GOGATE JOGALEKAR COLLEGE (AUTONOMOUS), RATNAGIRI

(Affiliated to University of Mumbai)

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Department of Chemistry

F.Y.B.Sc. CHEMISTRY PRACTICAL HANDBOOK

Semester I and II

For Internal Circulation Only

"Learning by doing – Strengthening concepts through hands-on experience"

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Note:
Students are expected to carry a separate notebook for writing observations, calculations, and results. This handbook provides standardized Aims, Theory, Procedure, and Lab Questions for all practicals.

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🔬 Experiment 1: Introduction to Chemistry Laboratory – Safety Rules and Precautions

Aim:

To understand the layout of the chemistry laboratory, recognize common laboratory apparatus, and learn essential safety rules and precautions for safe working in the lab.

Theory:

A chemistry laboratory is a place where chemical experiments and reactions are conducted. Working in a lab involves handling chemicals, glassware, and instruments that can be hazardous if not managed properly. Therefore, knowledge of safety protocols is essential to prevent accidents and ensure a safe working environment.

Key Concepts:

- Proper lab behaviour
- Use of personal protective equipment (PPE)
- Emergency protocols
- Identification of hazard symbols

Procedure:

1. Lab Tour:
    - Familiarize yourself with the lab layout: working benches, sinks, fume hood, chemical storage, and emergency exit.
2. Identify Common Apparatus:
    - Observe and identify beakers, burettes, pipettes, conical flasks, test tubes, etc.
3. Discuss PPE:
    - Learn about proper use of lab coat, gloves, goggles, and closed-toe shoes.
4. Review Safety Rules:
    - Instructor explains safety signs, labeling of chemicals, and rules of conduct.
5. Emergency Equipment:
    - Locate fire extinguisher, eye-wash station, and first aid box.

Observations:

Item Observed	Purpose / Safety Role
Fire Extinguisher	For controlling minor lab fires
Eye Wash Station	To flush eyes in case of chemical splash
Fume Hood	To safely handle volatile/toxic chemicals
PPE (Lab coat, etc.)	To protect from chemical splashes and burns

CHEMISTRY LAB SAFETY RULES

☑ BEFORE ENTERING THE LAB:

- 👕 Wear a Lab Coat – Protects your clothes and skin
- 👓 Use Safety Goggles – Always wear them when working with chemicals
- 👞 Closed-Toe Shoes Only – No sandals or slippers
- 💇 Tie Back Long Hair – Prevent it from catching fire or dipping into chemicals

🚫 GENERAL LAB CONDUCT:

- ❌ No Eating or Drinking in the lab
- ❌ Do Not Taste or Smell Chemicals Directly
- 📢 Work Silently & Stay Focused
- 📦 Keep Bags Outside the working area
- 🧼 Wash Hands After Experiments

🧯 HANDLING CHEMICALS & EQUIPMENT:

- ➕ Label All Chemicals Clearly
- 💦 Always Add Acid to Water (Not Water to Acid!)
- 🧪 Handle Glassware Carefully – Report broken items immediately
- 🔬 Do Not Touch Electrical Equipment with Wet Hands

⚠ IN CASE OF AN EMERGENCY:

- 🔥 Fire Extinguisher: Know where it is and how to use it
- 👁 Eye Wash Station: Flush for 15 minutes if chemicals enter the eye
- 🩹 First Aid Box: Report any injuries to your teacher immediately
- 🚪 Know Emergency Exits

📣 REMEMBER: SAFETY FIRST, EXPERIMENT SECOND!

🧠 A safe lab is a productive lab. Follow instructions. Ask when unsure.

Result:

Familiarized with the laboratory environment and understood safety practices and precautions to be followed during experiments.

Pre-lab Questions:

1. Why is it necessary to wear a lab coat and goggles in the lab?
2. What should you do if acid spills on your hand?
3. What is the correct method to dilute a concentrated acid?
4. Why is eating or drinking prohibited in the chemistry lab?

Post-lab Questions:

1. What should be done in case of a chemical fire?
2. Why should long hair be tied back in the lab?
3. Explain the importance of labelling reagent bottles.
4. Mention any two hazard symbols and their meanings.

🔬 Experiment 2: Introduction to Laboratory Apparatus and Their Handling

Aim:

To identify common laboratory apparatus and learn their correct handling techniques.

Theory:

A chemistry laboratory uses a wide variety of glassware, plasticware, and equipment. Each item has a specific purpose—such as measuring, mixing, heating, or storing chemicals. Knowing how to correctly identify and use these tools is essential for safe and effective experimentation.

Categories of Lab Apparatus:

1. Measuring Devices:
    - Measuring Cylinder, Burette, Pipette, Volumetric Flask
2. Containers and Glassware:
    - Beaker, Conical Flask (Erlenmeyer), Test Tube, Watch Glass
3. Heating Equipment:
    - Bunsen Burner, Tripod Stand, Wire Gauze, Crucible
4. Supporting Apparatus:
    - Clamp Stand, Test Tube Holder, Tongs, Funnel, Dropper
5. Cleaning & Safety Tools:
    - Brush, Wash Bottle, Gloves, Goggles

Procedure:

1. **Visual Identification:**
    - Observe the items placed on the demonstration table or lab bench.
    - Note the shape, markings, and material of each apparatus.
2. **Learn the Uses:**
    - Understand how and when to use each apparatus.
3. **Safe Handling Practice:**
    - Hold glassware from the bottom.
    - Use tongs/holders while heating.
    - Rinse glassware before and after use.
    - Never heat graduated glassware directly.

4. **Demonstration:**
    - Filling a pipette using a pipette bulb.
    - Reading a burette at eye level.
    - Assembling a titration setup using a clamp stand.

Observations:

Apparatus	**Use / Function**	**Handling Tip**
Burette	Delivers known volume of liquid in titration	Clamp vertically, read from bottom meniscus
Pipette	Measures a fixed volume of solution	Use pipette filler, never suck by mouth
Beaker	Holds liquids, for mixing/heating	Use tongs when hot
Measuring Cylinder	Measures approximate volumes	Read at eye level
Test Tube	Holds small quantities of substances	Use holder for heating

Result:

Successfully identified common laboratory apparatus and learned correct methods of handling them.

Pre-lab Questions:

1. What is the use of a burette in titration?
2. Why should we not heat a volumetric flask?
3. Name two apparatus used for accurate measurement of liquids.
4. What precautions should be taken while handling glassware?

Post-lab Questions:

1. What is the difference between a beaker and a conical flask?
2. Why should pipettes not be filled by mouth?
3. How will you clean a burette after use?
4. Why should you read the lower meniscus in a burette?

🔬 Experiment 3: Preparation of Molar and Normal Solutions

Aim:

To prepare molar and normal solutions of any four of the following:

NaOH, KOH, Succinic acid, Oxalic acid, $K_2Cr_2O_7$, $KMnO_4$, Iodine, $Na_2S_2O_3$.

Theory:

Standard Solution:
A standard solution is a solution whose concentration is precisely known. It is prepared by dissolving an accurately weighed amount of a substance in a definite volume of solvent.

- **Primary Standard:**
  A primary standard substance is highly pure, stable, non-hygroscopic, and has a high molar mass. It can be weighed directly to prepare a standard solution.
  Examples: Na_2CO_3, $K_2Cr_2O_7$, Oxalic acid, Succinic acid.
- **Secondary Standard:**
  A secondary standard solution is standardized against a primary standard. It is less stable and is often affected by light, temperature, or air.
  Examples: NaOH, $KMnO_4$, Iodine, $Na_2S_2O_3$.

Molarity (M): Number of moles of solute per litre of solution.

Normality (N): Number of gram equivalents of solute per litre of solution.

Procedure (for each selected compound):

1. Calculate amount of the compound to be weighted to prepare 0.1 M / 0.1 N 100 ml solution.
2. Accurately weigh the required amount of the compound using a digital balance.
3. Transfer it into a 100 ml beaker. Add some distilled water water (approximately 20-30 mL).
4. Stir using clean glass rod so that crystals of succinic acid dissolve completely.
5. Transfer the solution to 100 ml volumetric flask using a funnel.
6. Wash the beaker two three times with small amount of distilled water and collect the washings in volumetric flask.
7. Rinse the beaker and stirring rod several times with small portions of distilled water, pouring each rinsing into the volumetric flask via the funnel.
8. Make up the volume up to the 100 ml mark with distilled water.
9. Label the flask with name of solute, concentration (M or N), and date.

Observations:

(Example for Succinic acid)

Substance	Molar Mass (M.W)	**Weight Taken (g)**	**Volume Made (ml)**	**Molarity / Normality**
Succinic acid	118	1.18 gm	100	0.1 M
Oxalic Acid				
NaOH				
$K_2Cr_2O_7$				
$Na_2S_2O_3$				
$KMnO_4$				

Substance	Molar Mass (M.W)	**Equivalent weight (E.W)**	**Weight Taken (g)**	**Volume Made (ml)**	**Molarity / Normality**
Succinic acid	118	59	0.59 gm	100	0.1 N
Oxalic Acid					
NaOH					
$K_2Cr_2O_7$					
$Na_2S_2O_3$					
$KMnO_4$					

Calculations:

(Calculation explained with the example of succinic acid)

(Given atomic weight of C = 12, O = 16, H = 1)

For Succinic acid

- Molecular formula of Succinic acid = $C_4H_6O_4$
- Molecular weight (M. W.) of Succinic acid = 118

12 x 4 = 48

1 x 6 = 06

16 x 4 = 64

Total = 118

- Equivalent weight of succinic acid = M.W / No of replaceable H^+ ions (n)

= 118 / 2

= 59

Calculation for amount of compound to be weighed for preparing molar solution

$$\text{Amount to be weighed} = \frac{\text{M.W. (in gm) X M (required molarity of solution) X Volume (in ml)}}{1000}$$

Calculation for amount of compound to be weighed for preparing normal solution

$$\text{Amount to be weighed} = \frac{\text{E.W. (in gm) X N (required normality of solution) X Volume (in ml)}}{1000}$$

Result:

1. The amount of ------- to be weighed to prepare 0.1 N 100 ml ------ is = ----------
2. Prepared 0.1 N 100 ml ------- solution. Learnt the skill of preparing standard solution.

Pre-lab Questions:

1. What is the difference between molarity and normality?
2. What are the characteristics of a primary standard substance?
3. Why is NaOH considered a secondary standard?
4. Write the formula to calculate the weight required for a 0.1 M NaOH solution.

Post-lab Questions:

1. Why is $KMnO_4$ solution not used as a primary standard?
2. What precautions should be taken while preparing standard solutions?
3. If more water is added accidentally, how will it affect the molarity?
4. Why is oxalic acid suitable as a primary standard?
5. Calculate molecular weight and equivalent weight of oxalic acid, NaOH, $K_2Cr_2O_7$, $Na_2S_2O_3$, $KMnO_4$
6. Calculate amount to be weighted of oxalic acid, NaOH, $K_2Cr_2O_7$, $Na_2S_2O_3$, $KMnO_4$ if you want to prepare 0.1 M solution of each.
7. Calculate amount to be weighted of oxalic acid, NaOH, $K_2Cr_2O_7$, $Na_2S_2O_3$, $KMnO_4$ if you want to prepare 0.1 N solution of each.

🔬 Experiment 4: Preparation and Standardization of Solutions

Aim:

To prepare 0.1 N Succinic acid solution (primary standard) and use it to standardize two different concentrations of NaOH solution (secondary standard).

Requirements: Analytical balance, 100 cm^3 beaker, glass rod, 100 cm^3 standard measuring flask, 50 cm^3 burette, 10 cm^3 pipette, conical flask, etc.

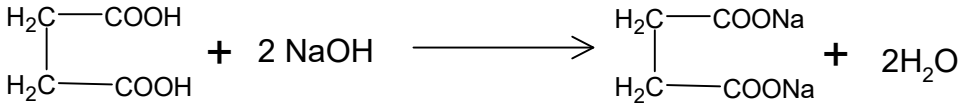
Succinic acid (solid), NaOH solution, distilled water and 1% Phenolphthalein indicator.

Theory:

Standard Solution:
A solution of accurately known concentration used in titrations.

- **Primary Standard:**
  Substances that are pure, stable, and non-hygroscopic. They can be directly weighed to prepare standard solutions.
  Example: Succinic acid ($HOOC-(CH_2)_2-COOH$), which is diprotic (n = 2).
- **Secondary Standard:**
  Solutions that are unstable or absorb moisture from air, so they cannot be weighed directly. Their concentration is determined by titrating against a primary standard.
  Example: Sodium hydroxide (NaOH).

For standardization of NaOH solution, succinic acid solution is prepared, and it is titrated against NaOH solution using phenolphthalein indicator. During titration NaOH reacted with succinic acid



At the end point of the titration, all the succinic acid has been reacted and any additional NaOH in the solution will turn the solution basic and the phenolphthalein indicator turn the solution from colourless to just pink.

Titration Principle:
The volume of NaOH required to completely neutralize a known volume of succinic acid solution can be used to determine its exact concentration using:

$N_1V_1=N_2V_2$

Procedure:

1. Prepare 100 cm^3 of 0.1N succinic acid solution (0.59g of succinic acid dissolved in 100 cm^3 of distilled water).

2. Pipette out 10 ml of prepared Succinic acid solution in a conical flask.

3. Add 2–3 drops of phenolphthalein indicator.

4. Fill the burette with the NaOH solution of unknown concentration.

5. Titrate the acid with NaOH until a light pink endpoint appears.

6. Repeat the same for the second NaOH solution.

7. Record the burette readings and calculate the normality of NaOH

Observations:

Solution in Burette – ----------

Solution by pipette – ----------

Indicator used – ---------

Colour change – --------

Observation Table

Burette level	**Burette reading in cm^3**			**Constant Burette Reading in cm^3 (CBR)**
	I	**II**	**III**	
Initial				**x cm^3**
Final				
Difference				

Calculations:

10 cm^3 of 0.1 N succinic acid solution required x cm^3 of NaOH solution.

Using the formula N1V1 = N2V2 calculate exact normality of NaOH solution.

NaOH Succinic acid

N1V1 = N2V2

N1 x CBR = 0.1 x 10

N1 = 0.1 x 10 / CBR

Follow the same procedure for the second sample of NaOH (C_2)

Result:

1. Normality of NaOH solution (C_1) : N
2. Normality of NaOH solution (C_2) : N

Pre-lab Questions:

1. What is a primary standard? Give examples.
2. Why is NaOH not used as a primary standard?
3. What is the equivalent weight of succinic acid?
4. What is the role of an indicator in titration?

Post-lab Questions:

1. Why do we need to standardize NaOH before use?
2. If you use a higher volume of succinic acid, how will it affect the titration?
3. What color change indicates the end point in this titration?
4. How can you prepare 1 N Succinic acid solution?

🔬 Experiment 5: Drawing Chemical Structures of Organic Molecules using ChemDraw / ChemSketch

Aim:

To draw the chemical structures of selected organic molecules using ChemDraw or ChemSketch software.

Theory:

ChemDraw and ChemSketch are computer-aided drawing tools used extensively in chemical education and research for:

- Drawing 2D and 3D chemical structures
- Generating IUPAC names
- Creating reaction mechanisms
- Calculating molecular formulas and weights
- Exporting structures into research papers or lab reports

These tools allow students to visualize the spatial arrangement and connectivity of atoms in organic compounds and help in understanding structure-property relationships.

Procedure:

1. **Launch the Software:** Open ChemDraw or ChemSketch on your computer.
2. **Select Tools:** Choose appropriate templates/tools for drawing bonds (single, double, triple), rings (benzene, cyclohexane), atoms, charges, and other symbols.
3. **Draw Molecules:** Using the tools, draw the following structures

   (choose any **5** molecules):

   - Methane (CH_4)
   - Ethanol (CH_3CH_2OH)
   - Acetone (CH_3COCH_3)
   - Benzene (C_6H_6)
   - Acetic Acid (CH_3COOH)
   - Glucose ($C_6H_{12}O_6$)
4. **Label the Structures:** Add names of molecules.
5. **Save and Export:** Save your file in native format (.cdx or .sk2).

   Export as image or PDF for report submission.

Observations:

(Sample table)

Molecule	**Molecular Formula**	**Structure Drawn**	**Software Used**
Ethanol	C_2H_6O		
Acetone	C_3H_6O		
Benzene	C_6H_6		
Acetic Acid	$C_2H_4O_2$		
Glucose	$C_6H_{12}O_6$		

Result:

Successfully drew the chemical structures of selected organic compounds using ChemDraw/ChemSketch software.

Pre-lab Questions:

1. What is the function of ChemDraw software?
2. What is the molecular formula of benzene?
3. Name one open-source alternative to ChemDraw.

Post-lab Questions:

1. What is the difference between skeletal and full structural formula?
2. How can you calculate molecular weight using ChemDraw?

🔬 Experiment 6: Determination of Enthalpy of Dissolution of Potassium Nitrate (KNO_3)

Aim:

To determine the enthalpy of dissolution of salt (KNO_3)

Requirements:

Polythene bottle, 0.1^0 thermometer, 25 cm^3 pipette, etc. KNO_3 solid and distilled water.

Theory:

When ionic compounds are dissolved in water, they either absorb energy (heat) from the surrounding or release energy to the surrounding. Thus, when any compound or substance is dissolved in water, heat either evolved or absorbed. The change in heat during the process is called as enthalpy of dissolution or enthalpy of solution.

Enthalpy of dissolution is defined as the heat change when one mole of the solute is dissolved in large volume of the solvent.

Enthalpy may be positive or negative. **For endothermic process**, when heat is absorbed by substance from surrounding the **enthalpy of dissolution is positive**.

For exothermic process, when heat is released by substance to the surrounding, the **enthalpy of dissolution is negative.**

When the salt, KNO_3 is dissolved in water, it absorbs the heat from the surrounding (water) and the temperature of the water is lowered. The process is endothermic, and the absorbed heat is utilized to break the intramolecular forces between KNO3 molecules.

The enthalpy of dissolution of KNO_3 salt is determined by measuring the change in temperature of water (i.e. before and after dissolution). The enthalpy of dissolution is represented as,

$$Q = (m)\ (\Delta t)\ (Cp)$$

where,

Q is enthalpy of dissolution; m is the mass of water,

Δt is change in temperature, Cp is specific heat of water (4.18 $j/(g^0c)$)

Procedure:

1. Obtain a Styrofoam cup “calorimeter” and add to it 40.0 mL of distilled water.
2. Secure a thermometer to stand up in the calorimeter, using your ring stand.
3. Weigh out 4.0 grams of potassium nitrate.
4. Record the initial temperature of the water in the calorimeter.

5. Add the potassium nitrate to the water in the calorimeter, all at once, and begin stirring solution to dissolve the salt as rapidly as possible. Stir by swirling the cup with your hand. DO NOT use a glass rod, as it will affect your results. Watch out for the fragile thermometer. Record the lowest temperature achieved during the dissolving of the salt.

6. Cleanup, Rinse the solution down the sink with LOTS of water. Rinse the thermometer and return it to your lab drawer.

Observation:

1. Mass of potassium nitrate used = _____g

2. Volume of water used. = _____mL

3. Initial temperature (T_1) = _____ ^0C

4. Final (lowest) temperature (T_2) = _____ ^0C

Calculation:

1. Δt = Change in temperature= (Initial temperature (T_1)) – (Final (lowest) temperature (T_2))

2. Calculate the amount of energy absorbed as the potassium nitrate dissolved. In order to do this, we must assume that the solution has the same specific heat (c_p) as pure water, and that the density of the water was 1.00 gram/mL. Express your final answer in kilojoules KJ.

$$g = c_p \times m \times \Delta T$$

where,

cp = specific heat of water = 4.18 J

m = mass of water = 40 mL

3. Calculate the number of moles of potassium nitrate used

$$\text{no. of moles} = \text{wt/ mol. wt}$$

$$\therefore \text{No. of moles} = 4/ \text{ mol. wt of potassium nitrate (101.10).}$$

4. Divide the heat absorbed (in KJ) by the moles of potassium nitrate dissolved. This is the heat of solution, expressed in KJ/mol of solute

$$\Delta H = c_p \times m \times \Delta T/(\text{No. of moles} \times 1000) = \ldots\ldots\ldots\ldots \text{ KJ/mol}$$

5. Using the known value of the heat of solution of potassium nitrate calculate the absolute error in your experimental result.

$$\text{Ab. error} = (\Delta H)_{known} - (\Delta H)_{calculated}$$

$$\text{Ab. error} = 34.89 - (\Delta H)_{calculated}$$

6. Calculate percentage error.

$$\% \text{ error} = (\text{Ab. error}/((\Delta H)_{known}) \times 100$$

Result:

1. Heat of solution = _____KJ/mole.
2. Absolute Error = _____

Pre-lab Questions:

1. What is the enthalpy of dissolution?
2. Why do we use a calorimeter in this experiment?
3. Define endothermic and exothermic dissolution.
4. What precautions are necessary while recording temperature?

Post-lab Questions:

1. Why is KNO_3 used in this experiment?
2. What would happen if the solution is not stirred properly?
3. Why is specific heat capacity of water taken as 4.18 J/g·°C?
4. How can heat loss to surroundings be minimized?

🔬 Experiment 7: Determination of Viscosity of Aqueous Solutions at Room Temperature

Aim:

To determine viscosity of aqueous solutions of (i) polymer (B) ethanol and (iii) sugar (any two) at room temperature.

Requirements:

Ostwald's viscometer, distilled water, 10% ethanol solution, 10% Sugar solution, 1% Polymer solution, stopwatch, beakers, 10 cm^3 pipette, etc.

Theory:

Viscosity is the resistance offered by a liquid to flow. It is caused by internal friction between the layers of fluid. The greater the viscosity, the slower the flow. The SI unit is **Pascal-second (Pa·s)**, but **poise** or **centipoise (cP)** is commonly used in the lab.

Time period required for the liquid to pass between two fixed points X and Y in the Ostwald's viscometer is called flow time or efflux time. Using the relation between the flow time and the viscosity of the solution, it is possible to find the viscosity of the given liquid or Solution

As it is difficult to measure the radius of the capillary accurately it is advisable to compare the flowtimes for the two same volumes of two liquids and the ratio of their viscosities. Knowing the absolute viscosity of one liquid, that of the other can be calculated. For comparison of viscosities, usually distilled water is preferred. If two liquids with viscosities η_1 and η_2 have the densities d_1 and d_2 and their flow times are t_1 and t_2 respectively for the same volumes, then; substituting the known values of viscosity of distilled water (η_1), density of distilled water (d_1), density of given liquid (d_2) and experimentally determined values of flow times of distilled water (t_1) and given liquid (t_2), the viscosity of given liquid (η_2) is calculated.

Procedure:

1. Clean the viscometer with distilled water, rinse it with acetone and dry it.
2. Pipette out-10 cm^3 of the distilled water and transfer it to big bulb A' of viscometer
3. Using rubber tubing suck the distilled water above the mark 'X' in bulb 'B' and screw clip the rubber tubing.
4. Gradually loosen the screw clip and allow the distilled water to reflow through the capillary tube under its own weight.
5. When distilled water touches the mark upper 'X', start the stop watch and when it touches the lower mark 'Y' stop the stop watch. Note down the flow-time in seconds.
6. Repeating the procedure, adjust the screw clip position in such a manner that, for distilled water it will give flow-time of about 70 to 80 seconds.
7. Determine the exact flow-time of distilled water in seconds (t_1).

8. Remove the distilled water from the viscometer and dry it.
9. Pipette out 10cm^3 of the given solutions and transfer it to big bulb 'A' and following the same procedure determine the flow-time in seconds (t_2).

Observations:

Room temperature :_____ K.

Density of distilled water (d_l) : _____ g/cm^3 (Given)

Density of 10% ethanol solution (d_2) : _____ g/cm^3 (Given)

Density of 10% sugar solution (d_2) : _____ g/cm^3 (Given)

Density of 1% Polymer solution (d_2) : _____ g/cm^3 (Given)

Viscosity of distilled water (η_1) : 0.00855 Poise (27 ºC) (Given)

Flow time of distilled water (t_1) : _____ seconds

Liquids	Flow times (Seconds)				Viscosity
	I	II	III	Mean	
Sugar solution					
Ethanol solution					
Polymer solution					

Calculations:

The viscosity of the solution is calculated using the relation,

$$\frac{\eta_l}{\eta_w} = \frac{d_l t_l}{d_w t_w} \quad \therefore \eta_l = \frac{t_l}{t_w} x \frac{d_l}{d_w} x \eta_w$$

Results:

Sr. No.	Solutions	Viscosity in Poise
1	Sugar solution	
2	Ethanol solution	
3	Polymer solution	

Pre-lab Questions:

1. What is viscosity and on what factors does it depend?
2. Why is water used as a reference in this method?
3. What are the units of viscosity?
4. What is the use of Ostwald's viscometer?

Post-lab Questions:

1. How does temperature affect viscosity?
2. Why should the viscometer be cleaned and dried before use?
3. Which solution showed higher viscosity and why?
4. Can this method be used for non-aqueous liquids?

🔬 Experiment 8: Determination of the rate constant for the hydrolysis of ester

Aim: To determine the rate constant of hydrolysis of Methyl Acetate using HCl as catalyst.

Requirements: Two glass stoppered (reagent) bottles, water bath, 1oo cm^3 measuring cylinder, 0.5 N HCl, 0.1 N NaOH, Methyl acetate, crushed ice, and phenolphthalein indicator.

Theory:

The reaction for hydrolysis of methyl acetate in absence of H^+ ions is a slower reaction and it is given by

$$CH_3COOCH_3 + H_2O \xrightarrow{H^+ \text{ catalyst}} CH_3COOH + CH_3OH$$

Here, methyl acetate reacts with water to give acetic acid and methanol in presence of mineral acid (HCl). The mineral acid (HCl) is used as a catalyst.

In reaction water is taken in excess and therefore concentration of water practically remains constant during the reaction. Also, concentration of H^+ inos which catalysed the reactions also remains constant. Thus, though the reaction is bimolecular, due to excess of water, the rate of reaction depends only on the concentration of methyl acetate and not on the concentration of water. Therefore the reaction is of first order and it is also called as pseudo unimolecular reaction. The large excess of water also prevents the reverse reaction.

The rate constant of this first order reaction is,

$$Rate\ constant\ k = \frac{2.303}{t}\log\frac{a}{a-x}$$

Where,

k is rate or velocity constant of the reaction, t is time, a is initial concentration of reactant (Methyl acetate), x is concentration of reactant (methyl acetate) reacted at time t, (a-x) is concentration of remaining unreacted at time t.

Thus rate constant of the reaction is determined by knowing the values of a and (a-x). the values of a and (a-x) are obtained by titrating definite volume of reaction mixture against standard NaOH solution at definite time interval. During titration following types titre readings are obtained.

T_0 is the reading at zero time (When hydrolysis of methyl acetate is not started).

T_t is the reading at time t.

T_∞ is the titre reading at infinite time (When hydrolysis of methyl acetate is completed).

From the above, a and (a-x) can be determined as

$$a = (T_\infty - T_0) and\ (a-x) = (T_\infty - T_t)$$

Also, T_t is defined as titer readings at definite time intervals which show concentration of methyl acetate reacted or concentration of acetic acid produced at a particular time.

Procedure:

1. Pipette out 5 cm^3 of methyl acetate in one dry glass stoppered bottle (Bottle No. 1)

2. Take 100 cm^3 of 0.5 N HCl using measuring cylinder in another stoppered bottle (bottle no. 2)

3. Keep both the bottles in the water bath to attain room temperature (about 5 minutes).

4. Fill the burette with 0.1 N NaOH.

5. To the conical flask add one piece of ice and two drops of phenolphthalein indicator and keep it ready for titration.

6. Transfer the content of bottle no.2 to bottle no.1 and shake the bottle for uniform mixing. Note the time of mixing as zero time. Keep this reaction mixture in a water bath throughout the experiment.

7. After mixing, immediately pipette out 5 cm^3 of reaction mixture in a conical flask containing ice and phenolphthalein indicator and titrate against 0.1N NaOH. The end point is colorless to just pink. Note the reading in the observation table.

8. Similarly, titrate 5 cm3 of reaction mixture at intervals of 10,20,30,40 and 50 minutes from the zero time against 0.1 N NaOH and record the readings.

Given:

T_∞ = ---------- cm^3 (To be given by the examiner)

Observations

1. Burette reading for zero time (Immediately after mixing) = T_0 = ---------- cm^3

2. T_0 =--------- cm^3

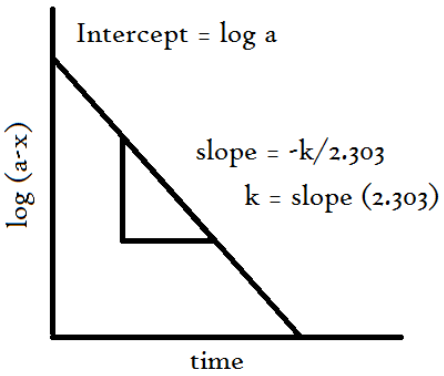
$a = (T_\infty - T_0)$ = ---------- cm^3

Observation Table:

Time (t) min	**Titer/ Burette Reading Tt (cm^3)**	**a= $(T_\infty - T_0)$**	**(a-x)= $T_\infty - T_t$ (cm^3)**	**log(a-x)**	**$\log\frac{a}{(a-x)}$**	**$k = \frac{2.303}{t}\log\frac{a}{a-x}$**
10						
20						
30						
40						
50						

Graph:

Plot the graph of log(a-x) against time and determine the slope.



Calculation:

1. (a-x)= $T_\infty - T_t$
2. log(a-x)
3. $k = \frac{2.303}{t} \log \frac{a}{(a-x)}$
4. Mean K (by calculation)
5. k = (-2.303)xslope (by graph)

Result:

1. The rate constant for hydrolysis of methyl acetate is k = _____ min^{-1} (by graph)

2. The rate constant for hydrolysis of methyl acetate is k = _____ min^{-1} (by calculation)

Conclusion: As the graph of log (a-x) against time is a straight line k values are constant, it is concluded that the hydrolysis of methyl acetate is first order reaction.

Pre-lab Questions:

1. What is meant by pseudo first-order reaction?
2. Why is HCl added to the ester in this experiment?
3. What is the role of phenolphthalein in this experiment?
4. Why is the reaction mixture quenched with ice before titration?

Post-lab Questions:

1. How reaction is quenched?
2. Why is excess water necessary for pseudo first-order conditions?
3. How do you ensure that the reaction has gone to completion?
4. What sources of error may affect the rate constant calculation?
5. Why do we take average of several k values instead of one?

Gravimetric Analysis

The quantitative analysis involves:

1. Gravimetric analysis

2. Volumetric analysis

Gravimetric analysis involves the determination of some constituent of a given sample by weighing operation. In volumetric analysis the substance to be determined is in the solution form and is made to react with the measured volume of a solution of known concentration. In short gravimetric analysis involves the measurement in terms of weights; whereas volumetric analysis involves the measurement in terms of volume.

In this experiment, the procedure mainly involves heating, cooling, and weighing of the crucible. Desiccator is used for cooling and keeping of the crucible. It contains anhydrous calcium chloride which absorbs moisture thus providing dry air for cooling. This avoids adsorption of water molecules on the surface of the crucible. The crucible made up of Porcelain, Silica or Platinum are used for gravimetric analysis.

Following precautions should be kept in mind during the heating of crucible:

- For heating the crucible, always use blue (non sooty) flame. If it is heated on yellow (sooty) flame, the soot will be deposited on crucible which will introduce the errors in the result.
- For heating the crucible, always use clean pair of tongs. Do not handle it with fingers.
- After heating the crucible, keep it on the asbestos sheet for cooling. Do not keep it directly on the table.
- Then keep the crucible in the desiccator. Do not keep a very hot crucible in desiccators; otherwise, it will take longer time for cooling. Vaseline is used to make the desiccators airtight and as for the smooth motion of the desiccators.

Substances commonly used in desiccators (in lower compartment) are anhydrous calcium chloride (widely used), concentrated sulphuric acid, magnesium perchlorate, calcium sulphate and solid potassium hydroxide.

🔬 Experiment 9: Determination of percentage composition of $BaSO_4$ and NH_4Cl in the given mixture gravimetrically.

Aim:

To determine percentage composition of $BaSO_4$ and NH_4Cl in the given mixture gravimetrically.

Requirements:

Crucible with lid, Pair of tongs, Asbestos sheet, Pipe clay triangle/sand bath, Desiccator, Balance etc.

Theory:

On heating entire quantity of NH_4Cl is sublimated. Loss is due to sublimation of NH_4Cl from the mixture and is not due to the $BaSO_4$

Reaction:

$$BaSO_4 + NH_4Cl \xrightarrow{\triangle} BaSO_4 + NH_4Cl$$

Original — Original — $(NH_3 + HCl)$

17.0337 + 36.4609 = 53.4846

Procedure:

1. Wash the crucible and lid with dil. HNO_3 and rinse with water.

2. Dry it with clean piece of cloth and heat it for 90 seconds. Cool it to room temperature and weigh the empty crucible (W1) and place it in desiccator.

3. Then weigh accurately about 1 gm (accurately weighed between 0.8 to 1.2g but not exactly 1 gm) of the supplied mixture to the crucible (W2).

4. Heat the crucible with partially open lid for about 20-25 minutes and see that weather the sublimation is deposited on inners side of the lid of the crucible, if deposited heat it. 5. Cool the crucible nearly to the room temperature and weigh the crucible (W3-weight of the residue after first heating). Record the weight.

6. Again, heat the crucible for about 5 minutes, cool it, weigh the crucible (W4-weight of residue after second heating).

7. Record your observation in the following tabular form.

Observations:

1) Weight of empty crucible = W_1= ----------gms

2) Weight of crucible + mixture = W_2= ---------gms

∴ Weight of mixture = W_2-W_1= ------ m gms

3) Weight of crucible + residue = W_3= --------gms (after first heating)

4) Weight of crucible + residue = W_4=---------gms (after second heating)

∴ Loss in weight on heating =(W_2-W_4)= ------- x gms

Thus m gm of the mixture lost x gm

1 gm of the mixture lost = x/m =**p** gm loss due to NH_3 and HCl

Calculations:-

Thus, loss in weight is due to the loss of NH_3 and HCl from 1 mole of NH_4Cl that decomposed on heating

Amount of NH_4Cl in I gm mixture= p gm

Therefore Amount of NH_4Cl in 100 gm mixture = $p \times 100\ gm = A\ \%$

Therefore Amount of $BaSO_4$ in 100 gm mixture = $100 - A\ gm = B\ \%$

Results:

Weight of mixture taken (gm)	Loss in Weight (gm)	Loss/gram (p)	% composition
			NH_4Cl =
			$BaSO_4$ =

Pre-lab Questions:

1. What is the principle of gravimetric analysis?
2. Why is $BaSO_4$ chosen for gravimetric estimation?
3. What happens to NH_4Cl on heating?
4. Why is a constant weight necessary before final weighing?
5. What are the precautions taken while igniting the mixture?
6. Why should the crucible be cooled in a desiccator before weighing?

Post-lab Questions:

1. How is the percentage of $BaSO_4$ calculated in the mixture?
2. Why does NH_4Cl not appear in the final residue?
3. What is the role of heating in this analysis?
4. How does incomplete decomposition of NH_4Cl affect the result?
5. Why is it important to weigh the crucible before and after ignition?
6. What sources of error could affect the accuracy of the gravimetric analysis?

🔬 Experiment 10: Determination of percentage composition of ZnO and $ZnCO_3$ in the given mixture gravimetrically.

Aim:

To determine percentage composition of ZnO and $ZnCO_3$ in the given mixture gravimetrically.

Requirements:

Crucible with lid, Pair of tongs, Asbestos sheet, Pipeclay triangle/sandbath, Desiccator, Balance etc.

Theory:

On heating the mixture only $ZnCO_3$ decompose as a CO_2 and ZnO. The weight lost is due to the decomposition of zinc carbonate. Zinc oxide will remain as it is.

Reaction:

$$\underset{\text{(Original)}}{ZnO} + \underset{}{ZnCO_3} \xrightarrow{\Delta} \underset{\text{(Newly formed)}}{ZnO} + \underset{}{CO_2} + \underset{\text{(Original)}}{ZnO}$$

Procedure:

1. Wash the crucible and lid with dil. HNO_3 and rinse with water.

2. Dry it with clean piece of cloth and heat it for 90 seconds. Then cool it to room temperature and place in desiccator.

3. Weigh the empty crucible (W1).

4. Then weigh accurately about 1 gm (accurately weighed between 0.8 to 1.2g but not exact 1 gm) of the supplied mixture to the crucible (W2) place it in desiccator.

5. Heat the crucible with partially open lid on a burner for about 20-25 minutes. Then cool the crucible nearly to the room temperature and weigh the crucible (W3-weight of the residue after first heating). Record the weight.

6. Again, heat the crucible for about 5 minutes, cool it, weigh the crucible (W4-weightr of residue after second heating).

7.Record your observation in the following tabular form.

Observations:

1) Weight of empty crucible = W1 = ----------gms

2) Weight of crucible + mixture = W2 = ---------gms

∴Weight of mixture =W2-W1 = ------ m gms

3) Weight of crucible + residue = W3 = --------gms (after first heating)

4) Weight of crucible + residue = W4 =---------gms (after second heating)

∴ Loss in weight on heating (W2-W4) = ------- x gms

Thus m gm of the mixture lost x gm

1 gm of the mixture lost = x/m = **p** gm

Calculations:-

$$ZnO + ZnCO_3 \xrightarrow{\triangle} ZnO + CO_2 + ZnO$$
$$125.37 \qquad\qquad 44$$

Thus, loss in weight is due to the loss of CO2 from 1 mole of $ZnCO_3$ that decomposed on heating

i.e. Loss of 44 gm of $CO_2 \equiv$ 125.37 gm of $ZnCO_3$

Therefore (Loss/gms i.e.) **p** gms = 125.37 x p / 44 = **n** gms of $ZnCO_3$

Thus, Amount of $ZnCO_3$ in 1 gm mixture = **n** gm

Therefore, Amount of $ZnCO_3$ in 100 gm mixture = $n \times 100\ gm = A\ \%$

Therefore, Amount of ZnO in 100 gm mixture = $100 - A\ gm = B\ \%$

Results:

Weight of mixture taken (gm)	Loss in Weight (gm)	Loss/gram (p)	% composition
			$ZnCO_3$ =
			ZnO =

Pre-lab Questions:

1. What is gravimetric analysis and what are its advantages?
2. What is the chemical reaction involved when mixture is heated?
3. What is the final form (compound) of zinc that is weighed in this experiment?
4. What precautions should be taken while igniting the precipitate in the crucible?

Post-lab Questions:

1. How does ignition help in distinguishing between ZnO and $ZnCO_3$?
2. Why is a desiccator used before weighing the crucible?

🔬 Experiment 11, 12: Purification of Organic Compound by recrystallization.

Aim:

To purify given organic compound by recrystallization

Requirements:

Beaker, Conical flask, funnel, wire gauze, glass rod, Theile's tube, thermometer, filter paper etc.

Procedure:

Part – I

Select solvent to be used for purification (D.W. / water: ethanol / ethanol)

1) For selection of solvent condition is: The substance should be insoluble in cold solvent and soluble in hot condition.

2) Take small amount of substance in a test tube, add 2-3 cm^3 distilled water and heat on wire gauge. If soluble in hot condition and reprecipitation in cold condition. Purify the substance in distilled water.

3) Otherwise carry out procedure in water ethanol mixture (50-50) or 100 % ethanol.

Part – II

i) Take melting point of the given crude substance.

ii) Transfer the given substance in a conical flask and add 10-15 cm^3 salvent (water or water – ethanol) and boil it.

iii) Filter the hot solution through cotton plug in a beaker.

iv) Cool the filtrate under tap water or in ice bath till the crystals are obtained

v) Filter the purified product through ordinary filter paper.

vi) Dry it by pressing using another filter paper and dry it under IR lamp. Then take the weight of purified substance.

Observation:

i) Melting point of the crude substance = _____°C

ii) Weight of purified substance = _____ g

iii) Melting point of the purified substance = _____ °C

iv) Selected Solvent for purification = _____

Result:

i) Melting point of the crude substance = _____°C

ii) Weight of purified substance = _____ g

iii) Melting point of the purified substance = _____ °C

iv) Selected Solvent for purification = _____

Pre-lab Questions:

1. What is recrystallization and why is it used for purification?
2. What are the essential criteria for selecting a suitable solvent for recrystallization?
3. Why is it important that the compound is sparingly soluble in cold solvent and highly soluble in hot solvent?
4. Why should the hot solution be filtered before cooling?
5. What precautions must be taken while heating the solution during recrystallization?

Post-lab Questions:

1. Why is slow cooling preferred over rapid cooling in recrystallization?
2. What could be the reason for obtaining a low yield of crystals?
3. How can you check the purity of the recrystallized compound?
4. What happens if too much solvent is used during recrystallization?
5. What is the purpose of washing the crystals with cold solvent after filtration?
6. What effect would using an impure solvent have on the recrystallization process?

🔬 Experiment 13 to 16: Basic Principles of Organic Compound Characterization

Aim:

To characterize unknown solid organic compounds by performing:

1. Preliminary tests (appearance, odor, ignition test)
2. Solubility tests (chemical nature)

And to recrystallize the compound using appropriate solvent and determine the melting point.

Procedure:

A Systematic Scheme for the identification of the organic compound is outlined below

A. Preliminary Test

B. Solubility Test

C. Detection of Elements

D. Detection of Functional Group

E. Determination of physical constant and Identification of the compound.

In SEM I, only first two tests, preliminary test and solubility test to be conducted followed by recrystallization.

A) Preliminary Test

Test	Observation	Inference
i) Colour	i) Yellow	Nitrobenzene, m-Dinitrobenzene, p- Nitro toluene, nitro phenol, nitro aniline etc. may be present
	iii) Brown	P – Toluidine, resorcinol etc. may be present
	iv) Buff or pinkish	ß – Naphthol may be present
	v) Blackish	α – Naphthol may be present
	vi) Reddish	Aniline, phenol, Aromatic amine etc. may be present
	vii) Colourless	Simple acid, alcohol, ester, ketone, aromatic hydrocarbon etc. may be present
ii) Odour	i) Carbolic	Phenol, cresol etc. may be present

	ii) Fishy	Amine may be present
	iii) Sweet and pleasant	Ester, alcohol and halogen derivatives etc. may be present
	iv) Bitter almonds	Nitrobenzene, Benzaldehyde etc. may be present
	v) Moth balls (Strong and characteristic)	Naphthalene may be present
	vi) No particular smell	Aromatic acid, amide, carbohydrate etc. may be present
iii) State	Solid / Liquid	-----
iv) Unsaturation test		
$KMnO_4$ test Substance + 2 ml of water shake well + 2 drops of dilute $KMnO_4$ solution.	**i)** Decolourisation of $KMnO_4$ **ii)** No decolourisation	Unsaturated or easily oxidizable compound may be present. Saturated compound may be present.
Bromine Test **For water insoluble subs.** Substance + chloroform … shake and add Bromine in CCl_4 **For water soluble subs.** Substance + 2 ml water shake + Bromine water	**i)** Decolourisation **ii)** Decolourisation with formation of precipitate. **iii)** No decolourisation.	Unsaturated compound may be present. Easily substituted compound may be present. Saturated compound may be present
v) Copper foil test Heat copper foil till red hot. Put small quantity of subs. on copper foil and observe the flame.	**i)** Sooty flame	Aromatic compound may be present
	ii) Non sooty flame	Aliphatic compound may be present
	iii) Initially sooty finally bluish green	Aliphatic or aromatic halogenated compound may be present.

B) Solubility test

Determination of Chemical nature of the substance

Perform the following tests If substance is insoluble / immiscible in water.		
Test	Observation	Inference
i) substance + water …shake well**.**	Insoluble	Water insoluble compound present
ii) Subs. + sat. $NaHCO_3$	Soluble with strong effervescence	Carboxylic acid present
(If sub. Dissolves and clear solution is obtained) To this clear solution add conc. HCl drop by drop.	A solid appear	Carboxylic acid present

iii) Subs. + NaOH	Soluble	Phenolic compound present
(If sub. dissolves, clear solution is obtained) To this clear solution add conc. HCl drop by drop.	A solid appear	Phenolic compound present
iv) Subs + HCl	Soluble	Base present
(If sub. dissolves, clear solution is obtained) To this clear solution add conc. NaOH drop by drop.	A solid or emulsion appear	Base present
v) Substance is insoluble in all (water, $NaHCO_3$, NaOH, HCl)	----	Water insoluble neutral compound present

[For solid substance use word soluble or insoluble, for liquid substance use word miscible or immiscible]

Conclusion: Given substance is water insoluble / immiscible ----------

Perform the following tests If substance is soluble / miscible in water.		
Test	Observation	Inference
i) substance + water …shake well. Use this solution for further analysis	soluble (subs. dissolves)	Water soluble compound present. (Lower member of alcohol, ester ketone, carbohydrate)
Test solution with litmus paper.	Blue litmus paper turns red Red litmus paper turns blue No action on the litmus paper	Water soluble acid or phenol present. Water soluble base present Water soluble neutral present
ii) Solution + sat. $NaHCO_3$	Effervescences No effervescences	Water soluble acid present Water soluble acid absent
iii) Solution + alcoholic $FeCl_3$	Blue to violet colour No characteristic coloration	Water soluble phenol present Water soluble phenol absent
iv) Effect on red litmus paper	Red litmus turns blue No effect on red litmus	Water soluble base present Water soluble base absent

Conclusion: Given substance is water soluble ----------

Recrystallize the given compound and take melting point of dried purified compound.

Result-

1. The given compound is water soluble / water insoluble ----------
2. Physical constant (M.P.) of recrystallized compound -

Pre-Lab Questions

1. What are the key physical properties that help in the identification of an organic compound?
2. Why is the flame test useful in organic compound characterization?
3. What is the principle behind the solubility test?
4. Why must we dry a compound before measuring its melting point?

Post-Lab Questions

1. Specify odours of organic compounds and relation with functional group.
2. How Copper foil test is useful to determine structure of compounds?
3. What inference can be drawn from unsaturation test?

SEM II

Index

Group A

Skill Experiments

🔬 **Experiment 1:** Conductometric Titration of a Strong Acid with a Strong Base

Aim: To determine the amount of a strong acid present in the given acid solution by titrating it against strong base conductometrically.

Requirements: Conductometer and conductivity cell, burette, beaker, pipette, conductivity water, 0.1N NaOH solution, 1N HCl solution etc.

Theory:

Titration of strong acid (HCl) against a strong base (NaOH).

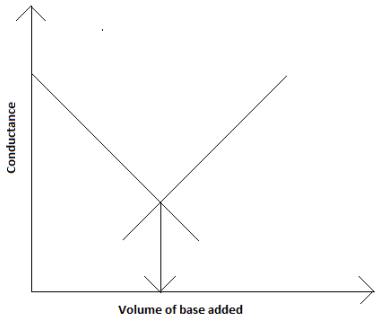


Fig. 1. Conductometric titration curve

(1) The initial conductance is high due to the complete dissociation of strong acid (HCL) and H^+ ions have high conductance value.

(2) The addition of strong base (NaOH) in the acid solution gives the following reaction

$$H^+ + Cl^- + Na^+ + OH^- \longrightarrow NaCl + H_2O$$

(3) As NaOH is added, H^+ ions combine with OH^- ions to form undissociated water molecules and H^+ ions get replaced by Na^+' ions having much lower conductance value. Hence, the conductance of the solution goes on decreasing and reaches a minimum (at equivalence point). After the equivalence point, NaOH added remains unreacted. Being a strong base, it ionises completely producing OH^- ions of high conductivity. Hence, conductance increases again.

(4) A V shaped curve is obtained. The point of intersection of the two segments in the graph gives the end point of the titration. (Fig. 1).

Procedure:

(1) Dilute the given acid solution using conductivity water to 100 cm^3 in a standard measuring flask. Shake the solution well.

(2) Rinse and fill the burette with supplied 0.1N NaOH solution.

(3) Pipette out 10 cm^3 of the diluted solution in a clean 100 cm^3 beaker.

(4) Place the conductivity cell in the beaker containing acid solution and add just sufficient quantity of conductivity water to it, so that platinum plates immerse in solution completely.

(5) Insert a glass rod and stir the solution well. Do not take it out of beaker till titration is complete.

(6) Connect the cell to the conductometer and determine the initial conductance of the solution by selecting the proper range (see instructions).

(7) Add a small volume (say 0.5 cm^3) of NaOH solution at a time from the burette, stir the solution well and note the resultant conductance value.

(8) Repeat step No. 7 until the conductance values start increasing. Take 5 to 6 more readings

(9) Plot the graph of conductance against the volume of NaOH added (Fig. 2.31. It gives a V shaped curve.

(10) The volume corresponding to the intersection of two lines (V_x) gives the end point of titration. Note the volume V_x.

(11) Calculate the normality and strength of the acid solution as given in the calculations.

<u>Observations and Calculations:</u>

(1) Temperature = _____ ^0C [_____K]

(2) Normality of NaOH solution =0.1N

(3) Volume of the diluted acid pipetted out = 10 cm^3

(4) Equivalent weight of the acid (HCl)=36.5.

Observation Table:

Observation Numbers	Volume of NaOH added in cm^3	Conductance (1/R) mhos [S]
1	0.0	
2	0.5	
3	1.0	

Calculations:

(1) Volume of NaOH solution required for end point (V_x) =_____cm^3 [from graph] …… (1)

(2) $Normality\ of\ HCl = \frac{N_{NaOH} \times V_x}{V_{HCl}} = \frac{0.1 \times V_x}{10} =$ _____N

(3) 10 cm^3 of the diluted solution required V_x cm^3 of 0.1N NaOH solution.

∴100cm^3 of the diluted solution will require 10 V_x, cm^3 of 0.1N NaOH solution... (2)

1000 cm^3 of 1 N NaOH = 36.5 g of HCl

$$\therefore 10V_x cm^3\ of\ NaOH = \frac{36.54 \times 10V_x \times 0.1}{1000} \ldots\ldots (3)$$

$$= \_\_\_\_\ g\ HCl$$

(Note: While doing calculations, substitute the titration reading obtained in place of V_x)

Results:

(1) Volume of NaOH solution required for the end point (V_x) = _____ cm^3.

(2) Normality HCl = _____ N.

(3) Amount of HCl present in the given solution= _____g.

Pre-lab Questions

1. What is the basic principle of conductometric titration?
2. Why is conductivity high for strong acids?
3. What is the unit of conductivity?
4. Why is stirring necessary during conductometric titration?

Post-lab Questions

1. What are the changes observed in conductance during experiment?
2. What happens to conductivity when NaOH is added to HCl before the equivalence point?
3. Why does conductivity increase after the equivalence point?
4. How is the equivalence point located from the graph?
5. Would the conductivity pattern be different if we titrated a weak acid with a strong base? Explain.

🔬 **Experiment 2:** Determination of Dissociation Constant (Ka) of Weak Acid using Henderson's Equation (pH-metric Method)

Aim: To determine dissociation constant (Ka) of weak acid (CH_3COOH) using Henderson's equation pH metrically.

Requirements: pH meter, combined glass electrode, burette, 25 cm^3 pipetted, 100 cm^3 beaker, glass rod or magnetic needle etc.

Theory: The acid dissociation constant or acid ionization constant is an equilibrium constant which refers to the dissociation or ionization of an acid. An acid dissociation constant is a quantitative measure of the strength of an acid in solution.

The weak acid (like CH_3COOH) dissociates partially in solution and hence its dissociation reaction is reversible which forms an equilibrium. The weak acid is dissociated as

$$HA \rightleftharpoons H^+ + A^-$$

The equilibrium constant for the above reaction is given as :

$$K_a = \frac{[H^+][A^-]}{[HA]}$$

$$\therefore [H^+] = \frac{[HA]}{[A^-]}\ K_a$$

On taking logs on both side

$$log[H^+] = \log\left(\frac{[HA]}{[A^-]}\ K_a\right)$$

$$log[H^+] = \log\frac{[HA]}{[A^-]} + \log K_a$$

$$-log[H^+] = -\log\frac{[HA]}{[A^-]} - \log K_a$$

$$pH = pK_a - \log\frac{[HA]}{[A^-]}$$

$$pH = pK_a + \log\frac{[A^-]}{[HA]}$$

The above equation is called as Hinderson – Hasselbalch equation and it is used to determine dissociation constant of weak acid. The equation is re arranged as

$$pK_a = pH - \log\frac{[A^-]}{[HA]}$$

It is concluded from equation that by knowing values of pH, $[A^-]$ and [HA], the values of pK_a of the weak acid is determined and form pK_a the value of K_a is determined.

In the current experiment, the CH_3COOH is titrated with NaOH. During titration CH_3COOH reacts with NaOH and the salt CH_3COONa is formed. The reaction between CH_3COOH and NaOH is represented as,

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

For the above reaction, $[A^-]$ = [CH3COONa] = Salt and [HA] = [CH3COOH] = [Acid] and therefore the Hinderson – Hasselbalch equationis written as,

$$pK_a = pH - \log\frac{Salt}{Acid}$$

During titration, a definite of NaOH is added to CH_3COOH and for each addition pH is measured experimentally. Also [Salt] and [Acid] are measure from experimental data. By using the values of pH, $[A^-]$ and [HA], the value of pKa of the weak acid is determined and from pKa, the value of Ka is determined.

Procedure:

1. Fill the burette with 0.1 N NaOH solution.

2. Pipette out 25 cm^3 of 0.1 N CH_3COOH in 100 cm^3 dry beaker and immerse the wash and cleaned combined glass electrode of pH meter to the same beaker.

3. Add 2 cm^3 of 0.1 N NaOH solution from the burette to the same beaker. Stir the solution and record its pH.

4. Continue the addition of 2 cm^3 of 0.1 N NaOH solution from the burette to the same beaker till 12 cm^3 and record the pH of the solution after each addition.

Observation:

Volume of CH_3COOH acid taken = V = 25 cm^3.

Observation Table:

Sr. No	Volume of 0.1 N NaOH solution added V_1 cm^3	pH	Volume of unreacted Acid V_2 cm^3	mmole of salt formed [Salt] (mmole)	Mmole of unreacted Acid [Acid] (mmole)	$\frac{[Salt]}{[Acid]}$	$\log\frac{[Salt]}{[Acid]}$	pK_a	Mean pK_a
1	2								
2	4								
3	6								
4	8								

5	10								
6	12								

Calculations:

1. Volume of unreacted acid:

When 2 cm^3 of 0.1 N NaOH solution is added to the beaker having 25 cm^3 of 0.1 N CH_3COOH. During the reaction 2 cm^3 of salt is formed and amount of acid remains unreacted is 25 -2 = 23 cm^3.

∴ Volume of acid remains unreacted = $V_2 = V - V_1$

2. mmol of the salt formed

[Salt] = (Volume of salt formed) (Concentration of Salt)

But,

Volume of salt formed = volume of NaOH added =V_1

Concentration of salt = Concentration of NaOH

∴ [Salt] = (Volume of NaOH added) (Concentration of NaOH)

∴[Salt] = $(V_1)(0.1)$

3. mmol of unreacted acid

[Acid] = (Volume of Unreacted Acid) (Concentration of Unreacted Acid)

∴ [Acid] = $(V_2)(0.1)$

4. Calculate $\frac{[Salt]}{[Acid]}$

5. Calculate $\log\frac{[Salt]}{[Acid]}$

6. $pK_a = pH - \log\frac{Salt}{Acid}$

7. Mean $pK_a = \frac{(pK_a)_1+(pK_a)_2+(pK_a)_3+(pK_a)_4+(pK_a)_5+(pK_a)_6}{6}$

8. $K_a = Antilog(-MeanpK_a)$

Result:

Dissociation constant of CH_3COOH = K_a= _____

Pre-lab Questions

1. State the Henderson–Hasselbalch equation.
2. What is the significance of the half-neutralization point in this experiment?
3. Why is a pH meter preferred over indicators for weak acid–strong base titrations?
4. What is the typical pKa value for acetic acid?

Post-lab Questions

1. Why does the pH at half-neutralization equal pKa for a weak acid?
2. How would the titration curve change for a strong acid instead of a weak acid?
3. What is the effect of ionic strength on the dissociation constant?

🔬 **Experiment 3:** Verification of Beer–Lambert's Law using $KMnO_4$ solution by colourimetry.

Aim: To Verify Beer-Lamberts Law Using $KMnO_4$ Solution by colourimetric Method.

Requirements: Colourimeter, cuvettes, two 10 cm^3 graduated pipettes, five stoppered test tube, etc. 0.001 M $KMnO_4$ and distilled water.

Theory: When beam of monochromatic radiation is incident on a coloured solution, part of radiation is absorbed by the solution and remaining part is transmitted through the solution. The transmitted radiation has less intensity as compared to intensity of incident tradition. The process is explained by Beer-Lamberts Law. The Beer-Lamberts law states that 'Equal fraction of incident radiation is absorbed by successive layers of absorbing medium of equal thickness and concentration.'

According to this law, the absorption of radiation by the coloured species depends on path length and concentration of the solution. The law shows relationship between absorption of radiation and concentration of the solution and it is mathematically expressed as;

$$A = \epsilon C l$$

Where,

A is absorption of radiation by the solution $A = log \frac{I_0}{I_t}$

ϵ is constant called as molar absorptivity
C is concentration of solution
l is path length
I_o is intensity if incident radiation
I_t is intensity of transmitted radiation

At a particular wavelength, the solution absorbs maximum amount of radiation and such wavelength is called as λ_{max}.

The Beer-Lambert law is verified by measuring the absorbance of the solution at different concentration and form the graph of absorbance against concentration. The straight line obtained from the graph of absorbance against concentration of solution shows the validity of Beer-Lambert law.

Procedure:

Part – I (Determination of λ_{max})

1. Pipette out 3 cm^3 of 0.001 M $KMnO_4$ solution in a stoppered test tube. Add 7 cm^3 of distilled water by pipette to the same test tube and shake the solution well.
2. Take sufficient quantity of this solution in cuvette and place the cuvette in sample holder of colourimeter.
3. Measure the absorbance of the solution at different filters and wavelengths by using distilled water as a blank. (Before measuring the absorbance, standardize the colourimeter by using distilled water as a blank for every wavelength)
4. Note the reading in the table and selects the wavelength which shows maximum absorbance. The maximum absorbance is called λ_{max}.

Part – II (Verification of Beer-Lambert Law)

1. Prepare the following solutions in stoppered test tubes by using graduated pipettes.

Test tube No.	Volume of 0.001 M $KMnO_4$ Solution (cm^3)	Volume of Distilled Water (cm^3)
1	1	9
2	2	8
3	3	7
4	4	6
5	5	5

2. Measure the absorbance of each of the above solutions at wavelength selected as λ_{max} by using distilled water as a blank and note the readings.

Observation Tables:

Part – I (Determination of λ_{max})

Sr. No	Wavelength (nm)	Absorbance
1		
2		
3		
4		
5		

The Wavelength which shows maximum absorbance = λ_{max} = _____nm

Part – II (Verification of Beer – Lamberts Law)

Test Tube No	Volume of 0.001 M $KMnO_4$ Solution (cm^3)	Volume of Distilled Water (cm^3)	Absorbance	Concentration of $KMnO_4$ solution (M)
1	1	9		
2	2	8		
3	3	7		

4	4	6		
5	5	5		

Calculations:

1. Concentration of $KMnO_4$ solution in test tube no. 1

$$KMnO_4 \text{ (Initial)} \equiv KMnO_4 \text{ (Final)}$$

$$M_1V_1 = M_2V_2$$

$$0.001 \times 1 = M_2 \times 10$$

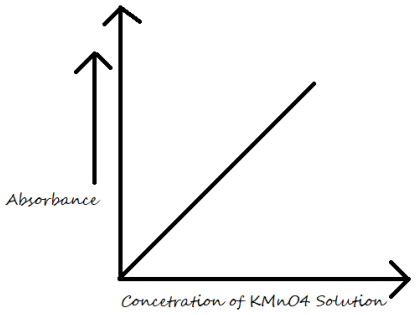
$$\therefore M_2 = \frac{0.001\ x1}{10}$$

The concentration of $KmNO_4$ solution in test tube no.1= M_2 =M

Calculate concentration of $KMnO_4$ solution (M) for remaining solutions.

Graph

Plot a graph of absorbance against concentration of $KMnO_4$ solution.



Result:

1. Maximum wavelength = λ_{max} = _____nm

2. The straight line obtained from the graph of absorbance against concentration of KMnO4 solution shows the validity of Beer – Lambert law.

Pre-lab Questions

1. State Beer–Lambert's Law.
2. What is the significance of λmax in colorimetric analysis?
3. Why is $KMnO_4$ suitable for this experiment?
4. How is absorbance related to transmittance?

Post-lab Questions

1. What would happen if you used a wavelength far from λmax?
2. Why should the Beer–Lambert's law plot pass through the origin?
3. What are the limitations of Beer–Lambert's Law?

4. How can this calibration curve be used to find the concentration of an unknown solution?

🔬 **Experiment 4:** Standardization of a Commercial Sample of HCl using Borax and Preparation of Material Safety Data Sheet (MSDS) for the Chemicals Involved

Aim: To Standardize Commercial Sample of HCl using Borax and to Write Material Safety Data of the Chemicals Involved.

Requirements: Burette, 250cm^3 conical flask, 25 cm^3 pipette, etc.

0.2N (Approximately) HCl, borax, previously boiled and cooled distilled water and 1% methyl red indicator.

Theory: From standardization the exact concentration of solution is determined. In order to determine exact concentration of solution, the solution is compared with another solution. This solution whose exact concentration is known is called as standard.

Hydrochloric acid is widely used for many laboratory reactions. Hence, it is necessary to standardize HCl solution when it is required for any laboratory work. For the standardization Sodium Tetraborate Decahydrate (Borax) is used as a primary standard. Borax has relatively high equivalent mass and is available in highly pure form (99.99%). It does not decompose at room temperature (non-hygroscopic) and reacts with another substance with a known stoichiometry. It can be easily weighed and used directly for standardization.

For standardization of HCl solution, borax solution is prepared and it is titrated with HCl solution.

In water, the borax dissociates as,

$$Na_2B_4O_7.10H_2O \longrightarrow 2Na^+ + B_4O_7^{2-} + 10H_2O$$

Anion formed in the dissociation reaction is hydrolyzed in water and the reaction is,

$$B_4O_7^{2-} + 7H_2O \longrightarrow 4H_3BO_3 + 2OH^-$$

During hydrolysis, hydroxyl ions (OH-) are liberated which react with HCl during standardization process and the reaction is represented as,

$$2OH^- + 2HCl \longrightarrow 2H_2O + 2Cl^-$$

The overall reaction of the above process is,

$$Na_2B_4O_7.10H_2O + 2HCl \longrightarrow 4H_3BO_3 + 2NaCl + 5H_2O$$

At the end point, all OH^- ions are consumed and only boric acid (H_3BO_3) has remained in the solution. But boric acid is so weak that it does not affect the end point. At the end point the pH of the solution is 5.1. Therefore, methyl red is the suitable indicator for the titration.

Procedure:

1. Fill the burette with supplied HCl solution.

2. Weigh accurately 0.38 g of borax in 250cm^3 conical flask.

3. Add 50 cm^3 of previously boiled and cooled distilled water in the same conical flask and shake the flask to dissolve borax.

4. Add two drops of 1% methyl red indicator to the same conical flask and titrate the total content of the conical flask against HCl solution. The end point is yellow to pink. Note the burette reading in the observation table.

5. Repeat the same procedure and take one more reading.

Observation Table:

Burette Level	Burette Reading			Constant Burette Reading (cm^3)
	1	2	3	
Initial				
Final				
Difference				

Calculation:

From the reaction

$$2HCl \equiv Na_2B_4O_7.10H_2O$$

2(1000 cm3 of 1 N HCl) ≡ 381.7 g of Borax

(1000 cm3 of 1 N HCl) ≡ $\frac{381.7g}{2}$ of Borax

(1000 cm3 of 1 N HCl) ≡ 190.68 g of Borax

(B.R cm^3 of x N HCl) ≡ w g of borax

$$(B.R.)(x)(190.68) = (1000)(1)(w)$$

$$\therefore x = \frac{(1000)(1)(w)}{(B.R)(190.68)}$$

∴ Normality of HCl = x= _____N

Result:

Normality of HCl = x= _____N

Material Safety Data of the Chemicals Involved in the above Experiment

Material Safety Data Sheet (MSDS) for HCl

1. Name
   - Chemical Name: Hydrochloric acid
2. CAS (Chemical Abstract Service) Number
   - 7647-01-0
3. Hazards to Person
   - Eye: Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
   - Skin: Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolour skin.
   - Ingestion: Swallowing hydrochloric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. May cause nausea, vomiting and diarrhea.
   - Inhalation: Inhalation of vapours can cause coughing, choking, inflammation of the nose, throat and upper respiratory tract, and in severe cases, pulmonary edema and circulatory failure.
4. First Aid Measures
   - Eye: Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.
   - Skin: Immediately flush skin with plenty of water for at least 15 minutes. Get medical attention immediately.
   - Ingestion: Do not force to vomiting. Give large quantities of wateGet meille available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
   - Inhalation: Remove from exposure and move to fresh air immediately. If no breathing, give artificial respiration. Get medical attention immediately.
5. Fire Fighting Measures
   - Flash Point: Non-flammable
6. Physical and Chemical Properties
   - Appearance: Clear and fuming liquid
   - Colour: Colourless to slight yellow

- B.P.: 86 °C at 760 mmHg
- M.P.:66 °C
- Odour: Pungent
- Solubility in Water: 100% soluble in water

7. Stability and Reactivity
   - Chemical Stability: Stable at room temperature
   - Hazardous Decomposition Products: When heated to decomposition, emits toxic hydrogen chloride fumes which react with water or steam to produce heat and toxic and corrosive fumes. Thermal oxidative decomposition produces toxic chlorine fumes and explosive hydrogen gas.
   - Hazardous Polymerization: Will not occur
8. Toxicological Information
   - Chronic Effects: May cause damage to the kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, circulatory system and teeth.
   - Carcinogenecity: No carcinogenic effects have noticed.
9. Ecological Information
   - Environmental Toxicity: It is toxic to aquatic life.
   - Biodegradability: When released into the soil, It will not biodegrade

Material Safety Data Sheet (MSDS) for borax

1. Name
   - Chemical Name: Sodium tetraborate decahydrate
   - Market Name: Borax
2. CAS (Chemical Abstract Service) Number
   - 1303-96-4
3. Hazards to Person
   - Eye: May cause eye irritation.
   - Skin: May cause skin irritation.
   - Ingestion: May cause irritation of the digestive tract.
   - Inhalation: May cause respiratory tract irritation.
4. First Aid Measures
   - Eye Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid.
   - Skin: Flush skin with plenty of soap Get medical aid if irritation develops or persists.
   - Ingestion: Do not force to vomiting. If conscious and alert, rinse mouth and drink 24 cups of milk or water. Wash mouth with water.
   - Inhalation: Remove from exposure to fresh air immediately. If not breathing, give artificial respiration. Get medical aid if cough or other symptoms appear.
5. Fire Fighting Measures
   - Flash Point: Non-flammable
6. Physical and Chemical Properties

- Appearance: Solid
- Colour: White
- B.P.: 1575 °C at 760 mmHg
- M.P.: 741 °C
- Odour: Odourless
- Solubility in Water: Partially soluble in water

7. Stability and Reactivity
   - Chemical Stability: Stable at room temperature
   - Hazardous Decomposition Products: Toxic gases and vapors may be released if involved in a fire.
   - Hazardous Polymerization: Will not occur
8. Toxicological Information
   - Chronic Effects: Prolonged or repeated ingestion or skin absorption may cause anorexia, weight loss, vomiting, mild diarrhea, skin rash, convulsions and anemia.
   - Carcinogenicity: No carcinogenic effects have noticed.
9. Ecological Information
   - Environmental Toxicity: Moderately toxic to mammals and low in toxicity to birds, bees, fish and other aquatic organisms.
   - Biodegradability: The chemical itself and its products of degradation are not toxic.

Pre-lab Questions

1. Why is borax considered a primary standard?
2. Write the chemical equation for the reaction between borax and HCl.
3. What is the role of an indicator in titration?
4. Why should borax be dried before weighing?

Post-lab Questions

1. Why is methyl orange preferred over phenolphthalein for this titration?
2. How would your result change if the borax was not completely dissolved?
3. What safety precautions are necessary when handling concentrated HCl?
4. Can borax be used to standardize other acids? Give examples.

Group B

🔬 Experiment 5 - 9: Semi-micro qualitative analysis of inorganic mixtures

Scheme of the Qualitative Analysis of Semi-micro scale.

The qualitative analysis of a mixture deals with the separation, identification of cations, anions and confirmation of the same by characteristic tests.
The analysis is generally carried out in the following manners,

1. Preliminary Tests
2. Preparation of the solution of the mixture for the separation and detection of cations.
3. Separation of cations into groups and identification of cations in each group.
4. Preparation of water extract / sodium carbonate extract for the identification of anions (acidic radicals).

PRELIMINARY TESTS AND DRY TESTS:

A. Physical Examinations :

Test	Observation(s)	Inferences(s)
a. Colour	Blue	Copper Salts
	Green	Ni^{+2},Cu^{+2},Cr^{+3}, Fe^{+3} salts
	Red, Orange	CrO_4^{-}, Bi^{+3}, Pb^{+2} , Cd^{+2}, Hg^{+2}, Fe^{+2}
	Black or Brown	Pb^{+2}, Sb^{+3}, $Cr_2O_7^{-2}$
	Pink or Violet	Mn^{+2} and Co^{+2} salts
	Yellow	Cu^{+2}, Mn^{+2}, Pb^{+2}, Sb^{+3} etc.
	Colourless	Coloured radicals are absent
b. Nature	Large Crystals	Water soluble salts.
	Hygroscopic	Insoluble salts
	Amorphous (Fine Powder)	Al^{+3},Cd^{+2}, Co^{+2} and Mg^{+2}

B. Dry Tests :

Heating Small quantity in a dry test tube	Sublimation (white)	NH_4^{+}, Hg^{+2}, As^{+3} salts
	Violet Fumes	Iodine from iodides.
	Reddish Brown fumes	NO_2 from nitrites and bromine from bromides.
Change in Colour of the substances		
When Cold	**When Hot**	
White	Yellow	Zn^{+2} salts
Yellow	Brown / Red	Bi^{+2},Cd^{+2},Sn^{+} salts
Green	Black	Ni^{+2},Cu^{+2},Cr^{+3} salts
Pink or Buff	Green	Mn^{+2} salts

Coloured Residue		Ni^{+2},Cu^{+2},Cr^{+3}, Co^{+2}, Mn^{+2},Fe^{+2} etc.
White infusible residue		Salts of alkali metals and some salts of Ca^{+2},Sr^{+2},Ba^{+2}, Mg^{+2} etc.

C. Treatment with Acids :

Mixture + dil. HCl or dil H_2SO_4	i. Brick effervescences, no effect on lead acetate paper	CO_3^{2-} present
	ii. Gas turning lead acetate paper black	S^{2-} present
	iii. Reddish brown fumes turning starch iodide paper blue	NO_2^- present
	iv. Colourless gas turning dichromate paper green	SO_3^{2-} present
Warm carefully Mix. + Conc. HCl	i. Gas turning lead acetate paper black S^{2-} present	S^{2-} present
	ii. Evolution of gas turning bleaching litmus paper black	Higher chlorides, chromates, dichromates etc.
Warm carefully Mix. + Conc. H_2SO_4	i. Evolution of CO_3^{2-}	CO_3^{2-} present
	ii. Evolution of Brown fumes	Br^-, NO_3^- present
	iii. Evolution of Violet fumes	I^- present
	iv. Evolution of gas giving white fumes with NH_3	HCl from chlorides present
D. Borate Test Mix. + ethyl alcohol +3-4 drops of Conc. H_2SO_4 stir and ignite ***(Only for SY)***	Green Colored fumes	BO_3^{3-} present
E. NH_4^+ Test Mix. + NaOH	Evolution of NH_3 turning turmeric paper Brown	NH_4^+ present
F. PO_4^{3-} Test Mix. + Conc. HNO_3 + excess ammonium molybdate warm if essential ***(Only for SY)***	Cannery yellow ppt.	PO_4^{3-} present

Conclusion: Interpretation of Dry Tests: i. Possible Cations: …… &
ii. Possible Anions: ……

Examination of cations in solutions:

Preparation of water extract (WE): Take 100 mg of mix. + 10 cm^3 distilled water and heat for 2-3 min. Cool and centrifuge. Use the centrigugate for the analysis of anions and to test K^+ and NH^{4+}.If mix. Is soluble in water, use it for anions and group I and subsequent groups.

Test for NH_4^+ 2-3 drops of *WE* + NaOH	Evolution of NH_3 turning turmeric paper Brown	NH_4^+ present
CT for NH_4^+ 2-3 drops of WE + Nesslers Reagent ($HgCl_2$ + KI till dissolve + equal volumes of NaOH)	Brown ppt. or coloration	NH_4^+ confirmed
Test for K^+ * WE (If NH_4^+ is present, add few drops of NaOH, boil till all NH_3 is evolved) + acetic acid +$HClO_4$ / picric acid * ***Concentrated WE and vigorous shaking is essential.***	Yellow or White ppt.	K^+ present & confirmed
Test for Halogens WE + dil. HNO_3 till acidic + $AgNO_3$	Yellow or White ppt. insoluble in dil. HNO_3	Halogens present & confirmed
Distinguish of Halogens : WE + chlorine water + $CHCl_3$	i. Colourless layer	Cl^- present
	ii. Brown / Yellow layer	Br^- present
	iii. Violet layer	I^- present
Test for NO_2^- 2-3 drops of *WE* + dil. Acetic acid + $FeSO_4$ ***(Only for SY)***	Deep brown ppt. or coloration	NO_2^- present
Test for NO_3^- Fresh solution of Conc. H_2SO_4 & $FeSO_4$ cool + WE	Brown ring	NO_3^- present
Test for CrO_4^- ***(Only for SY)***	If WE is yellow and colour deepen on addition of dil. H_2SO_4	CrO_4^- present
Test for $Cr_2O_7^{-2}$ ***(Only for SY)***	If WE is orange and colour lighten on addition of NaOH	$Cr_2O_7^{-2}$ present
Test for SO_4^{-2} **WE + $Ba(NO_3)_2$**	White ppt insoluble in dil. HNO_3	SO_4^{-2} present

Preparation of positive radical solution:

In of Br^- and / or I^- are present, they interfere with the analysis of the cations and hence they have to be removed as follows:

Mix. + 1-2 mL Conc. HNO_3 in evaporating dish. Evaporate till colored as well as acid fumes ceases. Extract the residue with 10 mL water, boil and centrifuge.

Proceed as given in step I

STEP I

In absence of Br^- and / or I^-

Mix. + water (10 mL) ⟶ boil and centrifuge.

Centrifugate (CF) is water solution (**WS**) used for test of NH_4^+ , K^+ and water soluble cations	**Residue**: (In case of some mixtures residue may not be obtained, in such

	cases after testing NH_4^+ & K^+, WS is itself used for further analysis) Residue + 5cm^3dil. HCL hear and centrifuge. centrifugate is acid extract (**AS**), if residue is obtained add few drops of conc. HCl to dissolve.

Test for NH_4+ 1. CF + NaOH (boil)gives smell of NH_3 turning turmeric paper Brown 2. CT : CF + Nesselers reagent gives Reddish brown ppt.	***Test for K^+*** **:** CF **(**If NH_4^+ is present, add few drops of NaOH, boil till all NH_3 is evolved) + sodium cobalt nitrite gives Yellow ppt.

STEP II

Mix water solution+ acid solution / water solution + dil. HCl	White ppt. (Chloride Group)	Pb^{+2} present then completely remove Pb^{+2} by adding dil. HCl to whole solution and centrifuge. ppt is analysed for Pb^{+2} as given in STEP III
*Test portion of CF (free from Pb^{+2}) + Sat. solution of Na_2SO_4 / $(NH_4)_2SO_4$	White ppt. (Sulphate Group)	Sulphate group radicals i.e. Ba^{+2} , Ca^{+2}, Sr^{+2} may present. Before further analysis, complete the analysis of sulphate group with CF as per **STEP IV**, completely remove sulphate radicals by the addition of Sat. solution of Na_2SO_4 / $(NH_4)_2SO_4$ to entire solution & centrifuge. Use sulphate ppt. for flame test of Sr^{+2} as ppt. + Conc. HCl gives Crimson Red flame.
Test portion of CF(free from Ba^{+2} , Ca^{+2}, Sr^{+2}) + $K_3[Fe(CN)_6]$	Blue colour	Fe^{+2}, Fe^{+3} present If present then oxidise as: whole CF + Conc. HNO_3, boil cool.
CF + solid NH_4Cl + 6N NH_4OH till strong smell of NH_3 stir well	i. Brown ppt.	Fe^{+3} present
	ii. Gelatous white ppt.	Al^{+3} present
	iii. White ppt.	Bi^{+3} present
@ Observe the colour of the ppt. & interpret it according and analyse as given in **STEP V (Hydroxyl Group)**	iv. Grayish green ppt.	Cr^{+3} present
	v. Buff ppt.	Mn^{+2} present
CF may contain following radicals Ni^{+2},Cu^{+2},Cd^{+2}, Zn^{+2}, Co^{+2}, Mg^{+2} (if CF deep blue in colour indicates presence of Ni^{+2},Cu^{+2} Refer STEP VI)		

STEP III:

White ppt of $PbCl_2$ + water (heat) ⟶ ppt. dissolves (Pb^{+2} solution)

I. Pb^{+2} solution + K_2CrO_4 ⟶ Yellow ppt. of PbCrO4

II. Pb^{+2} solution + KI $\longrightarrow$ Yellow ppt. of PbI

Spot test for Pb^{+2} : A drop of above solution + 2 drops of 3N NaOH + 1 drop od Sat. Br_2 water solution + 2 drops of 1:1 NH_3 + 2 drops of Benzidine reagent gives dark blue spot.

STEP IV:

Test for Ba^{+2} :

* CF + K_2CrO_4 $\longrightarrow$ Yellow ppt. of BaCrO4

If no ppt. is obtained then check for Ca^{+2}, Sr^{+2}

Spot test for Ba^{+2} : * CF + 2 drops of 2N NaOH + sodium Rhodizonate reagent gives Reddish brown spot.

Test for Ca^{+2} :

* CF + Ammonium oxalate $\longrightarrow$ White ppt. of calcium oxalate

Calcium oxalate + Conc. HCl gives Brick red flame.

STEP V

Analysis and group test for the hydroxyl group

Hydroxide ppt. + 5 mL NaOH boil well

Centrifugate (Al^{+3}, Cr^{+3})	**Residue (Mn^{+2}, Fe^{+3})**
If centrifugate is Yellow - Cr^{+3} present i.CF +1-2 drops of Conc. HNO_3 + ether + H_2O_2------ Blue ether layer **Spot test for** Cr^{+3}: CF + diphenyl carbazole reagent gives Violet spot **Test for Al^{+3}**: i. CF + solid NH_4Cl-------Gelatinous white ppt. **Spot test for Al^{+3}**: Dissolve above ppt. in dil. HCl + few drops of 10% ammonium acetate + few drops of ammonium carbonate + aluminon reagent gives bright red colour	Dissolve residue in two parts i. Test for Fe^{+3}: I st part dissolve in dil. HCl + NH_4CNS ------blood red colour **Spot test for Fe^{+3}:** Fe^{+3} solution + Ferroin reagent------- Green colour ii. **Test for Mn^{+2}**: II nd part dissolved in Conc. HNO_3 + solid PbO_2, boil, cool, and dilute it with H_2O----- Pink violet colour of permanganate ions

STEP VI

Analysis and tests for soluble complex cations (Cu^{+2}, Ni^{+2}, Cd^{+2}, Zn^{+2}, Co^{+2}, Mg^{+2}).

Perform the tests for individual radicals with centrifugate as follows:

Ions	**Test**	**Observation & Inference**
Copper : Cu^{+2}	CF+CH_3COOH + $K_4Fe(CN)_6$	Red brown ppt.
Spot test	2 drops of CF + 2 drops of CH_3COOH + 1 drop of Cupron reagent hold the paper in NH_3 vapours	Green colour confirms Cu^{+2}
Nickel : Ni^{+2}	CF + 6N NH_4OH + Dimethyl glyoxime	Scarlet red ppt.

Spot test	2 drops of CF +2 drops of rubenic acid hold the paper in NH_3 vapours	Bluish violet colour confirms Ni^{+2}
Cobalt : Co^{+2}	2 drops of CF+1 drop of CH_3COOH + 2 drops of 10% ammonium thiocynate + 3-4 drops of acetone	Blue acetone layer.
Spot test	4 drops of CF + 2 drops of CH_3 COOH +2 drops of 1% α-nitroso-β Napthol	Red spot confirms Co^{+2}
Zinc : Zn^{+2}	CF + CH_3COOH + $K_4[Fe(CN)_6]$	Greenish white ppt.
Spot test	2 drops of CF+ drops of dil. H_2SO4 + 1 drop $Co(NO_3)_2$ + 1 drop of ammonium-mercuri-thio-cynate	Blue spot confirms Zn^{+2}
Magnesium : Mg^{+2}	CF+Na_2HPO_4	Crystalline white ppt.
Spot test	i. 2 drops of CF + 1 drop of titan yellow solution + 1 drop NaOH	Bright red spot. Confirms Mg^{+2}
	ii. 2 drops of CF + 1 drops of Magneson reagent + 1 drop of 1N NaOH	

🔬 **Experiment 10:** Estimation of Copper(II) in a given sample by iodometric titration using standard Sodium thiosulphate solution

Aim: To determine the percentage of copper (II) present in a given sample by titration against a standard aqueous solution of sodium thiosulphate (iodometry titration)

Requirement: $K_2Cr_2O_7$ (solid) $Na_2S_2O_3$ (0.05 N approx), solid Na_2CO_3, CH_3COOH,

10% KI, starch

Theory: Iodometric titration involves estimation of free or liberated iodine with the help of standard solution of sodium thiosulphate. Estimation in which iodine is used as a standard solution is called iodimetry. In iodometric method, an excess of KI is added to slightly acidic solution and the equivalent amount of liberated iodine is titrated against standardized $Na_2S_2O_3$ solution.

When an excess of KI is added to a cupric salt solution, the cupric iodide (CuI_2) first formed decomposes to cuprous iodide (CuI_2) and I_2 The liberated iodine is equivalent to one gram atom of copper, which is titrated against standard $Na_2S_2O_3$ solution using starch as indicator

The solution should be slightly acidic which helps to suppress the hydrolysis of cupric salts. The free acid present is neutralized by adding a pinch of $Na_2S_2O_3$ powder till a slight turbidity (formation of Cu $(OH)_2$) is obtained which is dissolved by drop wise addition of 2N CH_3COOH solution.

Procedure:

It involves three steps:

A. Preparation of 0.05N $K_2Cr_2O_7$ solution

B. Standardization of sodium thiosulphate solution

C. Estimation of copper

Step A: Preparation of 0.05 N $K_2Cr_2O_7$ solution

1. Weigh accurately 0.245 g of pure crystals of $K_2Cr_2O_7$ on a watch glass.

2. Transfer the crystals in a clean 100 cm^3 beaker and rinse the watch glass with distilled water.

3. Stir with glass rod and dissolve the crystals.

4. Transfer this solution to 100 cm^3 standard volumetric flask, and dilute it with distilled water up to the mark (100 cm^3) gradually with vigorous shaking in between.

5. Take out all the solution in a clean beaker to get a homogeneous solution.

6. This becomes standard 0.05N $K_2Cr_2O_7$ solution. Use this solution for standardization of $Na_2S_2O_3$ solution.

Step B: Standardization of Sodium Thiosulphate Solution

1. Wash the burette with water, rinse it with $Na_2S_2O_3$ (Approx. 0.05N) and fill the burette with solution, taking precaution that nozzle of the burette is free from air bubble.

2. Pipette out 10cm^3 of 0.05 N $K_2Cr_2O_7$ solution in a conical flask. Add 5 cm^3 of conc. HCl to it.

3. Now add one test tube of 10% KI solution.

4. Shake the flask well and then titrate the liberated iodine with $Na_2S_2O_3$ solution till solution turns pale yellow.

5. Add 1 to 2cm^3 of freshly prepared starch indicator (solution turns blue) and continue the titration till blue colour of the solution changes to faint green colour (due to formation of 'Cr' salt solution)

6. Repeat the titrations in similar manner to get constant burette reading (let this reading be ‘A’ cm^3). From this reading calculate the exact normality of supplied $Na_2S_2O_3$ solution.

Step C: Estimation of copper

1. Dilute the given solution containing Cu^{2+} ions to 100cm^3 gradually using distilled water.

If solid sample is provided (W g) then transfer the entire quantity in 100cm^3 beaker. Add 15-20 cm^3 of distilled water. If solution is turbid add a few drops of conc. H_2So_4. Stir the solution. Transfer it quantitatively to 100cm^3 standard volumetric flask. Dilute to 100cm^3 gradually using distilled water. Shake the solution vigorously so that it becomes homogeneous.

2. Pipette out 10cm^3 of diluted Ca^{2+} solution in a conical flask.

3. Add solid N_2CO_3 pinch wise till the solution becomes turbid [due to precipitation of $Cu(OH)_2$]

4. Dissolve this turbidity by adding drop by drop 2N CH_3COOH. Shake the flask well and add 2 - 3 drops of CH_3COOH in excess. [Solution must be clear now]

5. To this clear solution add one test tube of 10% KI solution, solution turns yellow.

6. Titrate the liberated iodine with $Na_2S_2O_3$ solution till a pale yellow colour appears.

7. Add 1-2 cm^3 of freshly prepared starch indicator (solution turns dark blue) and continue the titration.

8. The end point of the titration is noted when colour of the solution changes from dark blue to colourless (or white ppt)

Repeat the experiment in similar way and note down the constant burette reading 'B' cm^3

Observation

Standardization of $Na_2S_2O_3$ solution

Principle of titration : Iodometry

Solution in burette : Sodium thiosulphate (approx 0.05)

Solution in the conical flask : 10 cm^3 of 0.05 N $K_2Cr_2O_7$ solution +

5 cm^3 of conc.HCl + 1 Test Tube 10% KI solution

Indicator used : Starch solution.

End point : Blue to green

Pilot reading between _____ and _____ cm^3

Observation Table

Burette level	Burette Reading in cm^3			CBR cm^3
	1	2	3	
Initial	00	00	00	A = cm^3
Final				
Difference				

Reactions

$K_2Cr_2O_7 + 8KI + 14HCl \longrightarrow 8KCl + 2CrCl_3 + 7H_2O + 3I_2$

$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$

Calculation of Normality

10 cm^3 of 0.05 N $K_2Cr_2O_7$ solution required (A) = _____ cm^3 of $Na_2S_2O_3$ solution

$Na_2S_2O_3 \longrightarrow K_2Cr_2O_7$

$N_1V_1 = N_2V_2$

$N_1 \times A = 0.05 \times 10$

$\therefore N_1 = \frac{0.05\ x\ 10}{A} = 'C'N$

Estimation of Copper

Observation

Principle of titration : Iodometry

Solution in Burette : $Na_2S_2O_3$ ('C' N)

Solution in the conical flask : 10 cm^3 of diluted solution + Solid Na_2CO_3 + CH_3COOH + 10% KI solution

Indicator used : Starch

End point : Blue to white (or colourless)

Pilot reading between : _____ and _____ cm^3

Observation Table

Burette level	Burette Reading in cm^3			CBR cm^3
	1	2	3	
Initial	00	00	00	A = cm^3
Final				
Difference				

Reactions:

i) $2CuSO_4 + 4KI \longrightarrow 2CuI_2 + K_2SO_4$

$2CuI_2 \longrightarrow Cu_2I_2 + I_2$

$2CuSO_4 + 4KI \longrightarrow Cu_2I_2 + K_2SO_4 + I_2\uparrow$

ii) $2NS_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$

Calculations:

10 cm^3 of diluted solution required = B = _____ cm^3 of 'C' N $Na_2S_2O_3$ solution

$\therefore$ 100 cm^3 of diluted Cu^{2+} solution required = 10 x B

i.e. 10x _____cm^3 of 'C' N $Na_2S_2O_3$ solution

$2Na_2S_2O_3 = 2I = 2Cu = 2\ CuSO_45H_2O$

$\therefore Na_2S_2O_3 = I = Cu = CuSO_45H_2O$

1000 cm^3 of 1 N $Na_2S_2O_3$ solution = 63.54 g of copper = 249.54 g of $CuSO_45H_2O$

∴ 10 x B cm^3 of 'C' N $Na_2S_2O_3$ solution

$$= \frac{63.54\ x\ 10\ x\ B\ x\ C}{1000\ x\ 1} =' D' g\ of\ copper$$

$$= \frac{249.54\ x\ 10\ x\ B\ x\ C}{1000\ x\ 1} =' E'\ g\ of\ copper$$

If sample is solid weighing 'W' g then

$$\%\ copper = \frac{D}{W}\ x\ 100$$

$$= F$$

Result:

1.	Normality of supplied $Na_2S_2O_3$ solution	'C' N =
2.	Volume of $Na_2S_2O_3$ solution required by 10 cm^3 of diluted Cu^{2+} solution	'B' cm^3 =
3.	Amount of copper in 100 cm^3 of diluted solution	'D' g = _____g
4.	Amount of $CuSO_4.5H_2O$ present in the given solution	'E' g = _____ g
5.	Percentage of copper in the sample	F% = _____ %

Pre-lab Questions

1. What is the principle of iodometric titration?
2. Why is KI added in excess during the titration?
3. Why is starch indicator not added at the beginning of the titration?
4. Write the balanced chemical equations involved in the titration.

Post-lab Questions

1. Why does the blue colour disappear at the end point?
2. What would happen if starch was added too early in the titration?
3. Why is the titration mixture kept away from sunlight?
4. Can iodometric titration be used to estimate other metal ions? Give examples.

Group C

🔬 **Experiment 11-16:** Characterization of organic compounds containing C,H,(O) N,S,X elements

Procedure:

A Systematic Scheme for the identification of the organic compound is outlined below

F. Preliminary Test

G. Solubility Test

H. Detection of Elements

I. Detection of Functional Group

J. Determination of physical constant and Identification of the compound.

C) Preliminary Test

Test	Observation	Inference
i) Colour	i) Yellow	Nitrobenzene, m-Dinitrobenzene, p- Nitro toluene,nitro phenol, nitro aniline etc. may be present
	iii) Brown	P – Toluidine, resorcinol etc. may be present
	iv) Buff or pinkish	ß – Naphthol may be present
	v) Blackish	α – Naphthol may be present
	vi) Reddish	Aniline, phenol, Aromatic amine etc. may be present
	vii) Colourless	Simple acid, alcohol, ester, ketone,aromatic hydrocarbon etc. may be present
ii) Odour	i) Carbolic	Phenol, cresol etc. may be present
	ii) Fishy	Amine may be present

	iii) Sweet and pleasant	Ester, alcohol and halogen derivatives etc. may be present
	iv) Bitter almonds	Nitrobenzene, Benzaldehyde etc. may be present
	v) Moth balls (Strong and characteristic)	Naphthalene may be present
	vi) No particular smell	Aromatic acid, amide, carbohydrate etc. may be present
iii) State	Solid / Liquid	-----
iv) Unsaturation test		
$KMnO_4$ test Substance + 2 ml of water shake well + 2 drops of dilute $KMnO_4$ solution.	**i)** Decolourisation of $KMnO_4$ **ii)** No decolourisation	Unsaturated or easily oxidizablecompound may be present. Saturated compound may be present.
Bromine Test **For water insoluble subs.** Substance + chloroform ... shake and add Bromine in CCl_4 **For water soluble subs.** Substance + 2 ml water shake + Bromine water	**i)** Decolourisation **ii)** Decolourisation with formation of precipitate. **iii)** No decolourisation.	Unsaturated compound may be present. Easily substituted compound may be present. Saturated compound may be present
v) Copper foil test Heat copper foil till red hot. Put small quantity of subs. on copper foil and observe the flame.	**i)** Sooty flame	Aromatic compound may be present
	ii) Non sooty flame	Aliphatic compound may be present
	iii) Initially sooty finally bluish green	Aliphatic or aromatic halogenated compound may be present.

D) Solubility test

Determination of Chemical nature of the substance

Perform the following tests If substance is insoluble / immiscible in water.		
Test	Observation	Inference
i) substance + water ...shake well**.**	Insoluble	Water insoluble compound present

ii) Subs. + sat. $NaHCO_3$	Soluble with strong effervescence	Carboxylic acid present
(If sub. Dissolves and clear solution is obtained) To this clear solution add conc. HCl drop by drop.	A solid appear	Carboxylic acid present
iii) Subs. + NaOH	Soluble	Phenolic compound present
(If sub. dissolves, clear solution is obtained) To this clear solution add conc. HCl drop by drop.	A solid appear	Phenolic compound present
iv) Subs + HCl	Soluble	Base present
(If sub. dissolves, clear solution is obtained) To this clear solution add conc. NaOH drop by drop.	A solid or emulsion appear	Base present
v) Substance is insoluble in all (water, $NaHCO_3$, NaOH, HCl)	----	Water insoluble neutral compound present

[For solid substance use word soluble or insoluble, for liquid substance use word miscible or immiscible]

Conclusion: Given substance is water insoluble / immiscible ----------

Perform the following tests If substance is soluble / miscible in water.		
Test	Observation	Inference
i) substance + water …shake well. Use this solution for further analysis	soluble (subs. dissolves)	Water soluble compound present. (Lower member of alcohol, ester ketone, carbohydrate)
Test solution with litmus paper.	Blue litmus paper turns red Red litmus paper turns blue No action on the litmus paper	Water soluble acid or phenol present. Water soluble base present Water soluble neutral present
ii) Solution + sat. $NaHCO_3$	Effervescences No effervescences	Water soluble acid present Water soluble acid absent
iii) Solution + alcoholic $FeCl_3$	Blue to violet colour No characteristic coloration	Water soluble phenol present Water soluble phenol absent
iv) Effect on red litmus paper	Red litmus turns blue No effect on red litmus	Water soluble base present Water soluble base absent

Conclusion: Given substance is water soluble ----------

E) Detection of Elements

Sodium Fusion Test (Lassaigne's Test)

1.Take a small piece of dry sodium metal in a fusion tube and heat it gently till the metal melts or fuses.

2. Add equal quantity of compound to this fused metal [If he compound is a liquid then add two drops of it with a capillary]

3. Heat it gently then strongly till it becomes red hot.

4.Plung the red hot tube in 10 ml or ¾ of a test tube of distilled water taken in a porcelain dish, covering it immediately with an asbestos sheet. Crush the fusion tube completely.

5. Boil the extract for five minutes reduce the volume to about 5 ml and filter. Perform following test using this filtrate

Test	Observation	Inference
Test for Nitrogen		
i) i) 2 ml of filtrate + freshly prepared $FeSO_4$ solution, boil for a few minutes, cool and acidify it by adding dil. HCl or dil. H_2SO_4	Blue or green colour solution	Nitrogen present.
Test for sulphur		
ii) 1 ml of filtrate + 1 drop of sodium nitroprusside solution.	A violet or purple coloration.	Sulphur present.
Test for Halogen		
(If copper foil test is initially sooty finally bluish green then perform this test) iii) 2 ml of filtrate + dilute HNO_3 (boil well if N and S are present) + 2 drops of 5% $AgNO_3$ solution.	A thick white precipitate.	Halogen present.
Distinction between Halogens: If halogen is present carry out the following test:		
2 ml of filtrate + 1ml of dilute H_2SO_4 + 0.5 ml of $CHCl_3$ and 0.5ml of chlorine water,	i) Colourless layer ii) Yellow or brown colour.	Chlorine present. Bromine present.

shake well and observe the colour of chloroform layer.	(i) Violet colour	Iodine present.

Conclusion: Given compound contains elements C, H, (O), ----------

Classification of the given compound on the basis of elements present

Group I: C, H, (O)	**Group III: C, H, (O), Halogen**
Acid	Acid
Phenol	Phenol
Neutral	Neutral
Group II: C, H, (O), N	**Group IV: C, H, (O), N, S**
Acid	Acid
Phenol	Neutral
Base	
Neutral	

Check the chemical nature of compound (conclusion of solubility test) and elements present. Find out the group of given compound and accordingly perform functional group test

F) Functional group test

Group I: C, H, (O) Carboxylic acids		
Test	**Observation**	**Inferences**
a) substance + sat. $NaHCO_3$ solution. Shake well.	Soluble with strong effervescences	Carboxylic acid present. (-COOH group)
To this clear solution add conc. HCl dropby drop.	A solid appear	Water insoluble Carboxylic acid confirmed.
	No solid reappear	Water soluble Carboxylic acid confirmed. (-COOH group)
b) Subs.+ 1ml of water, shake well + 1-2 drops of alcoholic $FeCl_3$ solution.	Buff coloured precipitate	Benzoic acid or phthalic acid.
	Violet colored precipitate	Salicylic acid
	Yellow colored precipitate	Cinnamic acid
	Faint reddish colored precipitate	Succinic acid
	Deep yellow colored solution	Citric acid
	No change in $FeCl_3$ solution	Oxalic acid

Group I: C, H, (O) Phenols		
a) substance + dilute NaOHsolution. Shake well. To this clear solution add conc. HCl dropby drop	Subs dissolves completely A solid or emulsion appear	Phenol present Phenol confirmed (-OH group present)
b) Subs.+ 1ml of water,shake well + 1-2 drops of alcoholic $FeCl_3$ solution.	Violet Color	Phenol Present
	Blue Violet Color	Resorcinol
	White ppt slowly changing to pink, blue or violet	α - napthol
	Green Color	ß - napthol
Phthalein test		
c) 0.01gm of compound + 0.01gm of phthalic anhydride + 2 drops of conc. H_2SO_4.Heat gently until the mixture fuses. Cool and pourit in a beaker containing 20 ml ogf very dilute NaOH solution	Pink colour Green or bluish green Yellowish-green flouresence	Phenol Present α – napthol/ß – napthol Resorcinol

Group I : C, H, (O) Neutrals		
Test	**Observation**	**Inferences**
Test for Carbohydrate		
Solid Subs.	Soluble in water but insoluble in ether	Carbohydrate present
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-napthol dissolved in ethyl alcohol, add carefully 1 ml of conc.H_2SO_4 along the sides of the test tube.	A Violet ring appears at the junction of two layers.	Carbohydrate confirmed
Test for Aldehyde		
Tollen's Test OR Silver Mirror test:	A silver mirror is formed on the inner sides of the test tube	Aldehyde present

0.1 gm of the compd + 2-3ml Tollents reagent … Heat it on a boiling water bath.		
Test for Ketone		
a) For solid substance 0.05 gm of the compound + 3 ml of 2,4 dinitrophenyl hydrazine. Shake well.	Yellow or orange red crystalline precipitate	Ketone present
b) For liq. Subs. 0.1gm of compd + 2ml of sodium nitroprusside solution + 2 drops of NaOH	Red coloration	Ketone present
Test for ester		
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	Pink colour disappears	Ester present (-COOR)
Test for alcohol		
Take a small piece of dry Na metal in a fusion tube and add a few drops of compound.	Rapid evolution of H_2 (Strong effervescence)	Alcohol present (R-OH)
Test for ether		
0.5ml of compound + 1 ml of iodine in carbon disulphide, shake well	Purple colour of CS_2 layer changes to brown colour	Ether present (R-O-R)
Test for hydrocarbon	If all the above tests are negative	Hydrocarbon present
Group II: C, H, (O), N Carboxylic acids		
Test	**Observation**	**Inferences**
a) substance + sat. $NaHCO_3$ solution. Shake well. To this clear solution add conc. HCl dropby drop.	Soluble with strong effervescences A solid appear No solid reappear	Carboxylic acid present. (-COOH group) Nitro Carboxylic acid present Amino Carboxylic acid present
i) If nitro carboxylic acid is present, perform the following test of nitro group – **Test for nitro group** – 0.2 gm of the compd + 2 ml of ethyl alcohol + 0.1	Black or grey precipitate	Nitro group present

gm of solid NH_4Cl + 0.1 gm of Zn dust. Boil for 5min and filter. Filtrate + Tollen's Reagent		
ii) If amino carboxylic acid present, perform the following test for amino gr- 0.5 gm of the compd +3 / 4 ml of 1:1 HCl, shake well. Cool and add few drops of 2% $NaNO_2$ solution. Add this clear solution to ß-napthol in NaOH	orange red dye	Amino group present
Group II: C, H, (O), N Phenols		
a) substance + dilute NaOHsolution. Shake well. To this clear solution add conc. HCl dropby drop	Compd dissolves producing deep yellow or orange colour A solid appears No solid reappears	Nitro phenol or Amino phenol present Nitro phenol present Amino phenol present
b) If nitrophenol present, perform the test for NO_2 group **Test for nitro group** – 0.2 gm of the compd + 2 ml of ethyl alcohol + 0.1 gm of solid NH_4Cl + 0.1 gm of Zn dust. Boil for 5min and filter. Filtrate + Tollen's Reagent	Black or grey precipitate	Nitro group present
ii) If amino phenol present, perform the following test for amino gr- 0.5 gm of the compd +3 / 4 ml of 1:1 HCl, shake well. Cool and add few drops of 2% $NaNO_2$ solution. Add this clear solution to ß-napthol in NaOH	orange red dye	Amino group present
Group II: C, H, (O), N Base		
0.5 gm of the compd + 5	Compd dissolves	Base present

ml of HCl, shake and filter. Filtrate + NaOH	 A solid reappears or emulsion obtained	 Base confirmed
0.5 gm of the compd + 5 ml of 1:1 HCl, shake well. Cool and add few drops of 2% $NaNO_2$ solution	Red coloration Yellow oil or ppt A clear solution is obtained which when added to a cold solution of ß- napthol in NaOH give orange red dye	Aromatic tertiary amine present (-N-) Aromatic secondary amine present (Ar-N-) Aromatic primary amine present (Ar-NH_2)
Group II: C, H, (O), N Neutral		
Test for Amides (perform this test if compound is colorless and odorless)		
0.2 gm of compound + 3ml of 20% NaOH ....Boil	Evolution of ammonia turning turmeric paper brown	Amide present
Test for Anilides (perform this test if compound is colourless and odourless)		
0.1gm of the compd + 3 ml of conc HCl. Boil for 2 min, cool and add 5 ml of water + a few drops of cold $NaNO_2$ sol. And mix well. Add this solution to a cold solution ofalkaline ß-napthol.	Orange red dye	Anilide Group present
Test for Nitro group		
<u>Mulliken's Test</u> 0.2 gm of the compd + 2 ml of ethyl alcohol + 0.1 gm of solid NH_4Cl + 0.1 gm of Zn dust. Boil for 5min and filter.	 Black or grey precipitate	 Nitro group present

Filtrate + Tollen's Reagent		
Test for Dinitro compound 0.5 g of the compound + 1-2 ml of acetone,shake well to dissolve the com pound+ 1-2drops of dilute NaOH solution.	Dark purple or Violet colour	Dinitro compound present

Group III: C, H, (O), X (Halogen)		
0.2 gm of compd + 2-3 of ml of dilute NaOHsolution Boil for a few min, cool + dilute HNO_3 till acidic + 1ml of $AgNO_3$ solution.	White or yellow ppt No Precipitate.	Aliphatic halide like $CHCl_3$, CCl_4 present Chlorobenzene or bromobenzene present

Group IV: C, H, (O), N, S - Acids		
This class includes amino sulphonic acid		
Test for Amino Sulphonic Acid a)0.2 gm of the compound + 3 -4 ml of saturated $NaHCO_3$ solution.	Effervescences and compound dissolves	Amino Sulphonic acid present
b) Perform test for $-NH_2$ group as above.	Orange red dye is formed	Aromatic primary amino group present
Group IV: C, H, (O), N, S - Neutral		
Test for Thiourea (Thiourea is soluble in water and neutral to litmus paper) 0.1 gm of comp + 2ml oof 20% NaOH solution, boil, cool and add a few drops oflead acetate solution.	Black ppt is obtained	Thiourea is confirmed.

E. Determination of physical constant

Test	Observation	Inference
Take organic compound into one end sealed capillary and determine m.p, For liquid substance, Take	Record M.P / B. P of the substance	From MP/BP, identify the given compound/ substance

organic compound into fusion tube insert sealed capillary into fusion tube from open end		

Result:

State:
Chemical nature:
Elements present:
Functional Group:
Physical Constant:
Probable Compound: