



STUDY MATERIAL

VIVEKANANDA COLLEGE

THAKURPUKUR

NAAC ACCREDITED GRADE—'A'

Subject: Chemistry

Topic

Systematic Qualitative Analysis of an
Organic Compound (Practical)

Name of the Teacher

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SYSTEMATIC QUALITATIVE ANALYSIS OF AN ORGANIC COMPOUND

SAMPLE NO.:

DATE:

1. PHYSICAL CHARACTERISTICS:

- (a) COLOUR:
- (b) ODOUR:
- (c) TEXTURE: (Crystalline or Amorphous)

2. DETECTION OF SPECIAL ELEMENTS (N, Cl, Br, I and S):

Lassaigne's Test: In a dry fusion tube a piece of metallic sodium, free from kerosene was taken. The fusion tube was then heated just to melt the sodium (Indication: dark gray sodium vapors come out of a shining surface appears). Some amount of organic sample (0.5 gm) was added portion wise directly onto the molten sodium (caution: there may be slight explosion). The tube was then further heated, first gently then strongly to red-hot. The red-hot tube was then plunged into distilled water (10-15 ml) taken in a mortar. The fusion tube cracked. The broken pieces of glass were ground with a pestle. After proper mixing, the solution was filtered. With the filtrate, the following tests were performed.

NOTE: If the sample is a liquid make a stiff paste of it with anhydrous Na_2CO_3 and add it to the fused sodium.

Sometimes explosion occurs or the sample catches fire during fusion with sodium, especially with nitro-compound. In such cases, the sample should be mixed with Na_2CO_3 before the sodium fusion.

Experiment	Observation	Inferences
1. To a little amount of fusion solution, few FeSO_4 granules were added, boiled for few minutes, cooled and acidified with dilute H_2SO_4 .	Prussian blue precipitate or Coloration.	Nitrogen present.
2. To a little amount of fusion solution, sodium nitroprusside solution was added	Purple coloration	Sulphur present
4. A little of fusion solution was acidified with dil HNO_3 , boiled to reduce the volume to 1/3 rd. The solution was cooled and AgNO_3 Solution added.	White ppt which was easily soluble in NH_4OH but Insoluble in dilute HNO_3 .	Chlorine present

3. SOLUBILITY TEST AND SOLUBILITY CLASSIFICATION:

100 mg of sample was added to 3 ml of solvent taken in a test tube. This was shaken for 2-3 minutes till clear solution was obtained.

Water	dil HCl	dil NaOH

4.SYSTEMATIC ANALYSIS OF FUNCTIONAL GROUPS:

A.Nitrogenous functional groups:

Experiment	Observation	Inference
<p><u>1.DYE TEST</u> To a solution of sample in 5% HCl, aqueous NaNO₂ solution was added at 0°-5°C. The resulting solution was added to alkaline β-Naphthol solution at 0°-5°C.</p>	<p>A red or orange precipitate (Dye) was obtained</p>	<p>Primary aromatic amine present</p>
<p><u>2.MULLIKEN BARKER TEST:</u> To a little amount of sample solution in 50% ethanol, Zn dust and NH₄Cl was added and boiled for few minutes. Cool and filter. (i) To one part of the filtrate add Tollen's reagent and warm on water bath. ----- (ii) To another part of the filtrate add Fehling's reagent and warm on water bath.</p> <p>NOTE: This test should not be performed if the original compound reduces Tollens' reagent.</p> <p>3. A small amount of the organic sample was boiled with tin and conc. HCl on a water bath for at least 5-10 minutes. Cool and add about 5 ml of distilled water and filter. Cool the filtrate to 0°C and then add a few drops of ice cold NaNO₂ solution. Pour this diazotized solution into a test tube containing an ice cold alkaline solution of β-naphthol.</p> <p>NOTE: This test should not be performed if the compound already contains an aromatic primary amino group.</p>	<p>A grey to black ppt. along with a silver mirror obtained</p> <p>A red ppt. was obtained</p> <p>A red or orange-red dye was obtained</p>	<p>Nitro group (-NO₂) present</p> <p>Nitro group (-NO₂) present</p>

B. Non nitrogenous functional groups:

Experiment	Observation	Inference
1. A little amount of the sample was dissolved in ethanol and neutral FeCl₃ solution was added to it dropwise.	Violet, blue, green or red coloration	Phenolic-OH or enolic-OH group present
2. A little amount of aniline was dissolved in dil.HCl, cooled and diazotised by adding NaNO₂ solution and cooled to 0-5⁰C.This cold solution was then added to the cold alkaline solution of the sample at 0⁰-5⁰C.	Red or orange dye was obtained	Phenolic-OH group present
3. Add 2-3 mL of the ethanolic solution of the sample to a saturated aqueous solution of NaHCO₃ in a test tube.	Effervescence took place	-COOH group present
4. Warm a small amount of the sample with two parts of absolute ethanol and one part of concentrated H₂SO₄ for two minutes. Cool it and pour cautiously into aqueous sodium carbonate solution contained in an evaporating dish, and smell immediately.	A sweet, fruity smell along with the separation of an oily layer was observed	-COOH group present
5. To a little amount of the sample solution in ethanol equal volume of Brady's reagent was added and the solution was warmed on water bath with the addition of 1 or 2 drops of Conc. H₂SO₄,if necessary.	An orange or red colored ppt. was obtained	Carbonyl group present.
6.Treat 5 ml of Tollen's reagent in a clean t.t with a small quantity of the sample or alcoholic solution of the sample. Warm the test tube on a water bath.	A grey to black ppt. along with a silver mirror obtained	Aldehyde(-CHO) group present

NOTE:

Tollens' reagent

Laboratory Preparation

1. To aqueous silver nitrate, a drop of dilute sodium hydroxide is added, forming a brown precipitate of silver oxide. (if ppt did not appear, add more)
2. Concentrated ammonia is then added until the precipitate just dissolves.
3. This is ammoniacal silver nitrate, $[\text{Ag}(\text{NH}_3)_2]\text{NO}_3$ (aq).The reagent should then be filtered.

The reaction is:



Preparation of neutral FeCl_3 solution:

To a little amount of FeCl_3 solution in a test tube, add NH_4OH solution (very dilute) drop by drop & shake after each drop until the ppt. of $\text{Fe}(\text{OH})_3$ just persists. Filter the solution & the filtrate is called neutral FeCl_3 solution.