

Ch.Id:-ASU/GRF/EB/AEHFPOC/2022/Ch-10 DOI: https://doi.org/10.52458/9789391842697.2022.eb.grf.asu.ch-10

AIM: TO CARRY OUT THE QUALITATIVE TEST OF AROMATIC HYDROCARBON (BENZENE, TOLUENE AND NAPHTHALENE)

¹Dr. KAPIL KUMAR

¹Associate Professor, School of Pharmaceutical Sciences, Apeejay Stya University, Gurugram, Haryana, India

²Mr. MANOJ KUMAR SHARMA ²Assistant Professor, School of Pharmaceutical Sciences, Apeejay Stya University, Gurugram, Haryana, India

Requirements

Chemicals

- 1. Conc. H₂SO₄
- 2. Conc. HNO₃
- 3. CH₃OH
- 4. C₂H₅OH
- 5. CHCl₃
- 6. Formaldehyde
- 7. AlCl₃

Glass wares

- 1. Test tubes
- 2. Beaker
- 3. Bunsen burner
- 4. Stirring rod
- 5. Thermometer
- 6. Porcelain dish

Theory: Aromatic Hydrocarbons are organic molecules having a circular shape that contain sigma bonds and delocalized pi electrons. These chemical substances are also referred to as arenes and aryl hydrocarbons. Aromatic hydrocarbons are "unsaturated hydrocarbons with hydrogen atoms connected to one or more planar six-carbon rings termed benzene rings." Numerous aromatic hydrocarbons include benzene rings (also referred to as an aromatic ring). Resonance stabilises the benzene ring and delocalizes the pi electrons inside the ring structure.

Preliminary test

| Nature(solid/liquid/gas) |
|---------------------------|
| Colour(colour/colourless) |
| Odour(odour/odourless) |
| Flame test(Sooty flame) |

Solubility test: Solubility sin water, Conc. H₂SO₄, dil. HCl

| S. No. | Test Procedure | Observation | Inference |
|--------|--|---------------------------------|--|
| 1. | Sulphuric acid test: Take the given sample in a properly cleans and dry test tube. Add conc. H ₂ SO ₄ . Then, boil the solution until it is clear. Then, chill and add H ₂ O to a beaker. | Clear solution | Aromatic compoun d may be present |
| 2. | Nitration: Add 0.4 mL of concentrated H ₂ SO ₄ to 100 mg of the aromatic compound while stirring. While cooling the reaction mixture in water, add 0.4 mL of concentrated HNO ₃ while stirring and shaking. The reaction mixture is then heated and shaken in a H ₂ O bath at about 50 °C for 15 minutes, poured into 2 ml of cold H ₂ O, and collected by filtration. From methanol, recrystallize to a constant melting point. Use fuming nitric acid instead of concentrated intrating unreactive | Greenish- yellow crystals | Aromatic compoun d is confirmed |

| | substances. A pale yellow, insoluble oil with an almond-like odour. It freezes to produce yellow-green crystals. | | |
|----|---|---|--|
| 3. | Formalin test (Le Rosen test for aromatic compounds): Take 0.6 g of material in CHCl ₃ and 4-5 drops of formaldehyde-H ₂ SO ₄ reagent and place them on a porcelain plate. [Reagent: 5-6 drops of 37% HCHO + 5 ml of Conc. H ₂ SO ₄ . It should be prepared freshly. | Red or Green colours | Aromatic compoun d is confirmed |
| 4. | Chloroform-aluminium Test: Heat 0.2 g of AlCl ₃ in a test tube until it sublimes and adheres to the test tube's walls. Run 2 ml of Substance in CHCl ₃ down the test tube's sides. Note the colour created by interaction with AlCl ₃ solution. Due to the synthesis of triphenylmethane dyes, a red, orange, blue, or green hue may be observed. | Red, orange, blue or green colour | Aromatic compoun d is confirmed |
| 5. | Permanganate method: In a flask with a round bottom, combine 1g of the unknown substance, 4g of potassium permanganate, 1g of sodium carbonate, and 100ml of water. Flux the mixture until the permanganate's colour is expelled. | Benzoic acid | Aromatic compoun d is confirmed |

| Acidify with diluted HCl and then | | |
|---|---|---|
| add a 25 percent sodium sulphite | | |
| solution while vigorously shaking | | |
| until the brown manganese dioxide | | |
| precipitate has disintegrated. On | | |
| cooling, a solid product separates | | |
| from water or aqueous ethanol, | | |
| which is then recrystallized. | | |
| $ \begin{array}{c c} & & \\ & & \\ \hline \\ & \\ \hline \\ & \\ \hline \\ & \\ \\ & \\ \\ & \\ \\ \hline \\ & \\ \\ \hline \\ & \\ \\ \hline \\ \\ & \\ \\ \hline \\ \\ \\ \\$ | | |
| | 1 | 1 |

Functional Group Test for Benzene or Toluene

| S. No. | Test Procedure | Observation | Inference |
|--------|--|------------------------|-------------------------------------|
| 1. | Nitration: To 5 ml of an equal volume mixture of conc. HNO ₃ and conc. H ₂ SO ₄ , add progressively 1 ml of benzene or toluene while shaking. Cool if the reaction begins to become too vigorous. After vigorously shaking the mixture for approximately 2 minutes, pour it into cold water. Yellow oil or solid will separate from the nitro-compound. Product is separate and washes with water, and then reduces it | Yellow oil or solid | Benzene/tol uene is confirmed |

| | with tin and concentrated HCl to demonstrate the existence of the nitro- group. | | |
|----|--|----------------|-------------------------------------|
| 2. | Sulphonation: Take 1 ml benzene or toluene and carefully add 1 ml of fuming 20 percent H ₂ SO ₄ . Shake thoroughly and see the resulting homogenous solution. Now, gently add the reaction mixture into roughly 20 ml of cold H ₂ O while stirring; a clear solution of sulphonic acid is produced. However, a little quantity of the equivalent sulphone, R ₂ SO ₂ , typically separates as fine, colourless crystals. | Clear solution | Benzene/tol uene is confirmed |

Functional Group Test for Naphthalene

| S. No. | Test Procedure | Observation | Inference |
|--------|---|-----------------------------|-----------------------------|
| 1. | Nitration:Atransparentyellowsolution is prepared bydissolving 1 g in 5 mlof glacial acetic acidusing mild heating,cooling, adding 1 ml of | Yellow 1-nitro- compound | Naphthalene is confirmed |

| | cone HNO ₃ , and heating to about 80 °C for 1 minute. Inject into water. The yellow 1- nitro compound solidifies at 61 degrees Celsius. A transparent yellow solution is produced. The 1-nitro compound becomes solid. | | |
|----|--|--------------------------------|-----------------------------|
| 2. | Picrates: After you have prepared a conc. picric acid solution in cold acetone, mix approximately 2 millilitres of this solution with approximately 2 millilitres of a cold conc. Naphthalene solution in acetone. Shake well. After approximately one minute of standing, yellow needles of naphthalene picrate crystallise out. The melting point of picrate after recrystallization from ethanol is 152 °C. Long, yellow crystals | Long yellow needles crystal | Naphthalene is confirmed |

| of naphthalene picrate | |
|------------------------|--|
| emerge. | |

Result: The results of the systemic qualitative tests performed and benzene/ toluene/ naphthalene were found and reported.