BVR SIR's

Microscale Organic Compound Analysis Procedure

Government College (A), Rajahmundry

Syllabus: Analysis of an organic compound through systematic qualitative procedure for functional group identification including the determination of melting point and boiling point with suitable derivatives. Alcohols, Phenols, Aldehydes, Ketones, Carboxylic acids, Aromatic primary amines, amides and simple sugars.

In carrying out identification of an organic compound following tests and observations are carried out:
1.Preliminary Tests and Physical Examination. 2. Determination of Physical Constants (M.P/B.P).
3. Detection of Elements. 4. Determination of Solubility Group.
5. Detection of Functional Group. 6. Special Tests, if any. 7. Derivatives preparation.

1.Preliminary Tests and Physical Examination:

Experiment	Observation	Inference
A) Physical State:	i. Solid:	Presence of high molecular weight organic compound (usually having more than 6 carbon atoms) . Eg: Acids, Sugars, Amides, etc.
	ii. Liquid:	Presence of low molecular weight organic compounds like Alcohols, Ethers, Esters, Aliphatic amines, Aldehydes, Ketones, Hydrocarbons, etc.
B) Colour: It gives an idea about the compound.	i.Yellow colour. ii. Brown-dark. iii. Colourless.	Nitro compounds. Phenols, amines. Compounds not containing strong Chromophore. Eg: Acids, Hydrocarbons, ketones, esters, urea, thiourea, acetamide, acetanilide, benzamide, naphalene, etc.,
C) Odour: Some organic compounds, especially liquids will have characteristic odours. Some times the smell itself will give an idea about the compound.	i.Smell of vinegar. ii. Pungent fruity odour. iii. Pleasant fruity odour. iv. Smell of bitter almonds. v. Characteristic strong odour. vi. Pleasant almond like odour. vii. Mousy odour.	Acetic acid. Acetaldehyde. Esters. Nitrobenzene. Aniline. Benzaldehyde. Acetamide.

CAUTION: • Do not taste an unknown compound. • To note the odour, cautiously smell the cap of the container and do it only once. Never smell the contents of the container directly.

2. Determination of melting point of an Organic Compound:

The temperature at which the chemical compound changes its state from solid to liquid is said to be the melting point of the compound. Let us learn to determine the melting point of solid organic compounds like naphthalene, benzoic acid, glucose, etc.,

Materials Required: Solid organic compound, boiling tube, stand with clamp, capillary tube, tripod, thermometer, sulphuric acid and burner, etc.,

The Procedure:

First the crystalline substance is powdered. Then take a capillary tube and seal one end by heating it.

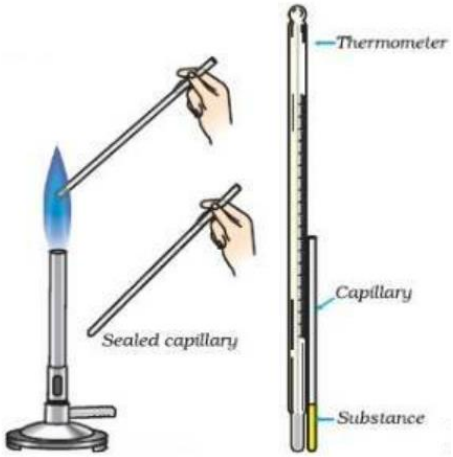
Fill the capillary tube with the substance. Fill the capillary tube upto 2-3 mm. Attach the capillary tube to a thermometer using a thread. Take liquid paraffin in a beaker and place it over a piece of wire gauze placed over a tripod stand. Clamp the thermometer carrying the capillary tube to an iron stand and immerse them in the bath of liquid paraffin. Heat the beaker slowly while constantly stirring the contents using a stirrer to maintain a uniform temperature throughout. The temperature rises slowly. Note the temperature (t_1) when the substance starts melting. Again note the temperature (t_2) when the substance has completely melted. The average of the two readings gives the correct melting point of the substance.

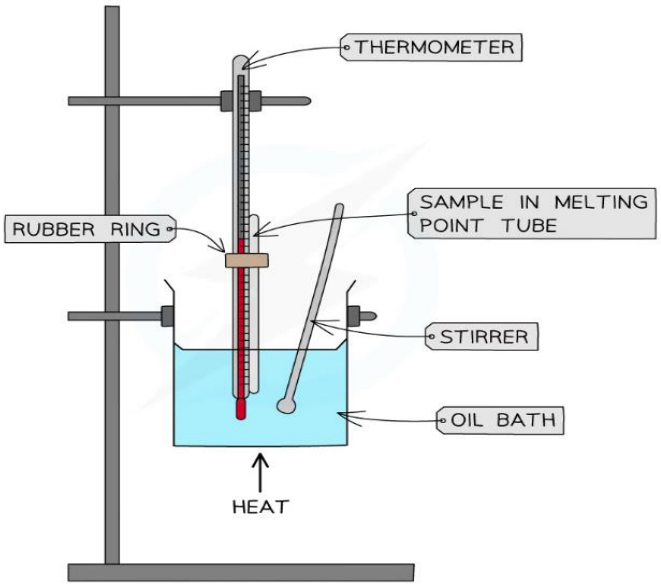
Observations: Record your observations in the table given below.

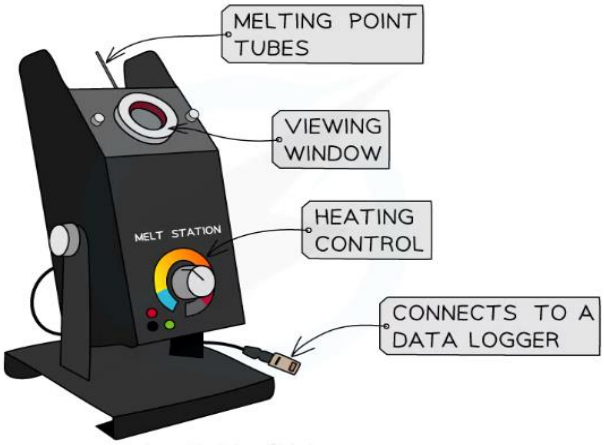
Note the temperature when the substance;		Melting point of the given substance ($t_1 + t_2 / 2$ (^oC)
Starts melting $t_1(^0C)$	Has completely melted t_2 (^0C)	

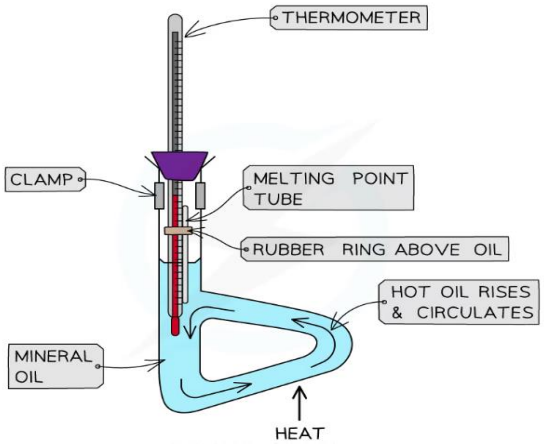
Inference: Melting point of the given substance = ... ^oC

Youtube link: https://www.youtube.com/watch?v=nQNaTfqXECk









Precautions: Use dry and powdered sample for the determination of melting point. Keep the lower end of the capillary tube and the thermometer at the same level. Packing of the powder should be uniform without any large air gaps in between the solid particles.

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3. Determination of the boiling point of the given organic compound:

Requirements- Liquid paraffin, given organic compound, Beaker, thermometer.

Theory- The boiling point of a compound is the temperature at which it changes from liquid to its vapour. The boiling point of a liquid is the temperature at which its vapour pressure becomes equal to atmospheric pressure. It is the property often used to check the purity of the given organic compound.

Procedure: 1. Take a small amount of liquid in an ignition tube and place a capillary tube sealed at one of its end in an inverted position in the same ignition tube. 2. Attach the ignition tube with the thermometer by means of a rubber band. 3. Introduce whole of the arrangement into the beaker containing liquid paraffin. 4. Heat the beaker gently with constant stirring until a stream of bubble gas goes outside the capillary tube rapidly. Note down the temperature as boiling point of the organic liquid.

Precautions: 1. Capillary tube must be sealed at one end. 2. Capillary must be placed in an inverted position. 3. Heating should be slow and uniform.

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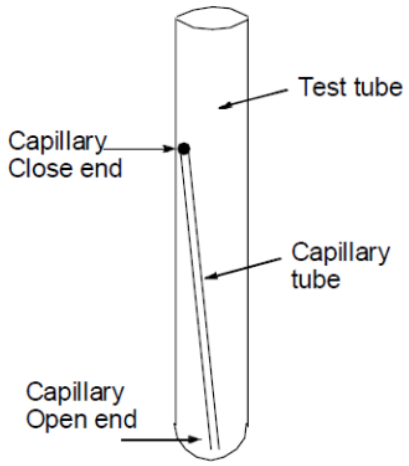

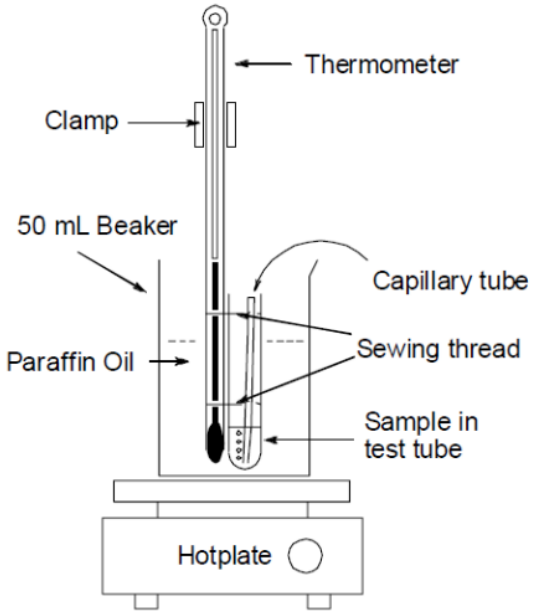

4.Test For Aromaticity:

Experiment	Observation	Inference
A) Substance is introduced into the flame using a nickel spatula.	i.Burns with a smoky flame. ii. Burns with non-smoky flame.	Presence of aromatic compound. Presence of aliphatic compound.
B) To the substance a mixture of 3 drops of concentrated sulphuric acid and 3 drops of concentrated nitric acid are added and warmed on a water bath for about 10 minutes. Then the solution is poured into water.	An yellow solution or precipitate is observed.	Presence of aromatic compound.

5. Test For Unsaturation:

Experiment	Observation	Inference
a) A little of the substance is taken on a tile. A drop of bromine water is added to it.	i. Decolorisation.	Presence of unsaturation.
	ii. Decolorisation followed by turbidity formation.	Presence of aniline or phenol.
b) To a pinch of the substance on the tile, a drop of dilute potassium permanganate solution is added.	Decolorisation occurs.	Presence of unsaturation or easily oxidizable compound.

6. Test for extra elements: Sodium fusion extract tests, (Lassaigne's test):

Two small cut pieces of sodium are fused in a semimicro hard glass tube. About 5 mg of the substance is added to it and fused again. The tube is cooled to room temperature. Keeping the tube in a slanting position 4

drops of water are added. (the first drop of water is allowed to react with excess of sodium. The second dop is added after the initial reaction is over. Then the third and the fourth drops of water are added slowly). This is the sodium fusion extract. The following tests are performed with it.

Green procedure for identification of Extra elements:

To 5 mg of the substance, Zn dust and Sodium carbonate are added and fused. The tube is cooled to room temperature. Keeping the tube in a slanting position, 4 drops of water is added and filtered. The following tests are performed with it.

Experiment	Observation	Inference
A) Test for nitrogen: A drop of the extract is placed on a tile. A drop of a concentrated solution of ferrous sulphate is added to it. Then it is treated with a drop of 50% Sulphuric acid.	Prussian blue colour	Presence of nitrogen.
B) Test for halogens: A drop of the extract is placed on a tile. A drop of concentrated nitric acid is added followed by a drop of silver nitrate solution.	i.Curdy white precipitate. ii. Pale yellow precipitate. iii. Yellow precipitate.	Presence of chlorine. Presence of bromine. Presence of iodine.
C) Test for Sulphur: A drop of the extract is placed on a tile and to it a drop of sodium nitroprusside is added.	Appearance of violet colour	Presence of sulphur.

7. Solubility Tests: Solubility of the substance tested in the following solvents:

Experiment	Observation	Inference
i.Water	Soluble	Urea, carbohydrates, etc. present
ii. 5% Sodium bicarbonate	Soluble	Presence of carboxylic acids.
iii. 5% Sodium hydroxide	Soluble	Presence of carboxylic acids.
iv. 5% Hydrochloric acid	Soluble	Presence of amines.

8. Litmus paper test:

Experiment	Observation	Inference
The substance is brought into contact with moistened litmus paper.	i.Blue litmus turns red. ii. Red litmus turns blue. iii. Neutral to litmus.	May be due to acids, phenols. May be due to amines. Presence of carbohydrates, esters, carbonyl compounds, etc.,

Tests for Functional Groups:

1. Test for carboxylic acids:

Experiment	Observation	Inference
A)About 5 mg of the substance is treated with a drop of saturated sodium bicarbonate solution on a tile.	Brisk effervescence evolved.	Presence of carboxylic acid.
B) A drop of neutral ferric chloride solution is added to a little substance.	i. Violet colour ii. Flesh colour.	Presence of salicylic acid. Acids like phthalic acid.

2. Test for Phenols:

Experiment	Observation	Inference
A)Neutral Ferric chloride test : A drop of the substance is mixed with a drop of neutral ferric chloride solution on a tile.	Violet colour appears.	Presence of phenol.
B) Phthalein Reaction: About 5 mg of the substance is heated with about 10 mg of phthalic anhydride and a drop of concentrated sulphuric acid in a semimicro tube. The solution is cooled and diluted with about 1 ml of water. A drop of the solution is mixed with a drop of 50%, sodium hydroxide solution on a tile.	Red, blue or green colour is produced.	Presence of phenol.
C) Liebermann's reaction : 5mg of the substance is heated with 5 mg of sodium nitrite and 2 drops of con. sulphuric acid in a semi micro tube. It is cooled and diluted with 1 ml of water. A drop of it is placed on a tile and mixed with a drop of 10% NaOH.	A bluish green colour is produced.	Presence of phenol.

3. Test for Alcohols:

Experiment	Observation	Inference
Ceric ammonium nitrate test: To the organic compound on a tile, a drop of ceric	Red coloured solution appears.	Presence of alcohol.

ammonium nitrate solution is added.		

4. Test for aldehydes / ketones (Carbonyl compounds):

Experiment	Observation	Inference
A) Borsche's reagent test: A drop of the substance (if it is a liquid) or a drop of alcoholic solution of the substance is placed on a tile. A drop of a concentrated solution of 2, 4 - DNP (Borsche's reagent) is added to it.	Red / orange precipitate	Presence of aldehyde / ketone.
B) Schiff's reagent test : A drop of the substance is of placed on a tile. A drop of Schiff's reagent is mixed with it.	Pink coloration.	Presence of aldehyde.
C) Fehling's Test: 2 drops or about 5mg of the substance is mixed with 2 drops of Fehling A and 2 drops of Fehling B solutions in a Semimicro test tube. It is heated in a water bath for about 5 minutes.	Red precipitate.	Presence of aldehyde / reducing sugars.

5. Test for Carbohydrates:

Experiment	Observation	Inference
1)Action of Sulphuric Acid : About 5mg of the substance is warmed with 3 drops of concentrated sulphuric acid.	Substance chars with the smell of burnt sugar.	Presence of carbohydrates.
2) Molisch's Test: A drop of an alcoholic solution of α - naphthol is mixed with a drop of the aqueous solution of the substance on a tile. This mixed solution is allowed to come in contact with a drop of concentrated sulphuric acid.	A deep blue coloration.	Presence of sugars (carbohydrates).

6. Test for Aromatic Primary Amines:

Experiment	Observation	Inference
Dye test: A drop of the substance is placed on a tile. A drop of dilute hydrochloric acid is added to it. A drop of saturated sodium nitrite solution followed by a drop of β -naphthol dissolved in 25% sodium hydroxide is added to it.	Red azo-dye is formed.	Presence of aromatic primary amine.

7. Test for Amides:

Experiment	Observation	Inference
Biuret test : About 5 mg of the substance is heated strongly in a DRY semi micro tube to its melting point. Cooled, dissolve residue in 3 drops of water. A drop of it is mixed with a drop of dilute copper sulphate and a drop of dilute sodium hydroxide solution on a tile.	Violet colour is observed.	Presence of Urea amides.

Preparation of Derivatives:

A)For Carboxylic Acids:

1.Amide Derivative : 0.5 g of the substance is mixed with twice its weight of phosphorous pentachloride in a DRY boiling tube with the help of a glass rod. The mixture is gently warmed for a minute. It is cooled and a few ml of liquor ammonia are added drop by drop, carefully. The amide is then recrystallised from hot water.

2.S- Benzylisothiuronium Chloride Derivative: 0.5g of the substance is suspended in 10 ml of hot water. A drop of phenolphthalein is added and then neutralised carefully with dil. sodium hydroxide solution. Then 2 drops of dil. hydrochloric acid are added to make it faintly acidic. This is added to a saturated solution of S-Benzylisothiuronium chloride. The mixture is then cooled till precipitation is completed. It is recrystallised from dil. alcohol acidified with dilute hydrochloric acid.

B) For Phenols:

1.Bromo Derivative: To about 0.5 ml of phenol, bromine water is added slowly with constant stirring until a pale yellow colour persists. The precipitated tribromophenol is filtered and crystallised from dilute alcohol.

2. . Benzoyl Derivative (Schotten - Baumann Reaction): The compound is treated with a little excess of sodium hydroxide (5 ml) in a boiling tube, 1 ml of benzoyl chloride is added in small amounts with constant shaking. The boiling tube is tightly corked and shaken well for 5-10 minutes. The solid is filtered and washed well with water to remove the excess alkali. It is recrystallised from alcohol.

C) For Carbonyl Compounds (Aldehydes and Ketones):

1) 2, 4 - Dinitrophenylhydrazone : About 0.2g of the substance is dissolved in alcohol. To this about 2 ml of Borsche's reagent and a few drops of conentrated hydrochloric acid are added. Heated to boiling and allowed to cool. The precipitate is filtered and recrystallised from alcohol.

2) Phenyl Hydrazone : 1 gm of phenylhydrazine hydrochloride and 1.5 gm of and sodium acetate are dissolved in minimum amount of water. The solution is then added to 0.5 gm of the substance in alcohol. The mixture is shaken well until a clear solution is obtained. Then warmed for about 15 minutes on a water bath and cooled. The precipitate is filtered and recrystallised from dilute alcohol.

D) For Carbohydrates:

1.Osazone Derivative: To 5 ml of a 1% solution of the compound, a mixture of 0.1g of phenylhydrazine hydrochloride and 0.25g of sodium acetate are added. Then 3 drops of glacial acetic acid is also added. The mixture is heated on a boiling water bath for 15 minutes. The osazone is recrystallised from dilute alcohol.

E) For Amines:

1.Benzoyl Derivative (Schotten - Baumann Reaction): The amine is treated with a little excess of sodium hydroxide (5 ml) in a boiling tube, 1 ml of benzoyl chloride is added in small amounts with constant shaking. The boiling tube is tightly corked and shaken well for 5-10 minutes. The solid is filtered and washed well with water to remove the excess alkali. It is recrystallised from alcohol.

2. Bromo Derivative: 1 gm of the substance is dissolved in 1 ml of glacial acetic acid. To this is added bromine in glacial acetic acid till the colour of the bromine persists. After 15 minutes the mixture is poured into cold water. It is filtered and recrystallised from alcohol.

F) For Amides:

1.Nitrate Derivative : To a saturated solution of the amide in water, con. nitric acid is added drop by drop till a precipitate is formed. The crystals are filtered and recrystallised from dilute alcohol.

2. Oxalate Derivative : (2 CO(NH2) 2 . H2C2O4) To a saturated solution of the substance, a saturated solution of oxalic acid is added slowly till a precipitate is formed. It is recrystallised from dil. alcohol.

3. For AROMATIC Amides: Acid Derivative: 1 gm of the substance is hydrolysed by heating with 10 ml of 10% sodium hydroxide and the acid is isolated after acidification with dilute hydrochloric acid. It is recrystallised from hot water.
