix 1cm^3 of A with a spatula endful of soda lime and heat the mixture.		
e) To a little of A add a few drops of neutral iron (iii) chloride solution.		

f) To 1cm^3 of A add 5 drops of acidified potassium permanganate solution and warm.		

Identify the nature of A

……

8. You are provided with an organic substance F. Carry out the following tests and record your observations and deductions in the table below.

TEST	OBSERVATIONS	DEDUCTIONS
(a) Burn a little of F on a spatula end		
(b) Add 1 cm^3 of water to 1 cm^3 of F and test the mixture with litmus.		
(c) Add 1 cm^3 of Brady's reagent to 4 drops of F.		
(d) Add 2 cm^3 of acidified potassium dichromate solution and heat		
(d) To 1 cm^3 of F add 2 cm^3 of iodine solution followed by sodium hydroxide solution dropwise until the		

colour of iodine is just discharged. Warm the mixture.		
(e) To 1 cm^3 of silver nitrate solution add aqueous sodium hydroxide (2 drops) followed by dropwise addition of aqueous ammonia until the ppt just dissolves. Add 1cm^3 of F and heat the mixture over a water bath.		

Identify the nature of F ……