

## QUALITATIVE ORGANIC ANALYSIS

### 1. PHYSICAL CHARACTERISTICS:

- a) Physical State: Crystalline / amorphous.
- b) Colour:
- c) Odour:

### 2. DETECTION OF SPECIAL ELEMENTS:

Lassaigne's Test:

A pea-sized sodium was taken in a clean and dry fusion tube. It was heated on a Bunsen flame till it melted and a shining surface appeared. A pinch of the supplied sample was added to it. Then it was heated first gently and then strongly to red hot. The hot tube was plunged immediately into 8-10 ml of distilled water taken in a mortar. It was pestled well and then filtered. With the filtrate the following tests were performed.

EXPERIMENT	OBSERVATION	INFERENCE
<b>i) Test for Nitrogen:</b> To a portion of the filtrate (ca. 2 ml) few drops of freshly prepared $\text{FeSO}_4$ solution was added. This was heated gently till it boiled and then cooled. The solution was then acidified with d. $\text{H}_2\text{SO}_4$ .	Prussian blue coloration (or precipitate) was observed	Nitrogen (N) present and confirmed.
<b>ii) Test for Sulfur:</b> To a part of the filtrate few drops of sodium nitroprusside solution was added.	Violet / purple coloration was observed.	Sulfur (S) present and confirmed.
<b>iii) Test for Chlorine:</b> To a part of the filtrate equal volume of c. $\text{HNO}_3$ was added. It was boiled till the volume was reduced to 1/2 of its original, the solution was cooled. To this $\text{AgNO}_3$ solution was added. <b>NOTE: If Nitrogen is found absent in the sample it is unnecessary to reduce the volume by boiling. Directly add <math>\text{AgNO}_3</math> to the acidified filtrate (with d. <math>\text{HNO}_3</math>).</b>	Curdy white ppt. was observed which is soluble in d. $\text{NH}_4\text{OH}$ , reappeared on acidification with d. $\text{HNO}_3$ .	Chlorine (Cl) present and confirmed.

### 3. SOLUBILITY CLASSIFICATION:

	Water	5% NaOH	5% $\text{NaHCO}_3$	5% HCl	Conclusion
i)	+	NP	NP	NP	Polar compound
ii)	—	+	—	—	Weakly acidic compound
iii)	—	+	+	—	Strongly acidic compound
iv)	—	—	NP	+	Basic Compound
v)	—	—	NP	—	Neutral, non polar compound
vi)	—	+	+	+	Amphoteric compound
vii)	—	+	—	+	Strongly basic and weakly acidic compound.

(+ means soluble, — means insoluble, NP means Not Performed.)

4. DETECTION OF FUNCTIONAL GROUPS:

A] DETECTION OF NITROGENOUS FUNCTIONAL GROUPS:

EXPERIMENT	OBSERVATION	INFERENCE
<p>i) <b>Test for aromatic primary amine (Ar-NH<sub>2</sub>):</b> A small amount of the sample was dissolved (/suspended) in d. HCl. It was cooled under tap water. To it aq. NaNO<sub>2</sub> solution was added drop wise with stirring. The resulting solution was poured into cold alkaline β-naphthol solution.</p>	Red / Orange dye was observed.	-NH <sub>2</sub> (aromatic) present and confirmed.
<p>iii) <b>Test for nitro:</b> a) Aromatic -NO<sub>2</sub>: A small amount of the sample was taken in a test tube with Zn-dust (/ Sn granules) and c. HCl. This was heated gently for 5 minutes the resulting solution was cooled, filtered and diluted. With the cold filtrate diazo test was performed as usual. <b>NOTE: if aromatic NH<sub>2</sub> is present in the sample this test for nitro group can not be performed. Go directly to Mulliken and Barker's test.</b> b) Mulliken – Barker's test (Aromatic / aliphatic -NO<sub>2</sub>): A small amount of the sample was dissolved in 50% aq. alcohol. To it was added Zn-dust and NH<sub>4</sub>Cl. This mixture was boiled for few minutes, cooled and allowed to stand for 5 minutes. This was then filtered into Tollens' reagent.</p>	Red / Orange dye was observed.  Black / grey ppt. / shining mirror on the side of the test tube was observed.	Aromatic -NO <sub>2</sub> present.  -NO <sub>2</sub> present and confirmed.
<p><b>Preparation of Tollens' reagent: to 2-3 ml of AgNO<sub>3</sub> solution taken in a clean test tube drop wise d. NaOH solution was added till a grey ppt. appeared. To this solution d. NH<sub>4</sub>OH was added drop wise till the ppt. just dissolved.</b></p>		
<p>iv) <b>Test for amide (-CONH<sub>2</sub>):</b> A small amount of the sample was heated in a test tube with few drops of water and 2-3 beads of NaOH.</p>	Pungent smell of ammonia and the issuing gas turned phenolphthalein soaked filter paper pink.	-CONH <sub>2</sub> present and confirmed.

**If N is absent:** As N was found absent in the sample, tests for nitrogenous functional groups (aromatic primary amine, nitro and amide) were not performed and these were absent.

B] DETECTION OF NON-NITROGENOUS FUNCTIONAL GROUPS:

EXPERIMENT	OBSERVATION	INFERENCE
<p>i) <b>Test for carboxylic acid (-CO<sub>2</sub>H):</b> a) A solution (/ suspension) of the sample in water / aq. alcohol was tested with blue litmus paper. b) To an aq. (/ aq. alcoholic) solution of the sample a pinch of solid NaHCO<sub>3</sub> was added. c) <b>(To be done only if the sample affords effervescence with sat. NaHCO<sub>3</sub> solution)</b> A small amount of the sample was taken in a dry test tube with 2 parts of absolute alcohol and 1 part of c. H<sub>2</sub>SO<sub>4</sub>. This was warmed in a water bath for few minutes. The resulting solution was cooled and poured into aq. Na<sub>2</sub>CO<sub>3</sub> solution taken in an evaporating dish, smelt immediately.</p>	Blue litmus turned red.  Effervescence was noted.  Sweet, fruity smell of ester was noted.	Carboxylic acid (-CO <sub>2</sub> H) may be present. -CO <sub>2</sub> H present.  -CO <sub>2</sub> H present and confirmed.
<p>ii) <b>Test for phenolic -OH:</b> a) A drop of neutral ferric chloride solution was added to aq. (/ aq. alcoholic) solution of the sample. <b>NOTE: always perform a blank test.</b> b) Back-dye test <b>(to be done only if the sample affords characteristic color with FeCl<sub>3</sub>):</b> A drop of aniline was dissolved in d. HCl. It was cooled under tap water, few drops of aq. NaNO<sub>2</sub> solution was added to it. This was poured into cold solution of sample in d. NaOH.</p>	Violet / green / blue / wine-red colouration was noted, which was discharged on acidification with d. HCl.  Red / Orange dye observed.	Phenolic -OH present.  Phenolic -OH present and confirmed.

<p>iii) <b>Test for carbonyl:</b></p> <p>a) <b>For Ketone / aldehyde:</b> a small amount of the sample was dissolved in minimum volume of absolute alcohol taken in a dry test tube. To it was added equal volume of Brady's reagent (2,4-dinitro phenylhydrazine (2,4-DNP)) solution. This was shaken vigorously; the inner walls of the test tube were scratched with glass rod. Finally this was kept in a hot water bath for ~ 10 minutes.</p> <p>b) <b>For aldehyde</b> (<i>to be done only if the sample affords ppt. with 2,4-DNP</i>): A small amount of the sample was added to 2 ml of Tollens' reagent taken in a clean test tube, warmed in water bath.</p>	<p>Red / orange / yellow crystalline ppt. appeared.</p> <p>Black / grey ppt. / shining mirror on the side of the test tube was observed.</p>	<p>Carbonyl (ketone or aldehyde) present.</p> <p>-CHO present and confirmed.</p>
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