



SEPARATION & IDENTIFICATION OF BINARY ORGANIC MIXTURE BY USING SYSTEMATIC QUALITATIVE ANALYSIS APPROVAL

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ABSTRACT

The separation and identification of binary organic mixtures are fundamental processes in organic chemistry, with wide-ranging applications in various industries such as pharmaceuticals, environmental science, and forensics. This project aims to develop a systematic approach for the qualitative analysis of binary organic mixtures, focusing on the separation and identification of individual components based on their unique chemical properties.

The outcomes of this project will contribute to the development of a systematic approach for the qualitative analysis of binary organic mixtures, providing valuable insights into the composition and properties of complex organic systems. The findings will have implications for various fields, including chemical education, analytical chemistry, and industrial applications. Overall, this project will advance our understanding of qualitative analysis techniques and their application in the separation and identification of binary organic mixtures, paving the way for future research and practical applications in organic chemistry.

KEYWORDS

- | | |
|---------------------------|-----------------------------|
| 1) Qualitative Analysis | 6) Systematic Approach |
| 2) Organic Chemistry | 7) Functional Group Tests |
| 3) Binary Mixtures | 8) Chromatography |
| 4) Separation Techniques | 9) Spectroscopy |
| 5) Identification Methods | 10) Fractional Distillation |

1. INTRODUCTION

Systematic quantitative analysis of binary compound mixtures involves separating and identifying components based on physical and chemical properties.

1. Qualitative analysis determines the nature, type, elements, and functional groups present in chemical substances.
2. Quantitative analysis determines the percent composition of various elements in the sample.
3. In organic qualitative analysis, elements or compounds are identified through chemical reactions.

1.1 What is mixture

A mixture arises from the combination of two or more materials or substances without undergoing chemical bonding. This allows them to retain their original properties and be separated by physical means.

1.2. Qualitative Analysis

Qualitative analysis refers to the identification of the chemical elements or compounds present in a sample based on their physical and chemical properties, rather than their quantities. This type of analysis is often used when the exact composition or concentration of substances is not required, but rather the presence or absence of specific components is of interest.

1.3. Binary Compound

Binary compounds consist of two elements only, with the number of atoms of each element not disclosed. The key criterion for binary compounds is that they contain only two elements; the number of atoms and the type of chemical bond are irrelevant. For example, despite containing three atoms, a water molecule is classified as a binary compound because it consists of two elements.

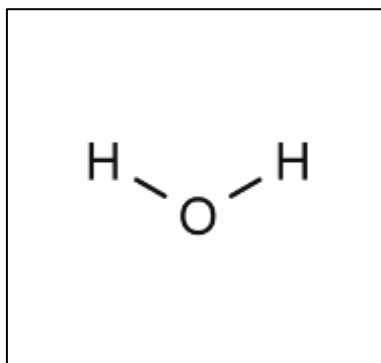


Figure 1: Water consists of three atoms: two hydrogen atoms and one oxygen atom.

1.4 Types of binary compound

Binary compounds encompass a broad category of compounds composed of two elements only. They can be classified into the following categories:

- 1) Binary acid compounds
- 2) Binary ionic compounds
- 3) Binary covalent/molecular compounds

The analysis and identification of unfamiliar organic compounds play a vital role in experimental organic chemistry. Qualitative analysis of organic mixtures typically involves two steps: separation of the mixture and systematic qualitative analysis of individual compounds.

1.4.1 Separation of mixture: The given mixture is separated into two components using a suitable reagent such as water, ether, hydrochloric acid, sodium hydroxide, sodium bicarbonate, or organic solvents.

1.4.2 Systematic qualitative analysis of individual compounds: Separated components in their pure form are analysed through the following steps:

1.4.3 Preliminary investigation: This step provides insights into the unsaturation, aromaticity, and functional groups of compounds. It includes the following tests:

- 1) Nature
- 2) Colour
- 3) Odor
- 4) Flame test
- 5) Test for unsaturation:
 - a. KMnO_4 Test
 - b. Bromine Water Test

1.5 Physical Properties of Substances

The substance to be analysed may be solid, liquid, or gaseous. Since gaseous substances pose difficulties in working and are rare among organic compounds, they will not be emphasized here.

The following properties are required for solid substances:

- 1) Taste
- 2) Odor
- 3) Colour
- 4) Solubility

1.5.1 Taste: Organic substances have unique tastes. Although the toxicity of many substances indicates that this feature is not necessary, determining taste with a small amount of substance in the experiment may not cause toxicity.

- **Bitter taste:** Alkaloids (quinine, morphine, strychnine), quinoline derivatives, glycosides, polyethylene glycol derivatives, barbital.
- **Sweet taste:** Carbohydrates, chloroform, glycol, resorcin, sucrose, sodium salicylate, phenol, etc.

1.5.2 Odor: Many organic substances have characteristic odours, which can indicate their class. For example, the odours of alkalines, esters, phenols, amines, aldehydes, and ketones differ. More volatile substances have more noticeable smells. For instance,



coumarin, eugenol, vanillin, ethyl acetate, methyl salicylate, phenols, thymol, menthol, ethanol, acetone, ether, chloroform, amyl alcohol, and pyridine have distinct odours. Sulphur compounds often have a rotten egg smell (e.g., mercaptans, isonitrile). Benzaldehyde, nitrobenzene, and similar compounds smell like bitter almonds. A strong, penetrating odour often indicates a volatile, small molecule.

1.5.3 Solubility test: Groups can be identified based on their solubility in solvents such as water, ether, hydrochloric acid, sodium hydroxide, and concentrated sulfuric acid.

1.5.4 Detection of extra- element: Detecting extra elements guides the next steps. For instance, the absence of nitrogen suggests avoiding testing nitrogen-containing compounds like amino, amido, nitro, and anilides.

1.5.5 Detection of element: Detecting elements in a binary compound mixture is crucial in qualitative analysis, involving preliminary tests, mixture separation, and element detection.

1.6 Preparation of the Mixture:

- **Accurate Weighing:** Ensure precise weighing of mixture components for correct proportions.
- **Mixing:** Thoroughly mix components for uniform distribution.

1.7 Physical Separation Techniques:

- **Distillation:** Separate based on differences in boiling points.
- **Chromatography:** Separate based on molecular size, shape, and polarity differences.
- **Extraction:** Separate based on solubility differences.

1.8 Identification of Components:

- 1) **Physical Properties:** Assess the physical state, colour, odour, and melting point of each component.
- 2) **Elemental Analysis:** Determine the percentage composition of elements such as carbon (C), hydrogen (H), nitrogen (N), sulphur (S), and halogens in each component.
- 3) **Functional Group Identification:** Identify the functional groups present in each component.
- 4) **Derivative Preparation:** Prepare derivatives of each component to confirm their identity.
- 5) **Melting Point Determination:** Confirm the identity of each component by determining their melting points.

1.9 Qualitative Analysis

- 1) **Elemental Analysis:** Determine the percentage composition of elements such as carbon (C), hydrogen (H), nitrogen (N), sulphur (S), and halogens in each component.
- 2) **Functional Group Analysis:** Quantify parameters like hydroxyl value, amine value, iodine value, saponification value, etc., based on the functional groups present.
- 3) **Spectrophotometric Analysis:** Employ spectroscopic techniques to quantify organic compounds like amino acids, carbohydrates, proteins, ascorbic acid, aspirin, and cholesterol.

1.10 Common techniques used for qualitative analysis of organic compounds

The most common techniques used for qualitative analysis of organic compounds are:

- 1) **Fractional Distillation:** If the two compounds have significantly different boiling points, fractional distillation can be used to separate them. This technique involves heating the mixture and collecting the fractions at different temperature ranges. The collected fractions can then be analysed individually using other techniques.
- 2) **Solubility Tests:** As with single compounds, solubility tests can be useful for binary mixtures. By testing the solubility of the mixture in various solvents, you can determine if one or both of the compounds are soluble in a particular solvent. This can provide initial information about the composition of the mixture.
- 3) **Chromatography:** Techniques like thin-layer chromatography (TLC) or column chromatography can separate the components of a binary mixture based on differences in polarity, size, or other properties. Each compound will elute at a different rate, allowing for separation and subsequent analysis.
- 4) **Fractional Crystallization:** If one compound in the mixture is significantly more soluble in a particular solvent than the other, fractional crystallization can be used. By repeatedly dissolving the mixture in a suitable solvent and allowing it to crystallize, you can separate the two compounds based on their differing solubilities.
- 5) **Spectroscopic Methods:** Techniques like infrared spectroscopy (IR), nuclear magnetic resonance spectroscopy (NMR), and mass spectrometry (MS) can provide information about the functional groups, molecular structure, and mass of the compounds present in the mixture. By comparing the spectra or mass spectra of the mixture to those of known compounds, you can identify the individual components.



- 6) **Chemical Reactions:** Chemical reactions specific to certain functional groups can help identify the presence of those groups in the mixture. For example, if one compound in the mixture contains an alkene group, it will react with bromine water, while the other compound won't.

1.11 Application of qualitative analysis of organic compounds

- 1) **Chemical Education:** Qualitative analysis is often taught in introductory chemistry courses as it provides students with hands-on experience in identifying functional groups, understanding chemical reactions, and interpreting spectroscopic data. It helps students develop essential laboratory skills and critical thinking abilities.
- 2) **Pharmaceuticals:** In the pharmaceutical industry, qualitative analysis is crucial for quality control and assurance. It is used to confirm the identity and purity of active pharmaceutical ingredients (APIs), detect impurities, and monitor the stability of drug formulations. Techniques like chromatography, spectroscopy, and mass spectrometry play a vital role in these analyses.
- 3) **Environmental Analysis:** Monitor and measure organic pollutants in air, water, and soil for environmental protection and remediation.
- 4) **Forensic Analysis:** In forensic science, quantitative analysis is utilized to identify and measure organic compounds in evidence such as drugs, toxins, and residues, assisting in criminal investigations.
- 5) **Pharmaceutical Industry:** Quantitative analysis is crucial for drug development, ensuring precise dosages and purity of active pharmaceutical ingredients in medications.
- 6) **Food Industry:** Quantitative analysis is employed to assess the nutritional content, additives, and contaminants in food products, ensuring food safety and quality.
- 7) **Material Science:** Quantitative analysis aids in characterizing organic materials for applications in electronics, coatings, polymers, and other advanced materials.
- 8) **Chemical Process Optimization:** Industries utilize quantitative analysis to optimize chemical processes, minimize waste, and enhance efficiency in organic compound synthesis.
- 9) **Academic Research:** Quantitative analysis is fundamental in academic research for advancing the understanding of organic compounds, their properties, and their interactions across various scientific disciplines.

1.12 Advantages of systematic qualitative analysis of binary organic compounds

- 1) **Identification of Components:** By systematically analysing the properties and behaviours of a binary mixture, qualitative analysis helps identify the individual components present in the mixture. This information is crucial for understanding the composition of complex mixtures and determining the nature of unknown substances.
- 2) **Confirmation of Presence or Absence:** Qualitative analysis provides confirmation of the presence or absence of specific functional groups or compounds in a mixture. This can help verify hypotheses about the composition of the mixture and guide further analysis or experimentation.
- 3) **Comprehensive Characterization:** Systematic qualitative analysis involves a thorough examination of various chemical and physical properties of the components, including solubility, melting point, boiling point, and reactivity. This comprehensive characterization provides valuable insights into the identity, structure, and properties of the compounds in the mixture.
- 4) **Diagnostic Information:** Qualitative analysis can provide diagnostic information about the functional groups, molecular structure, and reactivity of the components in the mixture. This information is useful for predicting chemical behavior, designing appropriate separation or purification methods, and elucidating reaction mechanisms.
- 5) **Educational Value:** Systematic qualitative analysis is often used as a teaching tool in chemistry education to develop students' analytical skills, critical thinking abilities, and understanding of chemical principles. Hands-on laboratory experiments involving qualitative analysis allow students to apply theoretical knowledge in practical contexts and gain valuable laboratory experience.
- 6) **Versatility and Applicability:** Qualitative analysis techniques can be applied to a wide range of organic compounds and mixtures, making them versatile and applicable across various fields, including chemistry, biology, environmental science, and forensic science. These techniques provide valuable qualitative information that complements quantitative analysis methods and contributes to a comprehensive understanding of complex systems.

1.13 Disadvantages of systematic qualitative analysis of binary organic compounds

- 1) **Time-Consuming:** Qualitative analysis can be a time-consuming process, especially when multiple techniques and tests are required to identify the components of a binary mixture. Each step of the analysis, including sample preparation, testing, and interpretation of results, can require significant time and effort.
- 2) **Labor-Intensive:** Qualitative analysis often involves hands-on laboratory work, which can be labor-intensive, particularly when analysing large numbers of samples or complex mixtures. Skilled personnel are required to perform the analyses accurately and interpret the results effectively.



- 3) **Subjectivity:** Qualitative analysis may involve subjective interpretation of experimental results, particularly when assessing the presence or absence of certain functional groups or compounds based on observed properties or reactions. Different analysts may interpret results differently, leading to variability in conclusions.
- 4) **Limited Quantitative Information:** Unlike quantitative analysis, which provides numerical data about the concentration or amount of specific compounds present in a sample, qualitative analysis primarily yields qualitative information about the identity and properties of compounds. This limitation can hinder the ability to quantitatively compare or measure components in a mixture.
- 5) **Complexity of Interpretation:** Interpreting the results of qualitative analysis can be complex, especially when dealing with mixtures containing multiple components or compounds with similar properties. Differentiating between closely related compounds or determining the exact structure of unknown substances may require additional confirmatory tests or advanced analytical techniques.
- 6) **Limited Sensitivity and Specificity:** Some qualitative analysis techniques may lack sensitivity or specificity for certain compounds or functional groups, leading to false-negative or false-positive results. Care must be taken to choose appropriate analytical methods and interpret results cautiously to minimize errors.
- 7) **Cost:** Qualitative analysis may involve the use of specialized equipment, reagents, and facilities, which can be costly to procure and maintain. Additionally, skilled personnel trained in qualitative analysis techniques may command higher salaries, adding to the overall cost of analysis.

1.14 Limitations of systematic qualitative analysis of binary organic compounds:

- 1) **Complexity of Mixtures:** Binary organic compounds may possess complex compositions, making it difficult to accurately separate and identify individual components, particularly when components share similar physical or chemical properties.
- 2) **Interference of Impurities:** Presence of impurities in the organic mixture can interfere with the qualitative analysis process, leading to inaccurate results and hindering the identification of the true components.
- 3) **Limited Specificity:** Certain qualitative tests may lack specificity, yielding similar results for different functional groups or compounds, thereby introducing ambiguity into the identification process.
- 4) **Subjectivity in Interpretation:** Interpretation of qualitative tests can be subjective, influenced by the observer's judgment, potentially introducing bias and compromising the analysis's reliability.
- 5) **Sensitivity to Experimental Conditions:** Qualitative test outcomes may vary based on experimental conditions such as temperature, pH, and reagent concentrations, necessitating careful control for accurate results.
- 6) **Time-Consuming Process:** Systematic qualitative analysis of binary organic compounds involves multiple tests and procedures, rendering it time-consuming, particularly with complex mixtures.
- 7) **Limited Information:** Qualitative analysis indicates the presence of functional groups and elements but lacks quantification, limiting the depth of information gleaned from the analysis.
- 8) **Need for Expertise:** Executing systematic qualitative analysis demands expertise and experience in organic chemistry to interpret results accurately, which may present a challenge for less experienced analysts.

2. Experimental Work

- **Aim:** The aim of the systematic quantitative analysis of ternary organic compound mixtures is to develop a structured and reliable method.
- **Objective**
 1. **Purity Assessment:** It helps in assessing the purity of the compounds present in the mixture by quantifying the impurities or other elements that may be present.
 2. **Research and Development:** By accurately quantifying the elements present, researchers can better understand the properties and behaviour of the compounds, aiding in the development of new materials or products.
 3. **Forensic Analysis:** In forensic science, quantitative analysis helps in identifying and quantifying substances found at crime scenes, providing crucial evidence for investigations.

2.1 Requirement

2.1.1 Chemical

- | | |
|---------------------------------|-------------------------------------|
| 1) Dilute KMnO_4 | 10) Lead Acetate Solution |
| 2) Bromine water | 11) Nitroprusside Solution |
| 3) Saturated NaHCO_3 | 12) Dilute HNO_3 |
| 4) Conc. HCL | 13) AgNO_3 solution |
| 5) FeCl_3 | 14) CHCl_3 |
| 6) NaOH Solution | 15) Ferus Sulphate |
| 7) FeSO_4 Solution | 16) Molish Reagent |
| 8) Dil. H_2SO_4 | 17) Tollens Reagent |
| 9) Acetic Acid | 18) Potassium permanganate Solution |



2.1.2 Apparatus

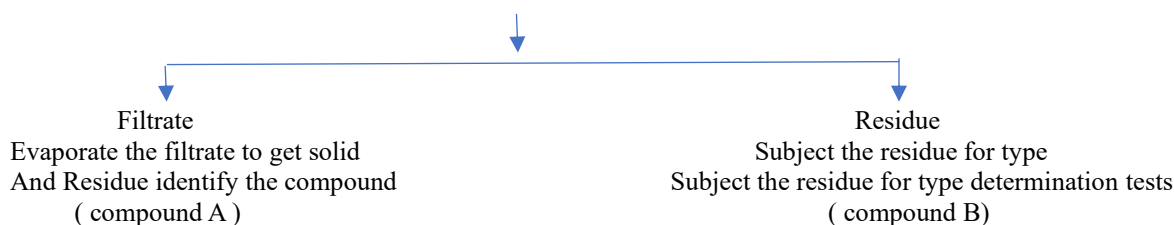
1. Beaker
2. Glass rod
3. Measuring Cylinder
4. Funnel
5. Tripod Stand
6. Test Tube
7. Test Tube Stand
8. Wire Gauze
9. Water bath
10. Fusion Tube
11. Capillary Tube
12. Dropping Funnel
13. China dish
14. Test Tube Holder
15. Spatula

2.2 Separation of organic compound

Separation of solid-solid mixture:

(a) Separation of solid-solid (mixture with water soluble compound) mixture:

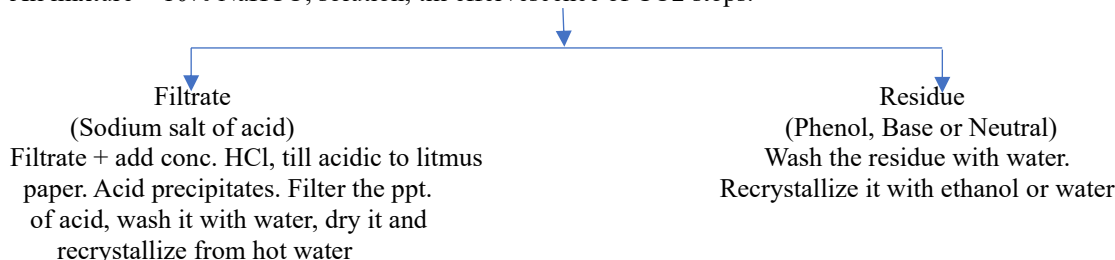
Place the mixture in 50 ml water in a beaker. Stir it for 10-15 minutes and filter.



b) Separation of solid-solid (water insoluble) mixture:

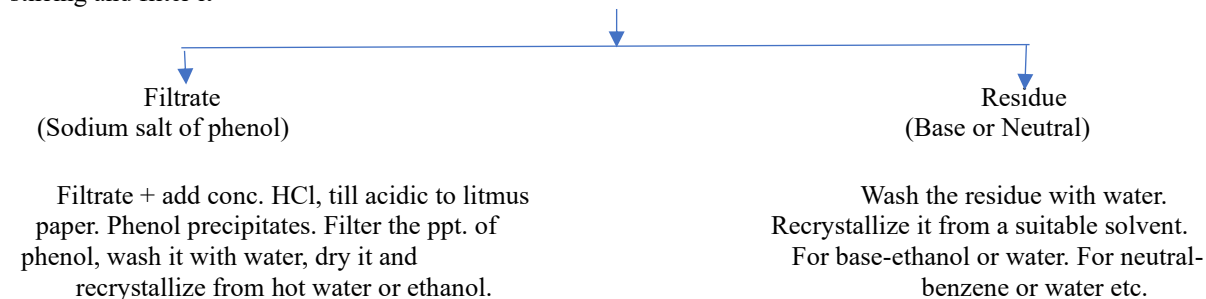
(i) Separation of Acid - Phenol / Acid - Base / Acid - Neutral type mixture:

All mixture + 10% NaHCO₃ solution, till effervescence of CO₂ stops.



(ii) Separation of Phenol-Base / Phenol-Neutral type mixture:

All mixture + 10% NaOH solution, till alkaline to litmus and add about 50 ml distilled water with stirring and filter it



(iii) Separation of Base-Neutral type mixture:

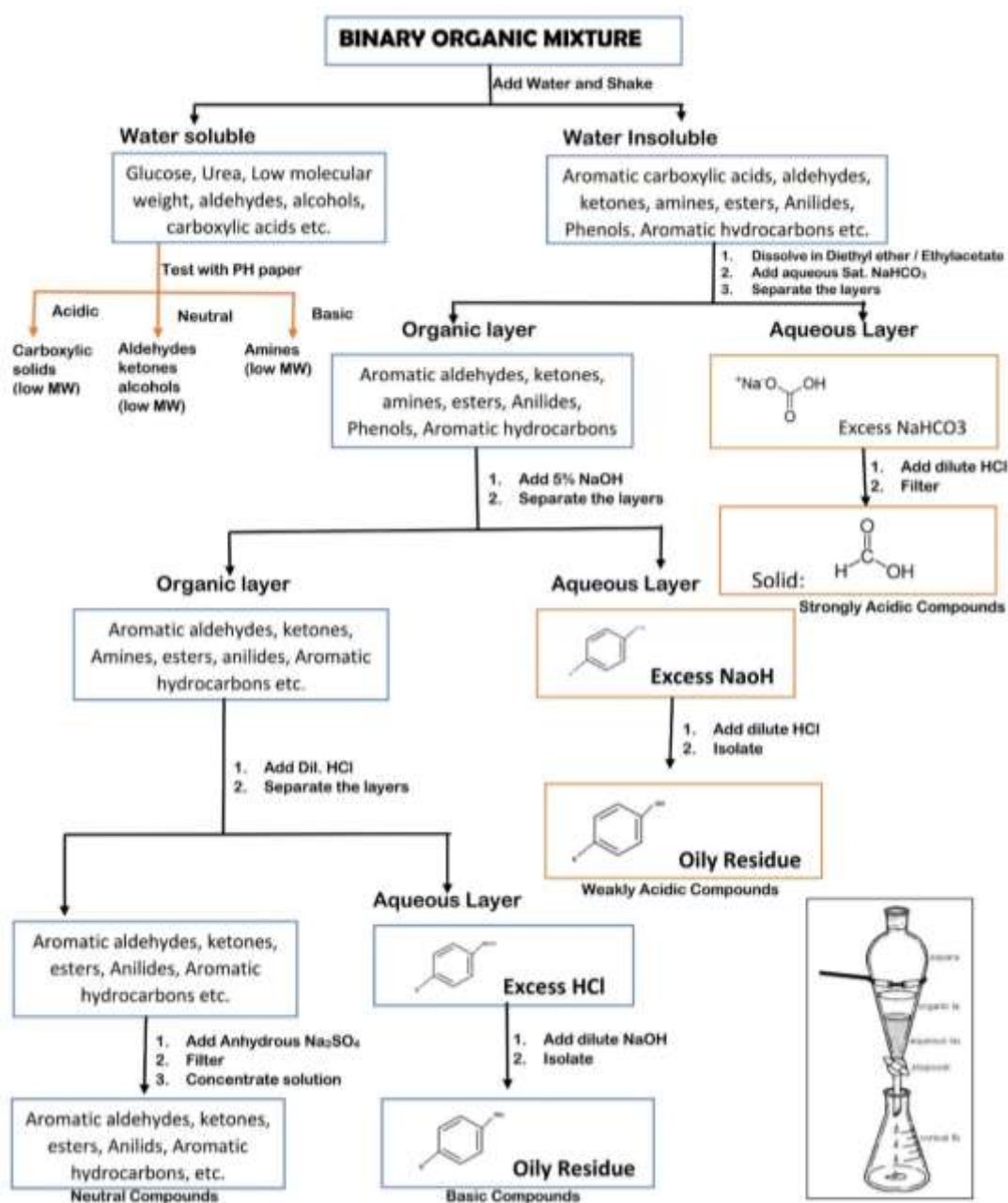
All mixture + 1:1 aq. solution of HCl + 50 ml of water. Stir the mixture for 10-15 minutes and filter it.

Filtrate
(Base)

Filtrate 10% NaOH till alkaline to litmus paper. Filter the ppt, wash it with water, dry it and recrystallize it from ethanol or ethanol- water

Residue
(Neutral)

Wash the residue with water.
Recrystallize from a suitable solvent.



**3. Organic compound identification follows a systematic scheme outlined below:**

1. Preliminary Test.
2. Solubility Test.
3. Detection of Extra Elements.
4. Detection of Functional Group.
5. Physical constants determination and compound identification.
6. Confirmatory Test.
7. Derivatives preparation and its m.pt determination.

3.1 Preliminary Test for Compound A:

Test	Observation	Inference
(a) Nature	i) Solid	Carbohydrate, acid, phenol, amine, higher hydrocarbon may be present.
(b) Colour	Colourless	Simple acid, alcohol, ester, ketone aromatic hydrocarbon.
(c) Odour	Moth balls	Naphthalene
(d) Flame Test	i) Sooty flame	Indicates the presence of an aromatic compound or an aliphatic compound containing a small proportion of hydrogen, such as CHCl_3 or CCl_4 .
(f) Test for unsaturation: 1. KMnO_4 test. Substance + 2 ml of water shake well + 2 drops of dilute KMnO_4 solution.	Decolourisation of KMnO_4	Unsaturated or easily oxidizable compound.
2. Bromine water Test: Substance + 2 ml of water shake well + 2 drops of bromine water.	Decolourisation of bromine water.	Unsaturated compound.

Conclusion: Unsaturated aromatic compound may be present.

3.2 Solubility Test

the substance exhibits insolubility or immiscibility in water.

Test	Observation	Inference
(a) 0.1 gm of substance + 3 ml Saturated NaHCO_3 solution. Shake well. The substance dissolves. To this clear solution add conc. HCl drop by drop.	No strong effervescence	Carboxylic acid Absent. Carboxylic acid Absent.
(b) 0.1 gm of substance + 3 ml dilute NaOH solution. Shake well. The substance dissolves. To this clear solution add conc. HCl drop by drop.	A solid or emulsion not appeared	Phenol Absent.
(c) 0.1 gm of substance + 3 ml 1:1 HCl solution. Shake well. The substance dissolves. To this clear solution add 20% NaOH solution drop by drop.	Solid is not appeared	Base Absent.
(d) If substance is insoluble in NaHCO_3 , NaOH , HCl solution.		Neutral compound present.

Conclusion: Neutral compound may be present.

3.3 Detection of Extra Elements: The Lassaigne's test is a chemical analysis technique used to detect the presence of extra elements like halogens, nitrogen, and sulphur in organic compounds. By fusing the organic compound with sodium metal, these elements are converted into their ionic forms, allowing for their detection through specific chemical reactions. The resulting extract, known as Lassaigne's extract, is then subjected to various tests



to identify the presence of halogens (chlorine, bromine, iodine), nitrogen, and sulphur based on characteristic reactions with reagents like silver nitrate, sodium nitroprusside, and ferrous sulphate.

Sodium Extract:

- 1) A small piece of freshly cut sodium metal is placed in an ignition tube along with the solid organic compound sample.
- 2) The tube is heated strongly with a Bunsen burner until the contents become red hot.
- 3) The hot tube is then quickly plunged into cold water in a beaker to quench the reaction
- 4) The contents are boiled with water for about 10 minutes.
- 5) The mixture is filtered to obtain the sodium fusion extract, also known as the Lassaigne extract.

Detection of Elements

Test	Observation	Inference
Test for Nitrogen: 1 ml of extract + 2-3 drops of NaOH solution to make it alkaline + a few drops of freshly prepared FeSO ₄ solution, boil for a few minutes, cool and acidify it with by adding dil. HCl or dil. H ₂ SO ₄ .	No Blue or green colour solution or Prussian blue coloration.	Nitrogen Absent.
Test for Sulphur: (i) 1 ml of extract + 1 ml of 2N Acetic Acid + 1 ml of Lead Acetate Solution.	No black precipitate.	Sulphur Absent.
(ii) 1 ml of extract + 1 drop of sodium nitroprusside solution.	No violet or purple coloration.	Sulphur Absent.
(iii) 1 ml of extract + 1 ml of aqueous FeCl ₃ solution.	No blood red coloration.	Nitrogen and Sulphur are Absent.
Test for Halogen: (i) 1 ml of extract + 1 ml of dilute HNO ₃ (boil well if N and S are present) + 1 ml of 5% AgNO ₃ solution.	No thick white precipitate.	Halogen Absent.
(ii) If halogen is present carry out the following test: 1ml of extract + 1ml of dilute H ₂ SO ₄ + 0.5ml of CHCl ₃ and 0.5ml of chlorine water, shake well and observe the colour of chloroform layer.	(i) No colour	Iodine Absent.
	(ii) No colour	Bromine Absent.
	(iii) No colour	Chlorine Absent.

Conclusion: The elemental composition of sample A is C,H and no any other element is present.

3.4 Detection of Functional Group test:

The atom or group of atom that defines the structure of particular family of organic compound and at the same time determines their properties is called functional group. It is also possible that a compound contains two or more identical or different functional group which are said to be poly-functional groups. Some of the important functional groups and their characteristic tests and reactions for their identification are discussed below.

Classify the provided compound based on its elemental composition.

Based on the elements found within the organic compound, it categorizes into one of four groups, each potentially further divided into subgroups as follows:

Group I: C, H, (O)	Group III: C, H, (O), N and S
(i). Carboxylic acids (ii). Phenol (iii). Neutrals	(i). Acids (ii). Neutrals
Group II: C, H, (O) and N	Group IV: C, H, (O) and Halogen
(i)Carboxylic acids (ii)Phenols (iii)Bases (iv)Neutrals	(i). Neutrals



3.5 Detection of Functional Groups

Group I: C, H, (O) Neutrals		
Test	Observation	Inferences
<p>(a) Test for Carbohydrates: Molish Test: (Perform this test only if the compound is colorless and soluble in water) Dissolve 0.5 gm of the compound in 2ml of water + 2/3 drops of 10% α-naphthol dissolved in ethyl alcohol, add carefully 1 ml of conc. H_2SO_4 along the sides of the test tube.</p>	A Violet ring is not appeared at the junction of two layers.	Carbohydrate Absent.
<p>(b) Test for Aldehydes and Ketones: (i) 0.05 gm of the compound + 3 ml of 2,4-dinitrophenyl hydrazine. Shake well.</p>	No precipitate.	Aldehyde or Ketone Absent.
If the result of this test is affirmative, proceed with the subsequent test to differentiate between aldehydes and ketones.		
<p>Test for Aldehydes: (i) Schiff's Test: 0.05 gm of the compound + 2/3 ml of Schiff's Reagent Shake well.</p>	violet color is not developed	Aliphatic aldehyde. Absent.
	Pink color is not developed	Aromatic aldehyde Absent.
<p>(ii) Tollen's Test OR Silver (i) Mirror test: 0.1 gm of the compound + 2-3ml Tollens reagent (i.e. Ammoniacal silver Nitrate solution) + Heat it on a boiling water bath.</p> <p>(iii) Fehling Solution Test: 0.1gm of the compound + 1ml Fehling A + 1ml Fehling B solution. Heat it gently</p> <p>(iv) Benedict's test: 0.1gm of the compound + Benedicts solution + Heat it gently.</p>	<p>A silver mirror is Not formed on the inner sides of the test tube</p> <p>No Formation of red ppt of Cuprous oxide</p> <p>No Formation of red ppt Cuprous oxide</p>	Aldehyde Absent.
<p>Test for Ketones: 0.1gm of compound + 2ml of sodium nitroprusside solution + 2 drops of NaOH</p>	Wine Red color or Orange red color is not appeared. CH_3-CO- gr gives this test	Ketone Absent.
<p>(c). Test for Esters: Dissolve 0.1gm or 0.5ml of compound in 1 ml of ethyl alcohol + a drop of phenolphthalein + 2 drops of very dilute NaOH solution. Heat on a boiling water bath.</p>	No color	Ester Absent
<p>(d). Test for Alcohols: (i). Place a small piece of dry sodium metal into a fusion tube and introduce a few drops of the compound. (ii). 1ml of acetyl chloride in a dry test tube + drops of the compound.</p>	<p>No evolution of H_2</p> <p>Weak effervescence</p>	<p>Alcohol Absent</p> <p>Alcohol Absent</p>



e). Test for Hydrocarbons 0.5ml of compound + 1 ml of iodine in carbon disulfide, shake well	Purple color of CS ₂ layer changes to brown color is not appeared.	Ether Absent
(f). Test for Hydrocarbons 0.1 gm of compound + 1-2 ml of water, shake well + 1-2 drops of very very dilute KMnO ₄ solution. Shake again.	If all the above tests fail	Hydrocarbon present
	Decolourisation	Unsaturated hydrocarbon present.
	No decolourisation	Saturated hydrocarbon Absent

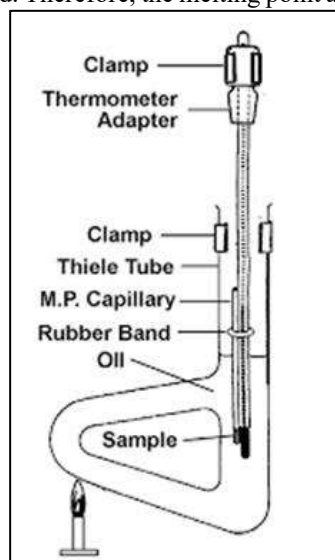
Conclusion: Unsaturated Hydrocarbon may be p

4. DETERMINATION OF PHYSICAL CONSTANTS AND COMPOUND IDENTIFICATION

Aim: To ascertain the melting point of the provided solid substance.

Material Required: Given Solid Substance, Capillary Tube, Paraffin, Laboratory thermometer.

Theory: The change from solid to liquid state of a compound in heating is called melting and the temperature at which a solid in its pure form melts is called the melting point. Each pure solid possesses a distinct melting point, thus the determination of melting point aids in the identification of the compound. The presence of impurities decreases the melting point of a solid. Therefore, the melting point also acts as an indicator of a compound's purity.



Procedure

- 1) Thiele's tube is the preferred apparatus for determining melting points. It comprises a hard glass test tube fused with V-shaped glass tubes at both the middle and bottom ends.
- 2) The tube can be sealed with a one-holed cork housing a sensitive mercury thermometer, featuring a side slit. Approximately three-fourths of Thiele's tube is filled with a high-boiling liquid such as concentrated sulfuric acid or liquid paraffin.
- 3) To determine the melting point of a substance, it's first powdered thoroughly. Then, a capillary tube, about 5 cm in length with one end fused, is selected.
- 4) The powdered substance is introduced into the capillary tube to form a column of roughly 0.5 cm.
- 5) The thermometer bulb is moistened with either liquid paraffin or concentrated sulfuric acid. Subsequently, the capillary tube is positioned to adhere to the thermometer in such a way that the bulb and the specimen are in close proximity.
- 6) The apparatus is then sealed with the cork, ensuring the thermometer is submerged in the liquid while the capillary tube remains above the liquid level. The bulb of the thermometer should be positioned at the upper side tube level, with the open end of the capillary tube above the liquid level.
- 7) The apparatus is securely fastened to an iron stand and gently heated by directing a flame at the side tube. As the liquid molecules are heated, uniform heating occurs.



- 8) To prevent high pressure buildup, vapor escapes through the slit in the cork. During heating, the mercury thread gradually rises until, at a particular moment, the solid substance in the capillary tube suddenly contracts and melts. The temperature is then noted as the melting point of the substance.

Observations

Melting point:

1. 80°C
2. 79°C
3. 81°C

Mean Melting point = $(t_1+t_2+t_3)/\text{no. of observation}$

$$\text{Melting point} = \frac{80^\circ\text{C}+79^\circ\text{C}+81^\circ\text{C}}{3}$$

Melting point=80°C

Conclusion: the melting point of benzoic acid is typically around 79-81°C, so 80°C could indeed be considered within the range. The Naphthalene is confirm.

5. CONFIRMATORY TEST COMPOUND (A):

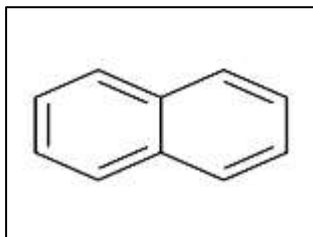
Test	Observation	Inference
Compound + Sodium Bicarbonate	White colour	Naphthalene present.

Conclusion: The conclusion is that Naphthalene is present in the compound being tested. This conclusion is based on the observation of a white colour when the compound is mixed with sodium bicarbonate, indicating the presence of Naphthalene.

Results

From the above step by step analysis of given organic compound shows that, is

Structure:



- **Formula:** C₁₀H₈
- **IUPAC Name:** Naphthalene
- **Other Name:** white tar, camphor tar, naphthene, naphthalin.
- **Molecular Weight:** 128.17 g/mol
- **Category:** Group C, possible human carcinogen
- **State:** Solid state
- **Colour:** White or colourless solid
- **Odour:** Faint, pleasant odour
- **Aromatic / Aliphatic:** Aromatic compound
- **Saturated / unsaturated:** unsaturated
- **Solubility:** Water insoluble.
- **Extra elements:** No present extra element.
- **Functional group:** carboxyl group, benzene ring.
- **Physical Constant:** Melting point: 80°C

**5. PRELIMINARY TEST FOR COMPOUND (B)**

Test	Observation	Inferences
(a) Nature	Solid	Carbohydrate, acid, phenol, amine, higher hydrocarbon may be present.
(b) Colour	No Colour	Simple acid, alcohol, ester, ketone aromatic hydrocarbon.
(c) Odour	No particular smell	Aromatic acid, amide, carbohydrate.
(d) Flame Test	Sooty flame	Aromatic compound or aliphatic compound containing small proportion of hydrogen e.g. CHCl_3 , CCl_4
(f) Test for unsaturation: 1. KMnO_4 test: Substance + 2 ml of water shake well + 2 drops of dilute KMnO_4 solution.	Decolourisation of KMnO_4	Unsaturated or easily oxidizable compound.
2. Bromine water Test: Substance + 2 ml of water shake well + 2 drops of bromine water.	Decolourisation of bromine water.	Unsaturated compound.

Conclusion: Unsaturated Aromatic compound may be present.

5.1 Solubility Test

(A) If the substance dissolves or mixes with water, proceed with the following test.

Test	Observation	Inferences
(a). 0.1gm of substance + 3ml of water shake well. Test the solution with litmus paper.	Blue litmus paper turns red. Red litmus paper is no blue colour.	Water soluble acid or phenol present. Water soluble base Absent.
(b). 0.1gm of substance + Saturated NaHCO_3 solution. Strong effervescence and substance dissolves. To this clear solution add conc. HCl	No solid appear	Water soluble acid present
(c). 0.1gm of substance + water shake well, substance dissolves. To this clear solution add alcoholic FeCl_3 Solution.	No Colour	Water soluble phenol Absent

Conclusion: Water soluble acid or phenol may be present.

5.2 Detection of Extra Element

The Lassaigne's test is a chemical analysis technique used to detect the presence of extra elements like halogens, nitrogen, and sulphur in organic compounds. By fusing the organic compound with sodium metal, these elements are converted into their ionic forms, allowing for their detection through specific chemical reactions. The resulting extract, known as Lassaigne's extract, is then subjected to various tests to identify the presence of halogens (chlorine, bromine, iodine), nitrogen, and sulphur based on characteristic reactions with reagents like silver nitrate, sodium nitroprusside, and ferrous sulphate.

Sodium Extract

- 1) small piece of freshly cut sodium metal is placed in an ignition tube along with the solid organic compound sample.
- 2) The tube is heated strongly with a Bunsen burner until the contents become red hot.
- 3) The hot tube is then quickly plunged into cold water in a beaker to quench the reaction.
- 4) The contents are boiled with water for about 10 minutes.



5) The mixture is filtered to obtain the sodium fusion extract, also known as the Lassaigne extract.

Detection of Elements

Test	Observation	Inference
Test for Nitrogen: 1 ml of extract + 2-3 drops of NaOH solution to make it alkaline + a few drops of freshly prepared FeSO ₄ solution, boil for a few minutes, cool and acidify it with by adding dil. HCl or dil. H ₂ SO ₄ .	No Blue or green colour solution or Prussian blue coloration.	Nitrogen Absent.
Test for Sulphur: (i) 1 ml of extract + 1 ml of 2N Acetic Acid + 1 ml of Lead Acetate Solution.	A black precipitate not Appeared	Sulphur Absent.
(ii) 1 ml of extract + 1 drop of sodium nitroprusside solution.	A violet or purple not appeared.	Sulphur Absent.
(iii) 1 ml of extract + 1 ml of aqueous FeCl ₃ solution.	A blood red not Appeared.	Nitrogen and Sulphur are Absent.
Test for Halogen: (i) 1 ml of extract + 1 ml of dilute HNO ₃ (boil well if N and S are present) + 1 ml of 5% AgNO ₃ solution.	A thick white precipitate is not Appeared.	Halogen Absent.
(ii) When halogen is detected, conduct the subsequent test: Mix 1ml of the extract with 1ml of diluted H ₂ SO ₄ , 0.5ml of CHCl ₃ , and 0.5ml of chlorine water. Shake the mixture thoroughly and observe the color of the chloroform layer.	(i) No colour	Iodine Absent.
	(ii) No colour	Bromine Absent.
	(iii) No Colour	Chlorine Absent.

Conclusion: The elemental composition of sample B is C,H and no any other element is present.

5.3 Detection of Functional Groups test: The atom or group of atom that defines the structure of particular family of organic compound and at the same time determines their properties is called functional group. It is also possible that a compound contains two or more identical or different functional group which are said to be poly-functional groups. Some of the important functional groups and their characteristic tests and reactions for their identification are discussed below.

Classify the provided compound based on its elemental composition.

Based on the elemental composition of the organic compound, it falls into one of four groups, each of which may be further subdivided into subgroups as follows:

Group I: C, H, (O)	Group III: C, H, (O), N and S
(i). Carboxylic acids (ii). Phenol (iii).Neutrals	(i). Acids (ii). Neutrals
Group II: C, H, (O) and N	Group IV: C, H, (O) and Halogen
(i). Carboxylic acids (ii). Phenols (iii).Bases (iv).Neutrals	(i). Neutrals

5.4 Detection of Functional Groups

Group I Test	C, H, (O) Carboxylic acids Observation	Inferences
(a) 0.1gm of substance +3 ml Saturated NaHCO ₃ solution. Shake well. The substance dissolves. To this clear solution add conc. HCl drop by drop.	Strong effervescence A solid appeared	Carboxylic acid present. Carboxylic acid confirmed.
(b) 0.05 gm of compound + 1ml of water,shake well + 1-2 drops of alcoholic FeCl ₃ solution	Buff coloured precipitate.	Benzoic acid or phthalic acid.

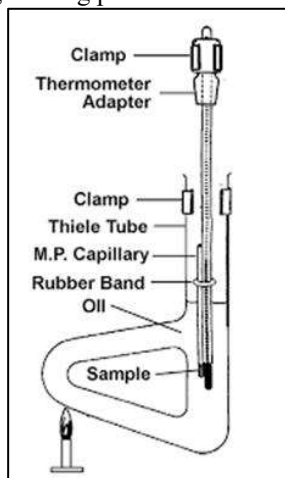
Conclusion: Carboxylic acids contain the carboxyl functional group (-COOH), which is present in both benzoic acid and phthalic acid. So, the conclusion is that both benzoic acid and phthalic acid are carboxylic acids.

6. DETERMINATION OF PHYSICAL CONSTANT AND IDENTIFICATION COMPOUND

Aim: To ascertain the melting point of the provided solid substance.

Material Required: Given Solid Substance, Capillary Tube, Paraffin, Laboratory thermometer.

Theory: The change from solid to liquid state of a compound in heating is called melting and the temperature at which a solid in its pure form melts is called the melting point. Each solid possesses a distinct melting point, making the determination of melting point crucial for compound identification. The presence of impurities reduces the melting point of a solid, making melting point a crucial criterion for assessing the purity of a compound.



Procedure

- Thiele's tube is the preferred apparatus for determining melting points. It comprises a hard glass test tube fused with V-shaped glass tubes at both the middle and bottom ends.
- The tube can be sealed with a one-holed cork housing a sensitive mercury thermometer, featuring a side slit. Approximately three-fourths of Thiele's tube is filled with a high-boiling liquid such as concentrated sulfuric acid or liquid paraffin.
- To determine the melting point of a substance, it's first powdered thoroughly. Then, a capillary tube, about 5 cm in length with one end fused, is selected.
- The powdered substance is introduced into the capillary tube to form a column of roughly 0.5 cm.
- The thermometer bulb is moistened with either liquid paraffin or concentrated sulfuric acid. Subsequently, the capillary tube is positioned to adhere to the thermometer in such a way that the bulb and the specimen are in close proximity.
- The apparatus is then sealed with the cork, ensuring the thermometer is submerged in the liquid while the capillary tube remains above the liquid level. The bulb of the thermometer should be positioned at the upper side tube level, with the open end of the capillary tube above the liquid level.
- The apparatus is securely fastened to an iron stand and gently heated by directing a flame at the side tube. As the liquid molecules are heated, uniform heating occurs.



- 16) To prevent high pressure buildup, vapor escapes through the slit in the cork. During heating, the mercury thread gradually rises until, at a particular moment, the solid substance in the capillary tube suddenly contracts and melts. The temperature is then noted as the melting point of the substance.

Observations

Melting point

1. 122°C
2. 123°C
3. 121°C

Mean Melting point = $(t_1+t_2+t_3)/\text{no. of observation}$

$$\text{Melting point} = \frac{122^\circ\text{C}+123^\circ\text{C}+121^\circ\text{C}}{3}$$

Melting point=122°C

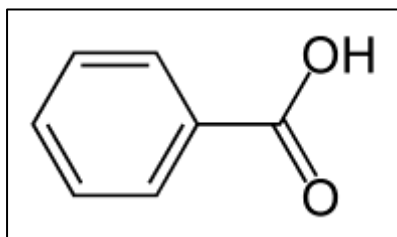
Precautions: Fill the capillary tube to one-third of its length. Control the rate of heating carefully.**Conclusion:** the observed melting point of 122°C falls within the typical range of 122-123°C for benzoic acid, confirming the identity of the compound as benzoic acid.**6.1 Confirmatory Test Compound (B):**

Test	Observation	Inference
Sodium Bicarbonate + Naphthalene +Benzoic Acid + KMNO ₄ + Conc.H ₂ SO ₄ + Resorcinol + NAOH	Colourless	Benzoic Acid present.

Conclusion: In the presence of benzoic acid, when treated with sodium bicarbonate (NaHCO₃), effervescence (bubbling) would occur due to the liberation of carbon dioxide gas. This is because benzoic acid reacts with sodium bicarbonate to form sodium benzoate, water, and carbon dioxide gas. So, the conclusion would be that the presence of benzoic acid is confirmed due to the observed effervescence when treated with sodium bicarbonate.

Results

Based on the step-by-step analysis above, it is evident that the organic compound is

Structure

- **Iupac Name:** Benzenecarboxylic acid
- **Formula:** C₇H₆O₂
- **Other Name:** benzenecarboxylic acid Carboxybenzene phenylformic acid
- **Molecular weight:** 122 g mol⁻¹
- **Category:** aromatic carboxylic acid.
- **State:** Solid.
- **Colour:** White crystalline solid.
- **Odour:** faint, pleasant odour.
- **Aromatic / Aliphatic:** Aromatic
- **Saturated / unsaturated:** unsaturated
- **Solubility:** soluble in water
- **Extra elements:** carbon atom, hydrogen
- **Functional group:** two conjoined benzene
- **Physical constant:** Melting point: 122°C



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