

Melting point determination of known Sample :-Procedure :-

A small quantity of dry and powdered D.S. is forced into the open end of a capillary tube, sealed at one end, by gently tapping on a hard surface or dropping the capillary vertically down ward through a glass tube of suitable height keeping the sealed end of capillary towards bottom when powdered solid is forced down to the closed end. The capillary is then attached to the calibrated thermometer, so that the enclosed sample is kept as close as possible to the middle of the thermometer bulb. This is done by the moistening outside of capillary with conc. H_2SO_4 . The thermometer is now suspended in a bath of conc. H_2SO_4 , so that the thermometer bulb is dipped into conc. H_2SO_4 [Fig-1 next page]

The bath is heated steadily and uniformly by a small flame and temperature at which the solid melts, is noted. The experiment is repeated heating the bath to ten degrees below the expected melting point and then heating the bath equitiously to get the exact melting point.

Compound name	Lt. mp	Observed mp
1. Oxalic acid	190°C	194°C
2. Salicylic acid	158.6°C	157°C
3. Resorcinol	110°C	112°C
4. 3-nitro aniline	114°C	110.5°C
5. Benzoic acid	122°C	125°C

Melting point determination of known mixture sample

procedure:-

A small quantity of dry and powdered mixture sample is forced into the open end of a capillary tube, sealed at one end by gently tapping on a hard surface on dropping the capillary vertical downwards through a glass tube of suitable height keeping the sealed end of capillary towards bottom when powdered solid is forced down to the closed end. The capillary is then attached to the calibrated thermometer, so that the enclosed sample is kept as close as possible to the middle of the thermometer bulb, this is done by the moistening outside of capillary with conc. H_2SO_4 . The thermometer is now suspended in a bath of conc. H_2SO_4 , so that the thermometer bulb is dipped into conc. H_2SO_4 .

The bath is heated steadily and uniformly by a small flame and temperature at which the solid melts, is noted. The experiment is repeated heating the bath to ten degrees below the expected melting point and then heating the bath cautiously to get the exact melting point.

Compound name	observed mp	mixture name	observed mp
1. Oxalic acid	190°C	<u>mixture-1</u> Oxalic acid	180°C
2. Salicylic acid	158.6°C	+ Salicylic acid	
3. Resorcinol	110°C	<u>mixture-2</u> Resorcinol	115°C
4. Benzoic acid	122°C	+ Benzoic acid	

Single detection test for of known organic compound:-

METHYL ALCOHOL, (CH₃OH)

■ physical characteristics and preliminary test :-

- 1. state - liquid.
- 2. Colour - Colourless.
- 3. Odour - pungent.
- 4. Miscibility - In water
- 5. Litmus - Neutral.
- 6. Action of heat - Volatilises.
- 7. Ignition test - Blue non-sooty flame -

■ Confirmatory test :-

Experiment	Observation
<u>Oil of wintergreen test :-</u> 0.5 ml of CH ₃ OH and 3 drops of conc. H ₂ SO ₄ are added to 0.5 g of salicylic acid and heated for one minute and finally the reaction mixture is poured into 50 ml of water taken in a beaker.	Characteristic smell of methyl Salicylate.

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ETHYL ALCOHOL ($\text{CH}_3\text{CH}_2\text{OH}$)

■ physical characteristics and preliminary tests :-

1. State - liquid.
2. Colour - Colourless.
3. odour - pungent.
4. Miscibility - In water.
5. Litmus - Neutral.
6. Action of heat - Volatilises.
7. Ignition test - Blue non-sooty flame.

■ Confirming test :-

Experiment	Observation
<u>Iodoform test</u> :-	
a) To 2ml of aqueous solution of ethyl alcohol an equal volume of conc. solution of iodine in potassium iodide is added and then NaOH solution is added drop wise with stirring. Then the mixture is warmed and cooled under tap with shaking.	a) yellow crystalline ppt. of CHI_3 having characteristic sweet smell formed.
b) The Iodoform test is repeated by adding NH_4OH instead of NaOH.	b) No ppt. of iodoform forms.

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Sema

BENZALDEHYDE (c1ccccc1C=O)

■ Physical characteristics and preliminary tests :-

1. State - Liquid.
2. Colour - Colourless.
3. odour - pungent smell.
4. Miscibility - Immiscible with water.
5. Litmus - No colour change.
6. Action of heat - Volatilises.
7. Ignition test - yellow sooty flame.

■ Confirming test :-

Experiment	Observation
<p><u>2-4-D.N.P. test :-</u></p> <p>A little Benzaldehyde is added to 2-4-dinitrophenylhydrazine.</p>	<p>Reddish yellow p.p.t. forms immediately simply on mixing</p>

FORMIC ACID ($\text{H} - \overset{\text{O}}{\parallel}{\text{C}} - \text{OH}$)

■ physical characteristics and preliminary test :-

1. State - Liquid.
2. Colour - Colourless.
3. Odour - Strong pungent smell.
4. Miscibility - Miscible with water.
5. Litmus - Blue litmus paper turns red. (Sample is acidic)
6. Action of heat - Completely volatilises without leaving any residue.
7. Action of H_2SO_4 -
8. Ignition test - Blue non-sooty flame.

■ Confirming test :-

Experiment	Observation
<u>Mercuric chloride test</u> :- A few drops of neutral solution of formic acid is added to a little HgCl_2 solution and warmed.	White ppt. of mercurous formate formed.
Then excess of neutral solution is added and warmed.	Grey ppt. of metallic mercury formed.

ANILINE (NH_2)

■ physical characteristics and preliminary test :-

1. State - Liquid.
2. Colour - brown.
3. Odour - Characteristic bad smell.
4. Miscibility - Immiscible with water but soluble in diethyl ether.
5. Litmus - Neutral [Feebly basic]
6. Action of heat - Volatilises without leaving any residue. The evolved vapour burns with a smoky flame.
7. Ignition test - yellow sooty flame.

■ Confirmatory test :-

Experiment	Observation
<u>Diazo-coupling test</u> :- 5 drops of aniline are dissolved in diethyl ether in a test tube and cooled in ice-water. Then 4 drops of very dilute ice-cold solution of sodium nitrite are added to it. Then the solution is added to 1 ml of ice-cold alkaline solution of β -naphthol.	Brilliant scarlet-red dye formed.

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DIMETHYL ANILINE ($N(CH_3)_2$)

■ physical characteristics and preliminary test:-

1. State - Liquid.
2. Colour - brown.
3. Odour - Characteristic bad smell.
4. Miscibility - Immiscible with water but soluble in hcl .
5. Litmus - Neutral.
6. Action of heat - Volatilises without leaving any residue.
7. Ignition test - yellow sooty flame.

■ Confirming test:-

Experiment	Observation
<u>malachite green test:-</u> 0.5 ml of Benzaldehyde is heated with 1 ml of dimethyl aniline and a small bit of anhydrous $ZnCl_2$ in a dry test tube for one minute. The leucobase produced is oxidised with lead dioxide in a solution of acetic acid and excess cone. HCl is added.	An intense green colouration formed

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NITROBENZENE (NO_2)

■ Physical characteristics and preliminary test :-

1. State - Liquid.
2. Colour - pale yellow.
3. odour - characteristics bad smell.
4. Miscibility - immiscible with water.
5. Litmus - Neutral
6. Action of heat - volatilises without leaving any residue.
7. Ignition test - yellow sooty flame.

■ Confirming test :-

Experiment	Observation
<p><u>Muliken-Barker test</u> :- 1.0 g of nitro is dissolved in 5 ml 50% alcohol, a little solid Mg solution and a pinch of boiled for a few minutes, cooled and allowed to stand for 5 minutes and then filtered with the filtrate the following three tests are performed.</p>	
a) A portion of the solution is added to Tollens reagent and then warmed in a water bath.	a) A black ppt formed.
b) Two drops of benzoylchloride and 2 drops of conc. HCl are added to another portion of the filtrate followed by 1-2 drops of FeCl_3 solution.	b) A wine red colour of ferric hydroxamate formed.
c) The last portion of the filtrate is warmed with a little ferric solution.	c) Red ppt. formed.

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GLUCOSE, $\text{HOCH}_2(\text{CHOH})_4\text{CHO}$

■ physical characteristic and preliminary test :-

1. State - Solid.
2. Colour - White.
3. Texture - crystalline.
4. Odour - Sweet smell.
5. Solubility - In water.
6. Litmus - Neutral.
7. Action of heat - melts brown due to charring and gives smell of burnt sugar.
8. Ignition test - Blue - non sooty flame.

■ Confirming test :-

Experiment	Observation
<u>osazone test</u> :- 2 ml of 1g of phenylhydrazine hydrochloride and 1g of sodium acetate are added to 0.5 of Glucose dissolved in 5ml of water and the mixture is then shaken to obtain clear solution. The test tube is then heated in a boiling water-bath for 10 minute.	yellow p.p.t Crystalline out of hot solution.

STARCH (C₆H₁₀O₅)_n■ physical characteristic and preliminary test :-

1. State - Solid.
2. Colour - White.
3. Texture - powder.
4. Odour - Odourless.
5. Solubility - Insoluble in any organic solvent from sticky coagulation with hot water.
6. Litmus - Neutral.
7. Action of heat - liquified and gives pungent smell.
8. Ignition test - Blue and sooty flame.

■ Confirming test :-starch-iodide test :-

A drop of dilute iodine in potassium iodide solution is added to 2 ml aqueous solution of starch.

A deep blue colour formed the colour disappears on boiling the solution and appears on cooling.

SUCROSE ($C_{12}H_{22}O_{11}$)

■ physical Characteristic and preliminary tests:-

1. State - Solid.
2. Colour - Colourless.
3. Texture - Crystalline.
4. Odour - Odourless.
5. Solubility - In water.
6. Litmus - Neutral.
7. Action of heat - Black residue obtained.
8. Ignition test - yellow non-sooty flame.

■ Confirming test:-

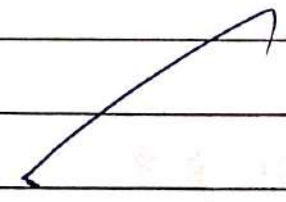
Experiment	Observation
<u>Lead acetate test:-</u> Lead acetate solution is added to a solution of sucrose and boiled for few seconds. Then dil NH_4OH is added dropwise and the mixture is boiled.	White p.p.t formed first which does not change its colour upon heating.

OXALIC ACID ($\begin{matrix} \text{COOH} \\ | \\ \text{COOH} \end{matrix}, 2\text{H}_2\text{O}$)

■ physical characteristic and preliminary test:-

1. State - Solid.
2. Colour - Colourless.
3. Texture - Crystalline.
4. Odour - Odourless.
5. Solubility - Water.
6. Litmus - Blue litmus turns red (acidic)
7. Action of heat - It liberates CO_2 and volatilises
8. Ignition test - Blue non-sooty flame.

■ Confirming test:-

Experiment	Observation
<p><u>potassium permanganate test:</u> A little of dil H_2SO_4 is added to a solution of oxalic acid. The solution is warmed and dil KMnO_4 solution is added drop by drop with shaking.</p>	<p>The pink colour of permanganate disappears.</p> 

SUCCINIC ACID ($\text{HO}_2\text{C} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CO}_2\text{H}$)

■ Physical characteristics and preliminary tests:-

1. State - Solid.
2. Colour - Colourless.
3. Texture - Crystalline
4. Odour - Odourless
5. Solubility - Soluble in water.
6. Litmus - Blue litmus turns red (acidic)
7. Action of heat - melts and then boils giving off extremely irritating vapours.
8. Ignition test - Blue non-sooty flame.

■ Confirming test:-

Experiment	Observation
<p><u>Floures</u> test:- A small amount of 0.5 and twice its weight of resorcinol are mixed thoroughly and taken in a dry test tube. A few drops of conc. H_2SO_4 is added.</p> <p>(a) The mixture is heated gently.</p> <p>(b) The deep red solution is poured in to a large volume of water taken in a beaker.</p> <p>(c) The contents of beaker is made alkaline with NaOH solution.</p>	<p>(a) A deep red solution</p> <p>(b) Deep red colour changes to organic yellow solution which emits an intense green fluorescence</p> <p>(c) The solution changes to bright red and fluorescence</p>

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SALICYLIC ACID O=C(O)c1ccccc1O

Physical Characteristics and preliminary test :-

1. State - Solid.
2. Colour - White.
3. Texture - needle-shaped crystals.
4. Odour - Odourless.
5. Solubility - Sparingly soluble in cold water but readily soluble in hot water.
6. Litmus - Blue litmus turns red (Acidic).
7. Action of heat - melts and volatiles.
8. Ignition test - yellow sooty flame.

Confirming test :-

Experiment	Observation
<u>Oil of wintergreen test :-</u> About 0.1 g of salicylic acid, 1 ml of methanol and few drops of conc. H_2SO_4 are warmed in a clean dry test tube.	A characteristic pungent fragrant odour which intensifies on pouring the mixture to dil. Sodium Carbonate solution.

RESORCINOL ($\text{C}_6\text{H}_4(\text{OH})_2$)

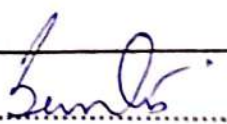
■ physical characteristic and preliminary test:-

1. State - Solid.
2. Colour - Colourless.
3. Texture - powder.
4. odour - Odourless.
5. Solubility - Soluble in cold water.
6. Litmus - Feebly acidic.
7. Action of heat - melts and volatilises.
8. Ignition test - yellow sooty flame.

■ Confirming test:-

Experiment	Observation
<p><u>Fluorescein test:-</u> A small quantity of Resorcinol is mixed with Succinic acid a few drops of conc. H_2SO_4 is added.</p> <p>(a) The mixture is heated gently.</p> <p>(b) The deep red solution is poured into a large volume of water taken in a beaker.</p> <p>(c) The contents of beaker is made alkaline with NaOH solution.</p>	<p>(a) A deep red solution.</p> <p>(b) Deep red colour changes to orange yellow soln. which emits an intense green fluorescence.</p> <p>(c) The solution changes to bright red fluorescence intensified.</p>

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Single detection test for unknown sample

Sample - 1

State - Solid.

Colour - Colourless.

Litmus - No change (Sample is not acidic or basic in nature)

Ignition test - Non sooty flame.

Solid sample

↓ NaHCO₃ (aq)

Since the effervescence of CO₂ did not appear so the sample is not acidic and the test for oxalic acid, succinic acid and salicylic acid was not formed.

As the sample is not acidic the sample may be starch, Glucose, sucrose or resorcinol.

↓ Water

Since the sample is clearly soluble in water the sample may be Glucose, Sucrose or resorcinol.

As the sample is miscible so starch is absent.

I₂/KI

Since no violet colour appears the sample is not starch and the test for starch was not performed.

The sample may be sucrose, Glucose or resorcinol

↓ H₂SO₄

Since black mass appears no black appears

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the sample may be glucose appears the sample
or sucrose. Feeling test is not resorcinol

As the [↓]is negative Glucose is absent.

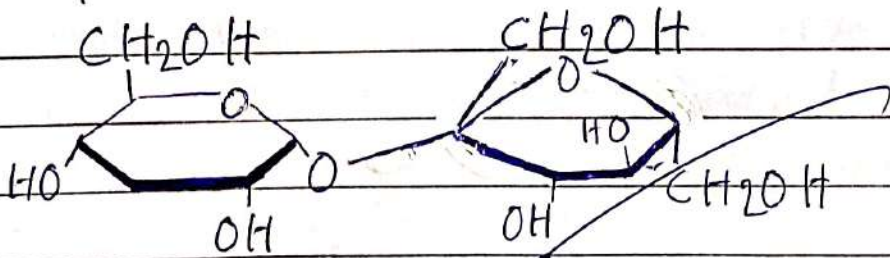
As the result [↓]is negative (i.e.) no ppt appears so the sample is sucrose.

■ Confirmatory test :-

Experiment	Observation	Inference
<u>Lead acetate test</u> :- Lead acetate sol ⁿ is added to a sol ⁿ of unknown sample and boiled for few seconds. Then dil NH ₄ OH is added dropwise till a permanent white ppt. appears then the mixture is boiled.	The white ppt does not change colour.	The unknown sample is sucrose.

■ The unknown Compound Name - Sucrose.

■ Structure -



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Single detection of unknown compound

Sample-2

State - Liquid.

Colour - Brown.

Litmus - No colour change (Sample is not acidic or basic in nature)

Ignition test - yellow sooty flame (Maybe the compound is aromatic)

Liquid sample

↓ H₂O

Since the compound is immiscible in water, so the test for formic acid and methanal and ethanal was not performed.

As the compound is immiscible in water the compound may be Aniline, Dimethyl aniline Benzaldehyde or nitrobenzene

Warmed with HCl

↓

Since the compound is miscible in dil HCl so the compound may be aniline or dimethyl aniline.

Since the compound is miscible in dil HCl the test for benzaldehyde and nitrobenzene was not performed.

↓ malachite green test was done

↓

↓

Since no colourisation so the sample was not NiN-dimethyl aniline.

As no colourisation so the sample is aniline.

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