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B.Sc. 1st YEAR CHEMISTRY

PRACTICAL FILE

(25 Practicals — Inorganic, Organic and Physical Chemistry, with Viva-Voce Questions and Answers)

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EXPERIMENT - 1

Aim

To estimate the strength of given oxalic acid solution by titrating it with $KMnO_4$ solution (approx. 0.05 N).

Apparatus Required

Burette, pipette, conical flask, burner, test tube, weighing balance, funnel, burette stand, measuring cylinder, measuring flask, watch glass.

Chemicals Required

Oxalic acid, $KMnO_4$, dilute sulphuric acid, distilled water.

Theory

1. This is a volumetric (redox titration) experiment in which the volume of standard $KMnO_4$ solution required to oxidise a known volume of oxalic acid solution is measured.
2. In acidic medium, $KMnO_4$ acts as a strong oxidising agent and oxalic acid acts as a reducing agent.
3. Oxidation and reduction take place simultaneously: Mn^{7+} is reduced to Mn^{2+} while $C_2O_4^{2-}$ is oxidised to CO_2.
4. $KMnO_4$ behaves as a good oxidising agent only in acidic medium, hence dilute H_2SO_4 is added before titration.
5. The oxalic acid solution is warmed to about 60 °C to speed up the initially slow reaction.
6. Mn^{2+} ions formed during the reaction act as an auto-catalyst, accelerating the subsequent reaction.
7. $KMnO_4$ acts as a self-indicator — the appearance of a permanent faint pink colour marks the end point.
8. Ionic equation: $2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

Procedure

I. Preparation of standard oxalic acid solution (0.05 N)

1. Weigh accurately 0.315 g of oxalic acid required for preparing 100 mL of 0.05 N solution.
2. Transfer it to a 100 mL standard volumetric flask through a funnel.
3. Add distilled water to dissolve and make up the volume to the mark.

II. Standardisation of given $KMnO_4$ solution

1. Pipette out 10 mL of standard oxalic acid solution into a conical flask and add 10 mL dilute H_2SO_4.
2. Warm the content to about 60 °C.
3. Titrate with the $KMnO_4$ solution taken in the burette until a permanent faint pink colour appears.
4. Repeat the titration to get three concordant readings.

III. Titration of given oxalic acid with the $KMnO_4$ solution

1. Pipette out 10 mL of the given oxalic acid solution into a conical flask and add 10 mL dilute H_2SO_4.
2. Warm the solution and titrate with the $KMnO_4$ solution taken in the burette until the colour turns permanent faint pink.
3. Repeat the titration to get three concordant readings.

Observation Tables

Observation Table 1: Standardisation of $KMnO_4$ against standard oxalic acid

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Sl. No.	Volume of standard oxalic acid (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Observation Table 2: Titration of given oxalic acid against $KMnO_4$

Sl. No.	Volume of given oxalic acid (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Calculation

Preparation of standard solution:

N = (wt. of oxalic acid / eq. wt. of oxalic acid) x (1000 / volume)

0.05 = (wt. of oxalic acid / 63) x (1000 / 100)

wt. of oxalic acid = (0.05 x 63 x 100) / 1000 = 0.315 g

N_1V_1 (oxalic acid) = N_2V_2 ($KMnO_4$)

0.05 x 10 = N($KMnO_4$) x V($KMnO_4$) [V($KMnO_4$) = mean burette reading from Table 1]

N($KMnO_4$) = (0.05 x 10) / V($KMnO_4$)

Now, for the given oxalic acid:

N_1V_1 (oxalic acid) = N_2V_2 ($KMnO_4$)

N(oxalic acid) x 10 = N($KMnO_4$) x V($KMnO_4$) [from Table 2]

N(oxalic acid) = [N($KMnO_4$) x V($KMnO_4$)] / 10

Strength of oxalic acid = N(oxalic acid) x Equivalent weight of oxalic acid

= N(oxalic acid) x 63 g/L

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What type of titration is this?

Ans. It is a redox (oxidation-reduction) titration, specifically a permanganometric titration.

Q2. Why is dilute H_2SO_4 added and not dilute HCl or HNO_3?

Ans. HCl can be oxidised by $KMnO_4$ to liberate Cl_2 (causing error), and HNO_3 is itself an oxidising agent that can interfere. Dilute H_2SO_4 provides H^+ ions without being oxidised or interfering.

Q3. Why is the oxalic acid solution warmed before titration?

Ans. The reaction between $KMnO_4$ and oxalic acid is slow at room temperature; warming to about 60 °C increases the rate of reaction.

Q4. What is meant by 'self-indicator'?

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Ans. $KMnO_4$ is intensely coloured (purple); a single drop in excess imparts a permanent pink colour to the solution, so no external indicator is needed.

Q5. What is autocatalysis, and which ion acts as the autocatalyst here?

Ans. Autocatalysis is when a product of the reaction catalyses the reaction itself. Here, Mn^{2+} ions formed during the reaction catalyse further reaction between $KMnO_4$ and oxalic acid.

Q6. What is the equivalent weight of oxalic acid ($H_2C_2O_4.2H_2O$)?

Ans. 63 (Molecular weight 126 divided by n-factor 2, since each oxalate ion loses 2 electrons).

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EXPERIMENT - 2

Aim

To estimate the strength of given Mohr's salt solution by titrating it with standardised $KMnO_4$ solution (approx. 0.05 N).

Apparatus Required

Burette, pipette, conical flask, burette stand, funnel, weighing balance, measuring flask, watch glass.

Chemicals Required

Mohr's salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$], $KMnO_4$, dilute H_2SO_4, distilled water.

Theory

1. This is a redox titration based on oxidation of Fe^{2+} to Fe^{3+} by $KMnO_4$ in acidic medium.
2. $KMnO_4$ is reduced from Mn^{7+} to Mn^{2+}, while Fe^{2+} present in Mohr's salt is oxidised to Fe^{3+}.
3. Dilute H_2SO_4 is added to maintain acidic medium since $KMnO_4$ acts as a strong oxidiser only under acidic conditions.
4. Unlike oxalic acid, this titration is carried out at room temperature (no heating required) and does not need an external indicator.
5. $KMnO_4$ acts as a self-indicator; the end point is the appearance of a permanent light pink colour.
6. Ionic equation: $MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

Procedure

I. Preparation of standard Mohr's salt solution (0.05 N)

1. Weigh accurately 1.96 g of Mohr's salt required for preparing 100 mL of 0.05 N solution.
2. Transfer to a 100 mL standard volumetric flask.
3. Add a few mL of dilute H_2SO_4 to prevent hydrolysis, then make up the volume with distilled water.

II. Titration of Mohr's salt with $KMnO_4$

1. Pipette out 10 mL of the given Mohr's salt solution into a conical flask.
2. Add about 10 mL of dilute H_2SO_4.
3. Titrate with $KMnO_4$ solution taken in the burette until a permanent light pink colour appears.
4. Repeat the titration to get three concordant readings.

Observation Table

Observation Table: Titration of Mohr's salt against $KMnO_4$

Sl. No.	Volume of Mohr's salt solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

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Calculation

Preparation of standard solution:

N = (wt. of Mohr's salt / eq. wt. of Mohr's salt) x (1000 / volume)

0.05 = (wt. of Mohr's salt / 392) x (1000 / 100)

wt. of Mohr's salt = (0.05 x 392 x 100) / 1000 = 1.96 g

N_1V_1 ($KMnO_4$) = N_2V_2 (Mohr's salt)

N($KMnO_4$) x V($KMnO_4$) = N(Mohr's salt) x 10

N(Mohr's salt) = [N($KMnO_4$) x V($KMnO_4$)] / 10

Strength of Mohr's salt = N(Mohr's salt) x Equivalent weight of Mohr's salt

= N(Mohr's salt) x 392 g/L

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. Why is the Mohr's salt titration done at room temperature, unlike oxalic acid?

Ans. Fe^{2+} is readily and rapidly oxidised by $KMnO_4$ even at room temperature, so heating is not required (and heating may cause aerial oxidation of Fe^{2+}).

Q2. Why is dilute H_2SO_4 added to the Mohr's salt solution?

Ans. To prevent hydrolysis of the Fe^{2+} salt and to provide the acidic medium needed for $KMnO_4$ to act as an oxidising agent.

Q3. What is the colour change at the end point?

Ans. Colourless/pale green to a permanent light pink, due to a slight excess of $KMnO_4$.

Q4. What is the formula and equivalent weight of Mohr's salt?

Ans. $FeSO_4.(NH_4)_2SO_4.6H_2O$; its equivalent weight equals its molecular weight, 392, since the n-factor is 1 (one electron lost per Fe^{2+}).

Q5. Why can't Mohr's salt solution be kept for long before titration?

Ans. Fe^{2+} is slowly oxidised to Fe^{3+} by atmospheric oxygen, which changes the strength of the solution.

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EXPERIMENT - 3

Aim

To estimate the strength of given $CuSO_4$ solution iodometrically by titrating it against standard sodium thiosulphate ($Na_2S_2O_3$) solution.

Apparatus Required

Burette, pipette, conical flask, burette stand, measuring cylinder, weighing balance, glass stoppered flask.

Chemicals Required

$CuSO_4$ solution, potassium iodide (KI), sodium thiosulphate ($Na_2S_2O_3$), starch indicator, glacial acetic acid/dilute acid, distilled water.

Theory

1. This is an indirect redox (iodometric) titration. Cu^{2+} oxidises iodide ion (I^-) to iodine (I_2), while Cu^{2+} itself is reduced to Cu^+ (as CuI precipitate).

2. The iodine liberated is then titrated against standard sodium thiosulphate solution.

3. Reaction: $2Cu^{2+} + 4I^- \rightarrow 2CuI$ (white ppt) + I_2

4. Iodine reacts with thiosulphate: $I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

5. Starch solution is used as an indicator near the end point; it forms a deep blue complex with iodine, which disappears sharply (changes to milky white, due to CuI) when all the iodine is consumed.

6. Starch is added only near the end point (when the solution becomes pale yellow) to avoid formation of an insoluble starch-iodine complex.

Procedure

I. Preparation of standard $Na_2S_2O_3$ solution

1. Weigh accurately the required amount of $Na_2S_2O_3.5H_2O$ for 100 mL of approx. 0.05 N solution.

2. Dissolve in freshly boiled and cooled distilled water and make up the volume in a 100 mL volumetric flask.

II. Titration of $CuSO_4$ solution

1. Pipette out 10 mL of the given $CuSO_4$ solution into a conical flask.

2. Add about 1 g of solid KI (or 10 mL of 10% KI solution); a brown colouration due to liberated iodine appears.

3. Titrate immediately with $Na_2S_2O_3$ solution from the burette until the solution turns pale yellow.

4. Add 1-2 mL of freshly prepared starch solution; the solution turns blue.

5. Continue the titration carefully until the blue colour disappears and a milky white (CuI) colour persists.

6. Repeat the titration to get three concordant readings.

Observation Table

Observation Table: Titration of $CuSO_4$ solution against $Na_2S_2O_3$

Sl. No.	Volume of $CuSO_4$ solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

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Calculation

N_1V_1 ($CuSO_4$) = N_2V_2 ($Na_2S_2O_3$)

$N(CuSO_4)$ x 10 = $N(Na_2S_2O_3)$ x $V(Na_2S_2O_3)$

$N(CuSO_4)$ = [$N(Na_2S_2O_3)$ x $V(Na_2S_2O_3)$] / 10

Strength of $CuSO_4$ = $N(CuSO_4)$ x Equivalent weight of $CuSO_4$

= $N(CuSO_4)$ x 159.5/2 (Eq. wt. of $CuSO_4.5H_2O$ = 249.5/2 if hydrated)

Strength (g/L) = _____

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. Is this a direct or indirect titration? Why?

Ans. It is an indirect (iodometric) titration — Cu^{2+} is not titrated directly; instead it liberates an equivalent amount of iodine from KI, which is then titrated against $Na_2S_2O_3$.

Q2. Why is starch added only near the end point and not at the beginning?

Ans. Starch forms an insoluble starch-iodine complex at high iodine concentration, which does not dissociate easily and gives an indistinct/delayed end point. Adding it near the end point (pale yellow stage) avoids this.

Q3. What is the colour change at the end point?

Ans. Blue (starch-iodine complex) to colourless/milky white (due to the white CuI precipitate).

Q4. Why does a white precipitate of CuI form?

Ans. Cu^{2+} oxidises I^- to I_2 while itself being reduced to Cu^+, which combines with I^- to form insoluble white CuI.

Q5. Why is excess KI added?

Ans. Excess KI ensures complete reaction with Cu^{2+}, helps keep the liberated iodine in solution as a soluble I_3^- complex, and drives the reaction to completion.

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EXPERIMENT - 4

Aim

To estimate the amount of Fe^{2+} present in the given solution by titrating it with standard $K_2Cr_2O_7$ solution using diphenylamine as internal indicator.

Apparatus Required

Burette, pipette, conical flask, burette stand, measuring flask, measuring cylinder.

Chemicals Required

Mohr's salt/Fe^{2+} solution, standard $K_2Cr_2O_7$ solution, dilute H_2SO_4, dilute H_3PO_4, diphenylamine indicator, distilled water.

Theory

1. This is a redox (dichrometric) titration in which Fe^{2+} is oxidised to Fe^{3+} by $K_2Cr_2O_7$ in acidic medium.

2. $K_2Cr_2O_7$ is reduced from Cr^{6+} to Cr^{3+}.

3. Ionic equation: $Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$

4. Unlike $KMnO_4$, $K_2Cr_2O_7$ is not a self-indicator (orange-green colour change is not sharp), so an internal redox indicator, diphenylamine, is used.

5. Phosphoric acid (H_3PO_4) is added to lower the redox potential of the Fe^{3+}/Fe^{2+} couple, which sharpens the colour change and prevents interference from the yellow colour of Fe^{3+} ions.

6. At the end point, diphenylamine gets oxidised and the solution changes from green to an intense blue-violet colour.

Procedure

I. Preparation of standard $K_2Cr_2O_7$ solution

1. Weigh accurately the calculated amount of pure $K_2Cr_2O_7$ (primary standard) for 100 mL of 0.05 N solution.

2. Dissolve in distilled water in a 100 mL volumetric flask and make up to the mark.

II. Titration of Fe^{2+} solution

1. Pipette out 10 mL of the given Fe^{2+} solution into a conical flask.

2. Add about 5 mL of dilute H_2SO_4 and 5 mL of dilute H_3PO_4.

3. Add 2-3 drops of diphenylamine indicator; the solution turns green.

4. Titrate with the $K_2Cr_2O_7$ solution taken in the burette until the colour changes sharply from green to blue-violet.

5. Repeat the titration to get three concordant readings.

Observation Table

Observation Table: Titration of Fe^{2+} solution against $K_2Cr_2O_7$

Sl. No.	Volume of Fe^{2+} solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

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Calculation

N_1V_1 (Fe^{2+}) = N_2V_2 ($K_2Cr_2O_7$)

$N(Fe^{2+}) \times 10 = N(K_2Cr_2O_7) \times V(K_2Cr_2O_7)$

$N(Fe^{2+}) = [N(K_2Cr_2O_7) \times V(K_2Cr_2O_7)] / 10$

Amount of Fe^{2+} per litre = $N(Fe^{2+})$ x Equivalent weight of Fe (55.85)

= _____ g/L

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. Why is $K_2Cr_2O_7$ preferred as a primary standard over $KMnO_4$?

Ans. $K_2Cr_2O_7$ is available in a highly pure, stable form, is not hygroscopic, does not decompose on keeping, and its solutions do not deteriorate, making it an ideal primary standard.

Q2. Why can't $K_2Cr_2O_7$ be used as a self-indicator?

Ans. The colour change from orange (Cr_2O_7 2-) to green (Cr^{3+}) is not sharp enough to detect the exact end point, so an external/internal indicator is required.

Q3. Why is diphenylamine used as the indicator here?

Ans. Diphenylamine is a redox indicator that gets oxidised at the end point, producing an intense blue-violet colour, giving a sharp end point.

Q4. What is the role of phosphoric acid (H_3PO_4) in this titration?

Ans. H_3PO_4 forms a colourless complex with Fe^{3+} ions, which prevents the yellow colour of Fe^{3+} from masking the indicator colour change and also lowers the redox potential of the Fe^{3+}/Fe^{2+} system, sharpening the end point.

Q5. What is the colour change observed at the end point?

Ans. Green to a sharp blue-violet (bluish-purple) colour.

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EXPERIMENT - 5

Aim

To determine the number of molecules of water of crystallisation present in a given sample of Mohr's salt [$FeSO_4.(NH_4)_2SO_4.xH_2O$] by titrating it against standardised $KMnO_4$ solution.

Apparatus Required

Burette, pipette, conical flask, burette stand, weighing balance, measuring flask, watch glass.

Chemicals Required

Given Mohr's salt sample, standardised $KMnO_4$ solution, dilute H_2SO_4, distilled water.

Theory

1. Mohr's salt has the general formula $FeSO_4.(NH_4)_2SO_4.xH_2O$, where x is the number of water molecules of crystallisation (normally 6).

2. A known weight of the given Mohr's salt is dissolved to prepare a solution of known volume, and the Fe^{2+} content is estimated by titrating with standardised $KMnO_4$ solution.

3. From the titre value, the actual equivalent weight (and hence molecular weight) of the given sample is calculated, from which the number of water molecules (x) is found.

4. Ionic equation: $MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

Procedure

I. Preparation of Mohr's salt solution

1. Weigh accurately a known mass (say W g) of the given Mohr's salt sample.

2. Dissolve it in a little dilute H_2SO_4 and distilled water, and make up to 100 mL in a standard volumetric flask.

II. Titration against standardised $KMnO_4$

1. Pipette out 10 mL of the above Mohr's salt solution into a conical flask.

2. Add about 10 mL of dilute H_2SO_4.

3. Titrate with the standardised $KMnO_4$ solution taken in the burette until a permanent light pink colour appears.

4. Repeat to get three concordant readings.

Observation Table

Observation Table: Titration of Mohr's salt solution against standardised $KMnO_4$

Sl. No.	Volume of Mohr's salt solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Calculation

Let weight of Mohr's salt taken = W g (dissolved in 100 mL)

N_1V_1 (Mohr's salt) = N_2V_2 ($KMnO_4$)

N(Mohr's salt) x 10 = N($KMnO_4$) x V($KMnO_4$)

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$N(\text{Mohr's salt}) = [N(KMnO_4) \times V(KMnO_4)] / 10$

Equivalent weight of Mohr's salt (experimental) = (W x 1000) / (N(Mohr's salt) x 100)

Molecular weight of Mohr's salt = Equivalent weight x 1 (n-factor = 1)

Theoretical formula weight (anhydrous part, $FeSO_4.(NH_4)_2SO_4$) = 284

Weight of water of crystallisation = Experimental mol. wt. - 284

Number of water molecules, x = (Experimental mol. wt. - 284) / 18

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is 'water of crystallisation'?

Ans. It is the fixed number of water molecules chemically bound within the crystal lattice of a salt, e.g., 6 molecules of water in Mohr's salt.

Q2. How is the number of water molecules calculated in this experiment?

Ans. By determining the experimental equivalent/molecular weight of the salt from the titration, then subtracting the anhydrous formula weight (284) and dividing the difference by 18 (molecular weight of water).

Q3. Why must the weighed Mohr's salt be dissolved immediately in acidified water?

Ans. To prevent aerial oxidation of Fe^{2+} to Fe^{3+} and to prevent hydrolysis, both of which would give an inaccurate titre value.

Q4. What is the theoretical number of water molecules in Mohr's salt?

Ans. Six (6) molecules of water of crystallisation.

Q5. Why is this method called an indirect method of determining water of crystallisation?

Ans. Because the amount of water is not measured directly; it is calculated indirectly from the experimentally determined molecular weight of the salt.

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EXPERIMENT - 6(a)

Aim

To purify the given sample of organic compound by recrystallisation using water as the solvent.

Apparatus Required

Beaker, glass rod, funnel, filter paper, conical flask, water bath, wire gauze, burner, tripod stand, watch glass.

Chemicals Required

Given impure organic compound (e.g., benzoic acid/impure salt), distilled water, ice (for cooling).

Theory

1. Recrystallisation is a technique used to purify a solid organic compound based on the difference in solubility of the compound and its impurities in a given solvent at different temperatures.

2. An ideal solvent dissolves the compound well at high temperature but poorly at low/room temperature, while the impurities either remain insoluble (removed by hot filtration) or remain in solution (removed in the mother liquor).

3. On cooling the hot saturated solution, the pure compound crystallises out, leaving the soluble impurities behind in solution.

4. Water is chosen as solvent for compounds that are sparingly soluble in cold water but freely soluble in hot water.

Procedure

Procedure

1. Take the given impure organic compound in a beaker and add the minimum amount of water required to dissolve it completely on heating.

2. Heat the mixture gently on a water bath with constant stirring until the solid dissolves completely to form a saturated solution.

3. If the solution is coloured or contains insoluble impurities, filter it hot through a fluted filter paper using a pre-heated funnel.

4. Collect the hot filtrate in a clean beaker/conical flask and allow it to cool slowly to room temperature, then further cool in an ice bath.

5. Pure crystals of the compound separate out on cooling.

6. Filter the crystals using suction/ordinary filtration and wash with a small amount of cold water.

7. Dry the crystals between folds of filter paper or in an oven at low temperature.

8. Determine the melting point of the purified crystals and compare with the literature value to check purity.

Calculation

Weight of impure compound taken = _____ g

Weight of pure crystals obtained after recrystallisation = _____ g

Percentage yield = (Weight of pure product / Weight of impure product) x 100 = _____ %

Melting point of purified compound = _____ °C (compare with standard value)

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Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the principle of recrystallisation?

Ans. It is based on the difference in solubility of a compound and its impurities in a solvent at different temperatures — the compound is more soluble in hot solvent and separates out as pure crystals on cooling.

Q2. How do you choose a suitable solvent for recrystallisation?

Ans. An ideal solvent dissolves the compound well when hot but poorly when cold, does not react with the compound, and dissolves impurities either completely (so they stay in the mother liquor) or not at all (so they are removed by hot filtration).

Q3. Why is hot filtration carried out?

Ans. To remove insoluble impurities while the compound is still in solution, before it has a chance to crystallise out in the funnel/filter paper.

Q4. Why is a fluted filter paper used for hot filtration?

Ans. It increases the filtration surface area, allowing faster filtration before the hot solution cools and crystals start depositing on the filter paper.

Q5. How do you check whether the recrystallised compound is pure?

Ans. By determining its melting point; a pure compound shows a sharp melting point close to the literature value.

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EXPERIMENT - 6(b)

Aim

To purify the given sample of organic compound by recrystallisation using alcohol (ethanol) as the solvent.

Apparatus Required

Beaker, glass rod, funnel, filter paper, conical flask, water bath, wire gauze, burner, tripod stand, watch glass.

Chemicals Required

Given impure organic compound, rectified spirit/ethanol, ice (for cooling).

Theory

1. Alcohol (ethanol) is used as the recrystallising solvent when the given organic compound is insoluble or poorly soluble in water but readily soluble in alcohol on heating and poorly soluble in cold alcohol.

2. The principle remains the same as in water recrystallisation: the compound is dissolved in the minimum volume of hot solvent, filtered hot to remove insoluble impurities, and the filtrate is cooled to allow pure crystals to separate, leaving soluble impurities in the mother liquor.

3. Since alcohol is inflammable, heating must be carried out carefully on a water bath, away from any naked flame.

Procedure

Procedure

1. Take the given impure organic compound in a beaker and add the minimum quantity of rectified spirit (ethanol) required to dissolve it.

2. Heat the mixture gently on a water bath (never over a direct flame) with constant stirring until a clear saturated solution is obtained.

3. If necessary, filter the hot solution through a fluted filter paper using a pre-warmed funnel to remove insoluble impurities.

4. Collect the hot filtrate and allow it to cool slowly to room temperature, then cool further in an ice bath to complete crystallisation.

5. Filter off the crystals and wash with a small quantity of cold alcohol.

6. Dry the purified crystals between folds of filter paper.

7. Determine the melting point of the purified compound to check its purity.

Calculation

Weight of impure compound taken = _____ g

Weight of pure crystals obtained after recrystallisation = _____ g

Percentage yield = (Weight of pure product / Weight of impure product) x 100 = _____ %

Melting point of purified compound = _____ °C (compare with standard value)

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

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Viva-Voce Questions

Q1. Why is alcohol chosen as a solvent instead of water for some compounds?

Ans. When a compound is insoluble or poorly soluble in water but readily soluble in hot alcohol and poorly soluble in cold alcohol, alcohol becomes the suitable recrystallising solvent.

Q2. Why should alcohol never be heated over a naked flame?

Ans. Alcohol is highly inflammable and its vapours can easily catch fire; it must always be heated on a water bath.

Q3. What happens if too much solvent is used while dissolving the compound?

Ans. Using excess solvent reduces the yield of crystals on cooling, since more of the compound remains dissolved in the mother liquor.

Q4. Why is the solution cooled slowly rather than rapidly?

Ans. Slow cooling allows the formation of larger, purer crystals, whereas rapid cooling gives small crystals that may trap impurities.

Q5. What is 'mother liquor'?

Ans. The solvent left behind after the pure crystals have separated out and been filtered off; it contains the dissolved impurities.

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EXPERIMENT - 7

Aim

To determine the melting point of the given organic compound using the Kjeldahl (capillary tube) method with an electrical melting point apparatus.

Apparatus Required

Electrical melting point (Kjeldahl type) apparatus, capillary tubes (sealed at one end), thermometer, rubber band/thread, mortar and pestle.

Chemicals Required

Given organic compound (finely powdered), liquid paraffin (if using the classical Kjeldahl flask method).

Theory

1. Melting point is the temperature at which a solid substance changes into its liquid state at atmospheric pressure, and is used as a criterion of purity of an organic compound.

2. A pure compound has a sharp melting point (melts within a range of 1-2 °C), while an impure compound melts over a wider range and generally at a lower temperature.

3. In the Kjeldahl method, a Kjeldahl flask containing a high-boiling liquid (such as liquid paraffin or conc. H_2SO_4) is used as the heating bath, with a capillary tube containing the sample and a thermometer tied together and immersed in the bath; in the electrical apparatus, this heating bath is replaced by an electrically heated mŋl block, making the determination faster and safer.

4. The temperature range during which the solid starts melting (first appearance of liquid) and completely turns to liquid is recorded as the melting point range of the compound.

Procedure

Procedure

1. Grind the given organic compound to a fine powder using a mortar and pestle.

2. Fill a small amount of the powdered sample into a capillary tube (sealed at one end) by tapping the open end on the powder, then invert and drop it through a long glass tube to pack the sample at the sealed end (height of column about 2-3 mm).

3. Attach the capillary tube to the thermometer using a thin rubber band or thread such that the sample-filled portion is next to the thermometer bulb.

4. Insert the thermometer-capillary assembly into the electrical melting point apparatus.

5. Switch on the apparatus and heat slowly, increasing the temperature gradually (about 1-2 °C per minute) as the expected melting point is approached.

6. Note the temperature at which the solid just starts to melt (T_1) and the temperature at which it melts completely (T_2) by observing through the magnifying lens/window of the apparatus.

7. Repeat the determination with a fresh capillary for a check reading.

8. Report the melting point as the range T_1-T_2 °C.

Observation Table

Observation Table: Melting point determination

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Sl. No.	Trial	Temp. at which melting starts (T_1) °C	Temp. at which melting completes (T_2) °C	Melting point range (°C)
1				
2				

Calculation

Melting point range observed = T_1 to T_2 = _____ to _____ °C

Mean melting point = (T_1 + T_2) / 2 = _____ °C

Literature (standard) melting point of the compound = _____ °C

Conclusion: The given compound is pure/impure, since the observed melting point range is sharp/wide and close to/deviates from the standard value.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is melting point and why is it used as a criterion of purity?

Ans. Melting point is the temperature at which a solid changes to liquid at atmospheric pressure. A pure compound has a sharp, well-defined melting point, while impurities lower and broaden this range, making it a simple purity check.

Q2. Why does an impure compound show a lower and broader melting point?

Ans. Impurities disrupt the regular crystal lattice, lowering the melting point (freezing point depression) and widening the melting range since different crystallite regions melt at slightly different temperatures.

Q3. Why is the capillary tube sealed at one end?

Ans. To hold the powdered sample in place at the bottom of the tube, next to the thermometer bulb, without the sample escaping or getting mixed into the heating bath/block.

Q4. Why should the rate of heating be slowed down near the expected melting point?

Ans. To allow enough time for uniform heat transfer and accurate observation of the exact temperature at which melting starts and completes.

Q5. What is the advantage of the electrical melting point apparatus over the classical Kjeldahl flask method?

Ans. It is faster, safer (no hot liquid bath/flame), gives more uniform and controllable heating, and allows easier visual observation through a built-in magnifying lens.

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EXPERIMENT - 8

Aim

To identify and separate the components of a given mixture of two amino acids by ascending paper chromatography and to determine their Rf values.

Apparatus Required

Chromatography chamber/jar with lid, Whatman filter paper (chromatography paper), capillary tube/micropipette, measuring cylinder, scale/ruler, pencil, hair dryer/oven, atomiser/sprayer.

Chemicals Required

Mixture of two amino acids (test solution), standard amino acid solutions, solvent system (n-butanol : acetic acid : water, e.g., 4:1:5), ninhydrin spray reagent, acetone.

Theory

1. Paper chromatography is a technique used to separate and identify the components of a mixture based on their differential movement (partition) between a stationary phase (water held in the fibres of filter paper) and a mobile phase (the moving organic solvent).

2. In ascending paper chromatography, the solvent moves upward through the paper by capillary action, carrying the components of the mixture at different rates depending on their relative solubility in the two phases.

3. Amino acids are colourless and are detected after development by spraying with ninhydrin reagent, which gives a characteristic purple/pink colour with amino acids on heating.

4. The Rf (rɳrdation factor) value is the ratio of the distance travelled by the solute (amino acid spot) to the distance travelled by the solvent front, and is characteristic of a compound in a given solvent system.

5. Rf = Distance travelled by the solute / Distance travelled by the solvent front

Procedure

Procedure

1. Take a rectangular sheet of Whatman chromatography paper and draw a pencil line (origin/base line) about 2-3 cm from one edge.

2. Mark three points on the base line for (i) standard amino acid 1, (ii) standard amino acid 2, and (iii) the given mixture.

3. Using a fine capillary tube, spot small quantities of the respective solutions at the marked points and allow each spot to dry.

4. Prepare the solvent system (n-butanol : acetic acid : water in the ratio 4:1:5) and pour it into the chromatographic chamber to a depth of about 1 cm; keep the chamber saturated with solvent vapours by closing the lid.

5. Suspend the chromatography paper in the chamber such that the base line is above the solvent level but the lower edge of the paper dips into the solvent, without the spots touching the solvent.

6. Close the chamber and allow the solvent to rise up the paper by capillary action (ascending technique) undisturbed until the solvent front reaches near the top of the paper.

7. Remove the paper, immediately mark the solvent front with a pencil, and dry the paper (in air or using a hair dryer).

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8. Spray the dried chromatogram with ninhydrin reagent and heat gently in an oven (or with a hair dryer) at about 100-105 °C for a few minutes until purple/pink coloured spots appear.

9. Mark the centre of each spot, measure the distances travelled by each spot and by the solvent front from the base line, and calculate the Rf values.

Observation Table

Observation Table: Rf values of amino acids

Sl. No.	Sample / Spot	Distance travelled by solute (cm)	Distance travelled by solvent front (cm)	Rf value
1				
2				
3				
4				

Calculation

Rf = Distance travelled by the solute (spot) / Distance travelled by the solvent front

Rf (standard amino acid 1) = _____ / _____ = _____

Rf (standard amino acid 2) = _____ / _____ = _____

Rf (component 1 of mixture) = _____ / _____ = _____

Rf (component 2 of mixture) = _____ / _____ = _____

Conclusion: By comparing the Rf values of the mixture components with those of the standard amino acids, the given mixture is identified as a mixture of _____ and _____.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the principle of paper chromatography?

Ans. Separation occurs due to differential partition of the components of a mixture between a stationary phase (water held in paper fibres) and a mobile phase (moving solvent), causing different components to travel different distances.

Q2. What is Rf value?

Ans. Rf (Rnrdation factor) is the ratio of the distance travelled by a solute (spot) to the distance travelled by the solvent front, measured from the same origin.

Q3. Why is the chromatography chamber kept saturated with solvent vapours?

Ans. To prevent uneven evaporation of the solvent from the paper during the run, ensuring uniform and reproducible movement of the solvent front.

Q4. Why are amino acids colourless and how are they detected on the chromatogram?

Ans. Amino acids have no chromophore that absorbs visible light; they are detected by spraying with ninhydrin reagent, which reacts with the amino group to give a purple/pink colour on heating.

Q5. Why should the spots not touch the solvent surface initially?

Ans. If the spots dip into the solvent, the compounds would dissolve directly into the solvent reservoir instead of being carried up the paper, ruining the separation.

Q6. Does the Rf value of a compound change with a different solvent system?

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Ans. Yes, Rf value depends on the solvent system used; it is a constant only for a given compound under fixed, identical experimental conditions (solvent, temperature, paper).

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EXPERIMENT - 9

Aim

To prepare a standard solution of a given substance of specified Normality/Molarity.

Apparatus Required

Weighing balance, watch glass, funnel, glass rod, volumetric flask, wash bottle.

Chemicals Required

Given primary standard substance (e.g., oxalic acid, $K_2Cr_2O_7$, Na_2CO_3), distilled water.

Theory

1. A standard solution is one whose concentration (strength) is accurately known.

2. Primary standard substances are those that are available in a pure, stable form, do not absorb moisture, and whose solutions can be prepared by directly weighing the calculated amount and dissolving in a known volume of solvent.

3. Normality (N) = (Weight of substance in g / Equivalent weight) x (1000 / Volume of solution in mL)

4. Molarity (M) = (Weight of substance in g / Molecular weight) x (1000 / Volume of solution in mL)

5. The calculated amount of the solid is weighed accurately, dissolved completely in distilled water, and the solution is made up to the mark in a standard volumetric flask to get the solution of exact known strength.

Procedure

Procedure

1. Calculate the weight of the substance required to prepare the given volume of solution of the desired normality/molarity.

2. Weigh the calculated amount accurately on a weighing balance using a clean watch glass.

3. Transfer the weighed substance quantitatively into a clean volumetric flask of the required capacity using a funnel; wash the watch glass and funnel with distilled water into the flask.

4. Add distilled water to about half the volume of the flask and swirl/shake gently to dissolve the solid completely.

5. Make up the final volume to the calibration mark with distilled water, ensuring the meniscus touches the mark exactly.

6. Stopper the flask and mix the solution thoroughly by inverting several times.

Calculation

N (or M) = (Weight of substance / Eq. wt. or Mol. wt.) x (1000 / Volume in mL)

Weight required = (N x Eq. wt. x Volume) / 1000 (for Normal solution)

Weight required = (M x Mol. wt. x Volume) / 1000 (for Molar solution)

Weight actually taken = _____ g

Strength of solution prepared = _____ N / M

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

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Viva-Voce Questions

Q1. What is a primary standard substance? Give an example.

Ans. A substance available in pure, stable form, unaffected by air/moisture, and with a known constant composition, used to prepare a solution of accurately known concentration directly by weighing. Example: oxalic acid, $K_2Cr_2O_7$, anhydrous Na_2CO_3.

Q2. What is the difference between Normality and Molarity?

Ans. Molarity is moles of solute per litre of solution; Normality is gram-equivalents of solute per litre of solution. Normality = Molarity x n-factor.

Q3. Why must a volumetric flask be used instead of a beaker for preparing a standard solution?

Ans. A volumetric flask has a single calibration mark guaranteeing an accurate, fixed volume, which a beaker (only approximately graduated) cannot provide.

Q4. Why is the solid dissolved in a small amount of water before making up to the final volume?

Ans. To ensure the solid dissolves completely before the final dilution; adding solvent directly to the mark first could make it difficult to dissolve the solute without exceeding the calibration mark.

Q5. What precaution should be taken while making up the solution to the mark?

Ans. The eye should be level with the calibration mark, and water should be added dropwise near the mark so that the bottom of the meniscus exactly touches the graduation line.

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EXPERIMENT - 10

Aim

To estimate the strength of the given Na_2CO_3/NaOH solution by titrating it against standard HCl solution.

Apparatus Required

Burette, pipette, conical flask, burette stand, measuring flask.

Chemicals Required

Given Na_2CO_3/NaOH solution, standard HCl solution, phenolphthalein indicator, methyl orange indicator, distilled water.

Theory

1. This is a simple acid-base (neutralisation) titration.

2. Reaction: $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$ (or $NaOH + HCl \rightarrow NaCl + H_2O$)

3. Phenolphthalein is used as indicator when titrating NaOH (colourless in acid, pink in base; end point = pink to colourless).

4. For Na_2CO_3, titration may be carried out using phenolphthalein (first end point, to $NaHCO_3$) followed by methyl orange (second end point, complete neutralisation), or methyl orange alone can be used for the complete reaction (yellow to orange/pink).

Procedure

Procedure

1. Pipette out 10 mL of the given Na_2CO_3/NaOH solution into a conical flask.

2. Add 2-3 drops of phenolphthalein indicator (for NaOH) or methyl orange indicator (for Na_2CO_3).

3. Titrate with standard HCl solution taken in the burette, with constant swirling, until the colour changes at the end point (pink to colourless for phenolphthalein; yellow to onion-pink for methyl orange).

4. Repeat the titration to get three concordant readings.

Observation Table

Observation Table: Titration of Na_2CO_3/NaOH against standard HCl

Sl. No.	Volume of Na_2CO_3/NaOH solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Calculation

N_1V_1 (Na_2CO_3/NaOH) = N_2V_2 (HCl)

N(Na_2CO_3/NaOH) x 10 = N(HCl) x V(HCl)

N(Na_2CO_3/NaOH) = [N(HCl) x V(HCl)] / 10

Strength (g/L) = N(Na_2CO_3/NaOH) x Equivalent weight

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Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. Why is phenolphthalein unsuitable for titrating a weak acid against a weak base?

Ans. Phenolphthalein changes colour only in a narrow alkaline pH range (8.3-10), and the pH at the equivalence point of a weak acid-weak base titration does not fall sharply within this range, giving no sharp end point.

Q2. What is the colour change of phenolphthalein at the end point?

Ans. Pink to colourless, when titrating a base with an acid.

Q3. What is the colour change of methyl orange at the end point?

Ans. Yellow to orange/onion-pink.

Q4. Why does Na_2CO_3 titration with HCl using phenolphthalein stop at $NaHCO_3$ and not go further?

Ans. Phenolphthalein's colour change range corresponds to the first equivalence point (conversion of Na_2CO_3 to $NaHCO_3$); the second equivalence point ($NaHCO_3$ to H_2CO_3/CO_2+H_2O) occurs at a lower pH detected by methyl orange.

Q5. What is a neutralisation reaction?

Ans. A reaction between an acid and a base that produces a salt and water, with the H^+ ions from the acid combining with OH^- ions from the base.

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EXPERIMENT - 11

Aim

To determine the temporary, permanent, and total hardness of the given sample of water by complexometric titration using standard EDTA solution.

Apparatus Required

Burette, pipette, conical flask, burette stand, beaker, burner, wire gauze, tripod stand.

Chemicals Required

Given hard water sample, standard EDTA (disodium salt) solution, ammonia buffer (NH_4Cl-NH_4OH, pH 10), Eriochrome Black-T (EBT) indicator.

Theory

1. Hardness of water is caused mainly by dissolved calcium and magnesium salts (bicarbonates, chlorides, sulphates).

2. Temporary hardness is due to bicarbonates of Ca and Mg, and can be removed by boiling (converted to insoluble carbonates).

3. Permanent hardness is due to chlorides and sulphates of Ca and Mg, and remains even after boiling.

4. Total hardness = Temporary hardness + Permanent hardness.

5. EDTA (ethylenediaminetetraacetic acid, disodium salt) forms stable 1:1 complexes with Ca^{2+} and Mg^{2+} ions at pH 10 (maintained using ammonia buffer).

6. Eriochrome Black-T (EBT) forms a wine-red complex with free Ca^{2+}/Mg^{2+} ions; at the end point, EDTA displaces the indicator from the mηl ion, and the colour changes from wine-red to steel blue (free indicator colour).

Procedure

I. Estimation of Total Hardness

1. Pipette out 10 mL of the given hard water sample into a conical flask.

2. Add about 1 mL of ammonia buffer solution to maintain pH 10.

3. Add 2-3 drops of Eriochrome Black-T indicator; the solution turns wine-red.

4. Titrate with standard EDTA solution taken in the burette until the colour changes sharply from wine-red to steel/pure blue.

5. Repeat to get three concordant readings — this gives the Total Hardness.

II. Estimation of Permanent Hardness

1. Take 100 mL of the given hard water sample in a beaker and boil gently for about 20-30 minutes to remove temporary hardness (as insoluble carbonates).

2. Cool, filter off the precipitate, and make up the filtrate to the original volume (100 mL) with distilled water.

3. Pipette out 10 mL of this boiled water sample, add buffer and EBT indicator as before.

4. Titrate with standard EDTA solution until the colour changes from wine-red to blue — this gives the Permanent Hardness.

5. Temporary Hardness = Total Hardness - Permanent Hardness.

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Observation Tables

Observation Table 1: Total hardness (before boiling)

Sl. No.	Volume of water sample (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Observation Table 2: Permanent hardness (after boiling)

Sl. No.	Volume of boiled water sample (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Calculation

N_1V_1 (water sample) = N_2V_2 (EDTA)

Total Hardness: N(water) x 10 = N(EDTA) x V_1(EDTA) => N(water) = [N(EDTA) x V_1] / 10

Permanent Hardness: N(water) x 10 = N(EDTA) x V_2(EDTA) => N(water) = [N(EDTA) x V_2] / 10

Hardness (as $CaCO_3$ equivalent, ppm) = N(water) x 50 x 1000

Total Hardness = _____ ppm

Permanent Hardness = _____ ppm

Temporary Hardness = Total Hardness - Permanent Hardness = _____ ppm

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is hardness of water and what causes it?

Ans. Hardness is the inability of water to produce lather easily with soap, caused mainly by dissolved calcium and magnesium salts (bicarbonates, chlorides, sulphates).

Q2. Differentiate between temporary and permanent hardness.

Ans. Temporary hardness is due to bicarbonates of Ca/Mg and is removed by simple boiling; permanent hardness is due to chlorides/sulphates of Ca/Mg and is not removed by boiling.

Q3. Why is ammonia buffer (pH 10) added before titrating with EDTA?

Ans. EDTA forms the most stable complex with Ca^{2+} and Mg^{2+} at pH about 10; the buffer maintains this optimum pH for a sharp end point with Eriochrome Black-T.

Q4. What is the colour change with EBT indicator at the end point?

Ans. Wine-red (mŋl-indicator complex) to steel/pure blue (free indicator), indicating that all mŋl ions are now complexed with EDTA.

Q5. Why is EDTA called a chelating agent?

Ans. EDTA has multiple donor atoms (two nitrogen and four oxygen atoms) that bind to a single mŋl ion simultaneously, forming a stable ring-like (chelate) complex.

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Q6. In what units is hardness usually expressed?

Ans. In parts per million (ppm) as calcium carbonate ($CaCO_3$) equivalents.

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EXPERIMENT - 12

Aim

To estimate the percentage of available chlorine in the given sample of bleaching powder iodometrically.

Apparatus Required

Burette, pipette, conical flask (glass stoppered), weighing balance, volumetric flask, funnel.

Chemicals Required

Given bleaching powder sample, potassium iodide (KI), dilute acetic acid/dilute H_2SO_4, standard sodium thiosulphate ($Na_2S_2O_3$) solution, starch indicator.

Theory

1. Bleaching powder, Ca(OCl)Cl, liberates iodine from potassium iodide solution in the presence of dilute acid; the hypochlorite (available chlorine) is responsible for the oxidising/bleaching action.

2. Reaction: $Ca(OCl)Cl + 2KI + 2CH_3COOH \rightarrow CaCl_2 + I_2 + 2CH_3COOK + H_2O$

3. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as indicator.

4. $I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

5. The amount of iodine liberated (equivalent to the thiosulphate consumed) is used to calculate the percentage of available chlorine.

Procedure

Procedure

1. Weigh accurately about 1 g of the given bleaching powder sample and make a smooth paste with a little distilled water.

2. Transfer to a 100 mL volumetric flask and make up to the mark with distilled water; shake well and filter if required.

3. Pipette out 10 mL of this solution into a conical flask, add about 1 g KI and 10 mL dilute acetic acid; a brown colour due to liberated iodine appears.

4. Titrate immediately with standard $Na_2S_2O_3$ solution from the burette until the solution turns pale yellow.

5. Add 1-2 mL of freshly prepared starch indicator; the solution turns blue.

6. Continue titrating until the blue colour just disappears.

7. Repeat to get three concordant readings.

Observation Table

Observation Table: Titration of bleaching powder solution against $Na_2S_2O_3$

Sl. No.	Volume of bleaching powder solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

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Calculation

Weight of bleaching powder taken = W g (made up to 100 mL)

Moles of $Na_2S_2O_3$ used = ($N(Na_2S_2O_3)$ x $V(Na_2S_2O_3)$) / 1000

Equivalents of I_2 liberated = Equivalents of $Na_2S_2O_3$ used = Equivalents of available Cl_2

Weight of available chlorine (in 10 mL) = $N(Na_2S_2O_3)$ x $V(Na_2S_2O_3)$ x 35.5 / 1000

Weight of available chlorine (in 100 mL) = above x 10

Percentage of available chlorine = (Weight of available chlorine / W) x 100

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is meant by 'available chlorine' in bleaching powder?

Ans. It is the amount of chlorine that bleaching powder can liberate when treated with a dilute acid; it is a measure of the oxidising/bleaching power of the sample.

Q2. What is the chemical formula of bleaching powder?

Ans. Ca(OCl)Cl, i.e. calcium oxychloride (calcium hypochlorite-chloride).

Q3. Why is acetic acid preferred over a strong mineral acid in this estimation?

Ans. Acetic acid is a weak acid that liberates iodine from KI without causing side reactions, whereas strong mineral acids may cause additional oxidation side-reactions affecting accuracy.

Q4. Why is bleaching powder not very stable on storage?

Ans. It slowly decomposes on exposure to air, moisture, and light, losing available chlorine over time.

Q5. What is the use of starch indicator here?

Ans. It forms a deep blue complex with the last traces of iodine, giving a sharp end point (blue to colourless) when all the iodine has reacted with thiosulphate.

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EXPERIMENT - 13

Aim

To estimate the amount of calcium present in the given solution by complexometric titration with standard EDTA solution.

Apparatus Required

Burette, pipette, conical flask, burette stand, measuring cylinder.

Chemicals Required

Given Ca^{2+} solution, standard EDTA solution, NaOH solution (to maintain pH ~12-13), Patton and Reeder's/Murexide indicator, distilled water.

Theory

1. EDTA forms a stable 1:1 complex with Ca^{2+} ions.

2. At a high pH (about 12-13, maintained using NaOH), Mg^{2+} (if present) precipitates as $Mg(OH)_2$ and does not interfere, allowing selective estimation of Ca^{2+} alone.

3. Murexide (or Patton and Reeder's indicator) forms a pink/red complex with free Ca^{2+} ions at this pH; at the end point, EDTA displaces the indicator, and the colour changes from pink to violet/purple.

4. The volume of EDTA consumed is directly proportional to the amount of Ca^{2+} present.

Procedure

Procedure

1. Pipette out 10 mL of the given Ca^{2+} solution into a conical flask.

2. Add about 2 mL of NaOH solution to raise the pH to about 12-13.

3. Add a pinch of Murexide indicator; the solution turns pink.

4. Titrate with standard EDTA solution taken in the burette until the colour changes sharply from pink to violet/purple.

5. Repeat the titration to get three concordant readings.

Observation Table

Observation Table: Titration of Ca^{2+} solution against EDTA

Sl. No.	Volume of Ca^{2+} solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume Consumed (mL)
1	10			
2	10			
3	10			

Calculation

N_1V_1 (Ca^{2+}) = N_2V_2 (EDTA)

$N(Ca^{2+}) \times 10 = N(EDTA) \times V(EDTA)$

$N(Ca^{2+}) = [N(EDTA) \times V(EDTA)] / 10$

Amount of Ca^{2+} per litre = $N(Ca^{2+})$ x Equivalent weight of Ca (20) = _____ g/L

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Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. Why is a high pH (about 12-13) maintained in this titration unlike the total hardness titration (pH 10)?

Ans. At pH 12-13, any Mg^{2+} present precipitates as $Mg(OH)_2$ and no longer reacts with EDTA, allowing selective/direct estimation of only Ca^{2+}.

Q2. Why is Murexide used as indicator here instead of EBT?

Ans. Murexide (ammonium purpurate) is specific and gives a sharp pink-to-violet colour change with Ca^{2+} at high pH, whereas EBT does not give a clear end point under these strongly alkaline conditions.

Q3. What is the colour change at the end point with Murexide?

Ans. Pink to violet/purple.

Q4. What is the stoichiometry of the Ca^{2+}-EDTA complex?

Ans. It is a 1:1 complex — one mole of EDTA combines with one mole of Ca^{2+}.

Q5. Why is NaOH, and not ammonia buffer, used to raise the pH in this experiment?

Ans. NaOH effectively raises the pH to the required high value (12-13) and simultaneously precipitates Mg^{2+} as $Mg(OH)_2$, which ammonia buffer (pH ~10) cannot achieve.

Teacher's Signature: _____

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EXPERIMENT - 14

Aim

To analyse the given simple inorganic salt and identify the acid radical (anion) and basic radical (cation) present in it by systematic semimicro qualitative analysis.

Apparatus Required

Test tubes, test tube stand, test tube holder, burner, watch glass, china dish, glass rod, dropper, platinum/nichrome wire.

Chemicals Required

Given salt sample, dilute HCl, dilute H_2SO_4, dilute/conc. HNO_3, $BaCl_2$, $AgNO_3$, NH_4OH, group reagents for cation analysis (H_2S, NH_4Cl, $(NH_4)_2CO_3$ etc.), specific confirmatory reagents.

Theory

1. Qualitative analysis of an inorganic salt involves identifying its constituent acid radical (anion) and basic radical (cation) through a systematic series of preliminary tests, dry tests, and wet (confirmatory) tests.

2. Preliminary tests include observation of colour, odour, and action of heat on the dry salt, which give preliminary clues about the nature of the salt.

3. The acid radical (anion) is first identified by preliminary tests (dilute H_2SO_4 test, conc. H_2SO_4 test) followed by confirmatory tests specific to the suspected anion (e.g., $AgNO_3$ test for halides, $BaCl_2$ test for sulphate/carbonate).

4. The basic radical (cation) is identified by the systematic group separation scheme, where the solution of the salt is treated with group reagents in sequence (Group I to Group VI reagents) to separate cations into groups, followed by specific confirmatory tests for the individual cation.

Procedure

I. Preliminary tests

1. Note the colour, physical state, and odour of the given dry salt.

2. Perform the dry heating test (in a dry test tube) and note any colour change, gas evolution, sublimation, etc.

3. Perform the flame test using a platinum/nichrome wire to get preliminary information about the cation present.

II. Analysis for acid radical (anion)

1. Treat a small quantity of the salt with dilute H_2SO_4 (cold, then warm) and note the gas evolved, if any, along with its characteristic properties (smell, effect on litmus, lime water, etc.).

2. Treat a fresh portion with concentrated H_2SO_4 and note the observations.

3. Based on the preliminary observations, perform specific confirmatory tests (e.g., $AgNO_3$ test for Cl^-/Br-/I^-, $BaCl_2$ test for SO_4^{2-}/CO_3^{2-}, ferrous sulphate (brown ring) test for NO_3^-) to confirm the acid radical present.

III. Analysis for basic radical (cation)

1. Prepare the original solution of the salt in distilled water/dilute acid as appropriate.

2. Carry out the systematic cation analysis by adding group reagents in sequence and separating the precipitate of each group by filtration.

3. Confirm the individual cation present in the precipitate/filtrate of the relevant group by specific confirmatory tests.

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Observation Tables

Observation Table: Analysis of acid radical

Sl. No.	Experiment / Test performed	Observation	Inference
1			
2			
3			

Observation Table: Analysis of basic radical

Sl. No.	Experiment / Test performed	Observation	Inference
1			
2			
3			

Calculation

No numerical calculation is involved in this qualitative experiment.

Result: The given salt contains:

Acid radical (anion) = _____

Basic radical (cation) = _____

Hence, the given salt is identified as _____.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the purpose of the preliminary dry heating test?

Ans. It gives early clues about the nature of the salt — e.g., colour change, evolution of gas, sublimation, or decrepitation — which help narrow down possible radicals before wet tests.

Q2. Why is a flame test performed?

Ans. Certain cations impart characteristic colours to a non-luminous flame (e.g., sodium - golden yellow, potassium - lilac, calcium - brick red, copper - green), giving a quick preliminary indication of the cation present.

Q3. Why is the salt treated with dilute H_2SO_4 before concentrated H_2SO_4 in anion analysis?

Ans. Dilute H_2SO_4 identifies anions that react even with a weak acid (like carbonates, sulphides), while concentrated H_2SO_4 is needed to detect anions that require a stronger oxidising/dehydrating acid (like chlorides, nitrates).

Q4. Why are cations divided into analytical groups?

Ans. To systematically separate cations based on the differing solubility of their compounds with specific group reagents, simplifying identification of the individual cation present.

Q5. What is a confirmatory test?

Ans. A specific chemical test that gives a unique, unambiguous result (precipitate/colour) for a particular ion, confirming its presence beyond doubt after preliminary tests.

Teacher's Signature: _____

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EXPERIMENT - 15

Aim

To analyse the given mixture of two inorganic salts and identify the acid radicals and basic radicals present in it.

Apparatus Required

Test tubes, test tube stand, test tube holder, burner, watch glass, china dish, glass rod, dropper, platinum/nichrome wire, filter paper, funnel.

Chemicals Required

Given mixture of salts, dilute HCl, dilute H_2SO_4, dilute/conc. HNO_3, group reagents for systematic cation analysis, specific confirmatory reagents.

Theory

1. A mixture of salts may contain two (or more) different cations and/or anions which may interfere with each other during analysis; hence a careful systematic procedure is followed.

2. Preliminary dry tests (colour, heating, flame test) give an idea whether the mixture contains more than one radical.

3. The acid radicals present are identified by carrying out the usual anion tests and noting if the salt gives evidence of more than one anion (for example, both an effervescence as well as a precipitate test).

4. The basic radicals (cations) are separated systematically into their analytical groups using the group reagents, and each group precipitate/filtrate is analysed individually for the presence of more than one cation using confirmatory tests.

Procedure

I. Preliminary tests

1. Note the colour and physical state of the given mixture.

2. Perform the dry heating test and note the observations.

3. Perform the flame test for preliminary cation information.

II. Analysis for acid radicals

1. Treat the mixture with dilute H_2SO_4 (cold and warm) and note all observations (more than one gas/reaction may be noted if two anions are present).

2. Treat a fresh portion with concentrated H_2SO_4.

3. Carry out confirmatory tests for each suspected anion separately to confirm the presence of two different acid radicals.

III. Analysis for basic radicals

1. Prepare the original solution of the mixture.

2. Pass the solution through the systematic group separation scheme, adding group reagents in order (Group I, II, III, IV, V, VI) and separating the precipitate formed at each stage.

3. Analyse the precipitate of each group (and the final filtrate) for the possible presence of one or two cations using specific confirmatory tests.

Observation Tables

Observation Table: Analysis of acid radicals

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Sl. No.	Experiment / Test performed	Observation	Inference
1			
2			
3			

Observation Table: Analysis of basic radicals

Sl. No.	Experiment / Test performed	Observation	Inference
1			
2			
3			

Calculation

No numerical calculation is involved in this qualitative experiment.

Result: The given mixture contains:

Acid radicals = _____ and _____

Basic radicals = _____ and _____

Hence, the given mixture consists of _____ and _____.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the main challenge in analysing a mixture of two salts compared to a single salt?

Ans. The ions of one salt may interfere with, mask, or give a false test result for the other, so tests and separations must be carried out carefully and systematically.

Q2. How can you tell from preliminary tests that a sample contains more than one anion?

Ans. Observing more than one type of reaction (e.g., both effervescence and the formation of a precipitate/odour) when treated with dilute/concentrated acids suggests more than one anion is present.

Q3. Why is systematic group analysis especially important for a mixture?

Ans. It ensures that cations are separated into their correct groups one at a time, preventing one cation from being missed or wrongly identified due to the presence of another.

Q4. What precaution is taken while adding group reagents to a mixture?

Ans. The exact specified quantity of reagent must be added and the solution tested for complete precipitation, ensuring that no cation of an earlier group remains to contaminate a later group's precipitate.

Q5. Can an anion test be applied directly to a solid mixture?

Ans. Yes, preliminary anion tests are generally done on the solid mixture directly with dilute/concentrated acids, before cations are brought into solution for systematic group analysis.

Teacher's Signature: _____

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EXPERIMENT - 16

Aim

To detect the presence of extra elements — nitrogen, sulphur, and halogens — in the given organic compound by Lassaigne's test.

Apparatus Required

Ignition tube/fusion tube, burner, tongs, test tubes, china dish, filter paper, funnel, dropper.

Chemicals Required

Given organic compound, sodium mŋl, $FeSO_4$, $FeCl_3$, conc. HCl, dilute H_2SO_4, sodium nitroprusside, lead acŋte, dilute HNO_3, $AgNO_3$.

Theory

1. Organic compounds often contain elements such as nitrogen, sulphur, and halogens in addition to carbon, hydrogen, and oxygen; these are detected by Lassaigne's sodium fusion test, which converts these covalently bound elements into easily detectable ionic (inorganic) forms.

2. On fusion with sodium mŋl, nitrogen is converted to sodium cyanide (NaCN), sulphur to sodium sulphide (Na_2S), and halogens to sodium halide (NaX).

3. If both N and S are present, sodium thiocyanate (NaSCN) is formed.

4. The sodium fusion extract (Lassaigne's extract) is then tested as follows: for nitrogen — Prussian blue test with $FeSO_4$/$FeCl_3$; for sulphur — sodium nitroprusside test (violet colour) and lead acŋte test (black precipitate); for halogens — $AgNO_3$ test after boiling with dilute HNO_3 (to remove interference from CN-/S2-).

Procedure

I. Preparation of sodium fusion extract (Lassaigne's extract)

1. Take a small piece of clean, dry sodium mŋl in a dry ignition/fusion tube.

2. Heat the tube gently until the sodium melts; then add a small quantity of the organic compound directly onto the molten sodium.

3. Heat the tube strongly (to red heat) for 1-2 minutes to ensure complete fusion.

4. Plunge the red-hot tube into a china dish containing about 10-15 mL of distilled water (handle with care).

5. Break the tube and boil the contents for a few minutes; filter the solution. The clear filtrate is the sodium fusion (Lassaigne's) extract.

II. Test for Nitrogen

1. Take 2 mL of the Lassaigne's extract in a test tube; add a few drops of freshly prepared $FeSO_4$ solution.

2. Boil the mixture, then cool and acidify with dilute H_2SO_4.

3. Add a drop of $FeCl_3$ solution. Formation of a Prussian blue colour/precipitate confirms the presence of nitrogen.

III. Test for Sulphur

1. Take a portion of the extract and add a few drops of freshly prepared sodium nitroprusside solution. A violet/purple colouration indicates the presence of sulphur.

2. Alternatively, acidify another portion with acetic acid and add lead acŋte solution; a black precipitate of PbS confirms sulphur.

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IV. Test for Halogens

1. Take a portion of the extract, boil it with dilute HNO_3 (to decompose any cyanide/sulphide present), then cool.

2. Add $AgNO_3$ solution: a white precipitate soluble in NH_4OH indicates chlorine; a pale yellow precipitate, partially soluble in NH_4OH, indicates bromine; a yellow precipitate insoluble in NH_4OH indicates iodine.

Observation Table

Observation Table: Detection of extra elements

Sl. No.	Element Tested	Test / Reagent used	Observation	Inference
1				
2				
3				

Calculation

No numerical calculation is involved in this qualitative experiment.

Result: The given organic compound contains _____ (nitrogen/sulphur/halogen, as confirmed by the tests).

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the purpose of sodium fusion in Lassaigne's test?

Ans. To convert covalently bound nitrogen, sulphur, and halogens present in an organic compound into ionic sodium salts (NaCN, Na_2S, NaX) that can be easily detected by simple inorganic tests.

Q2. Why must the fusion tube be red hot before plunging into water?

Ans. Strong heating ensures complete fusion/decomposition of the organic compound with sodium, maximising the conversion of covalent N, S, and halogens into their ionic forms.

Q3. What happens if both nitrogen and sulphur are present together in the compound?

Ans. Sodium thiocyanate (NaSCN) is formed instead of separate NaCN and Na_2S, which gives a blood-red colour with $FeCl_3$ instead of (or in addition to) the Prussian blue test.

Q4. Why is the extract boiled with dilute HNO_3 before the halogen test?

Ans. To decompose any NaCN or Na_2S present, which would otherwise interfere by giving precipitates with $AgNO_3$ and causing a false-positive halogen test.

Q5. How do you distinguish between chloride, bromide, and iodide in the $AgNO_3$ test?

Ans. AgCl is white and completely soluble in NH_4OH; AgBr is pale yellow and partially soluble in NH_4OH; AgI is yellow and insoluble in NH_4OH.

Teacher's Signature: _____

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EXPERIMENT - 17

Aim

To detect the functional group present in the given organic compound by chemical tests.

Apparatus Required

Test tubes, test tube stand, dropper, burner, water bath.

Chemicals Required

Given organic compound, $NaHCO_3$ solution, neutral $FeCl_3$, Tollens' reagent, Fehling's solution A and B, 2,4-DNP reagent, Schiff's reagent, dilute HCl, sodium nitrite.

Theory

1. Different functional groups present in organic compounds give characteristic colour changes or precipitate formation with specific reagents, which helps in their identification.

2. Carboxylic acids liberate CO_2 (brisk effervescence) with $NaHCO_3$ solution.

3. Phenols give a characteristic violet/blue/green colouration with neutral $FeCl_3$ solution.

4. Aldehydes and ketones both give an orange-yellow precipitate with 2,4-dinitrophenylhydrazine (2,4-DNP, Brady's reagent); aldehydes can be distinguished from ketones since aldehydes reduce Tollens' reagent (silver mirror) and Fehling's solution (brick-red precipitate of Cu_2O), while ketones (except alpha-hydroxy ketones) do not.

5. Primary aromatic amines give a characteristic dye (orange-red/yellow) on diazotisation with $NaNO_2$/HCl followed by coupling with bŋ-naphthol (azo dye test).

Procedure

I. Test for Carboxylic acid

1. Take 1 mL of the given compound (or its solution) in a test tube and add a pinch of solid $NaHCO_3$.

2. Brisk effervescence of a colourless, odourless gas (CO_2, turns lime water milky) confirms a carboxylic acid group.

II. Test for Phenol

1. Dissolve a small amount of the given compound in water/alcohol and add 2-3 drops of neutral $FeCl_3$ solution.

2. Appearance of a violet, blue, or green colouration confirms the presence of a phenolic group.

III. Test for Aldehyde/Ketone (2,4-DNP test)

1. Add a few drops of the given compound to 1 mL of 2,4-DNP reagent.

2. Formation of a yellow/orange precipitate confirms the presence of a carbonyl (aldehyde or ketone) group.

IV. Distinguishing test (Tollens'/Fehling's test)

1. Add a few drops of the compound to freshly prepared Tollens' reagent in a clean test tube and warm gently on a water bath.

2. A bright silver mirror on the walls of the test tube indicates an aldehyde; no change indicates a ketone.

3. Alternatively, add the compound to Fehling's solution A and B (mixed) and heat; a brick-red precipitate of Cu_2O indicates an aldehyde.

V. Test for Amine

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1. Dissolve the compound in dilute HCl, cool in ice, and add cold $NaNO_2$ solution dropwise (diazotisation).
2. Add this diazotised solution to an alkaline bŋ-naphthol solution.
3. Formation of an orange-red dye confirms a primary aromatic amine.

Observation Table

Observation Table: Detection of functional group

Sl. No.	Test performed	Observation	Inference (Functional group)
1			
2			
3			
4			
5			

Calculation

No numerical calculation is involved in this qualitative experiment.

Result: The given organic compound contains the _____ functional group.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. How would you distinguish between an aldehyde and a ketone chemically?

Ans. Aldehydes reduce Tollens' reagent (silver mirror test) and Fehling's solution (brick-red Cu_2O precipitate) on warming, while ketones (except alpha-hydroxy ketones) generally do not give these tests.

Q2. Why does $NaHCO_3$ test confirm a carboxylic acid specifically (and not a phenol)?

Ans. Carboxylic acids are acidic enough to liberate CO_2 from the weak base $NaHCO_3$ (brisk effervescence), whereas phenols are too weakly acidic to react with $NaHCO_3$.

Q3. What colour does phenol give with neutral $FeCl_3$?

Ans. A violet, blue, or green colouration, depending on the specific phenolic compound.

Q4. What is Tollens' reagent, and what happens in a positive test?

Ans. Tollens' reagent is an ammoniacal solution of silver nitrate; aldehydes reduce Ag+ to mŋllic Ag, which deposits as a bright, mirror-like coating on the test tube wall.

Q5. What is the chemical basis of the 2,4-DNP test?

Ans. 2,4-dinitrophenylhydrazine reacts with the carbonyl group (C=O) of aldehydes and ketones to form a yellow/orange precipitate of the corresponding hydrazone.

Teacher's Signature: _____

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EXPERIMENT - 18

Aim

To purify the given liquid organic compound (mixture) by simple distillation.

Apparatus Required

Distillation flask, condenser, thermometer, receiver flask, tripod stand, burner/heating mantle, clamp stand, wire gauze, anti-bumping chips.

Chemicals Required

Given liquid organic compound (e.g., mixture of water and a volatile liquid with a single, well-separated boiling point), anti-bumping granules.

Theory

1. Simple distillation is a technique used to separate and purify a liquid from a non-volatile solid or from another liquid having a sufficiently different (more than about 25 °C) boiling point.

2. It is based on the difference in boiling points — the liquid is heated until it vaporises; the vapour is then cooled and condensed back to liquid form in a different vessel (receiver), leaving behind the non-volatile/higher boiling impurities.

3. The temperature recorded on the thermometer during the process corresponds to the boiling point of the liquid being distilled.

4. Anti-bumping chips/granules are added to ensure smooth, uniform boiling and prevent sudden bumping of the liquid.

Procedure

Procedure

1. Set up the distillation apparatus: clamp the distillation flask (containing the given liquid and a few anti-bumping chips) over a burner/heating mantle, fit it with a thermometer (bulb level with the side arm) and connect the side arm to a condenser.

2. Connect the lower end of the condenser to a receiver flask to collect the distillate.

3. Heat the distillation flask slowly and steadily.

4. Note the temperature at which the liquid begins to boil and vapour starts passing into the condenser.

5. Continue heating at a steady rate and collect the distillate in the receiver, noting the constant temperature (boiling point) at which most of the liquid distils over.

6. Stop heating when about 90% of the liquid has distilled over or when the temperature starts to rise sharply (indicating the compound is exhausted).

7. Record the volume and boiling point of the purified distillate collected.

Observation Table

Observation Table: Simple distillation

Sl. No.	Volume/Weight taken	Temperature reading (°C)	Volume/Weight of distillate collected	Observation
1				
2				

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Sl. No.	Volume/Weight taken	Temperature reading (°C)	Volume/Weight of distillate collected	Observation
3				

Calculation

Volume of liquid taken for distillation = _____ mL

Volume of pure distillate collected = _____ mL

Boiling point of the distillate (as observed) = _____ °C

Percentage recovery = (Volume of distillate / Volume of liquid taken) x 100 = _____ %

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. On what principle is simple distillation based?

Ans. On the difference in boiling points of the components of a mixture — the more volatile component vaporises first and is condensed and collected separately.

Q2. Why are anti-bumping chips added?

Ans. To provide nucleation sites for the formation of vapour bubbles, ensuring smooth, even boiling and preventing sudden, violent bumping of the liquid.

Q3. Why is the thermometer bulb positioned level with the side arm of the distillation flask?

Ans. This ensures the thermometer measures the temperature of the vapour actually passing into the condenser, which corresponds to the true boiling point of the distilling liquid.

Q4. When is simple distillation not suitable for purification?

Ans. When the liquids to be separated have very close boiling points (within about 25 °C), or when the compound decomposes at or before its boiling point.

Q5. What is the function of the condenser?

Ans. It cools the hot vapour, converting it back into liquid (condensate), which is then collected in the receiver flask.

Teacher's Signature: _____

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EXPERIMENT - 19

Aim

To purify/separate the given organic compound (which is steam-volatile and immiscible with water) by steam distillation.

Apparatus Required

Round-bottom flask, steam generator (or direct steam source), condenser, receiver (round-bottom/conical flask), delivery tube, clamp stand, burner.

Chemicals Required

Given organic compound (steam-volatile, water-immiscible, e.g., containing essential oil/aniline-type compound), water, separating funnel, anhydrous sodium sulphate (drying agent).

Theory

1. Steam distillation is used to separate/purify organic compounds that are volatile in steam, insoluble (or only sparingly soluble) in water, and may decompose if distilled alone at their normal boiling point.

2. The compound is distilled along with steam; since the compound is immiscible with water, according to Dalton's law, the mixture boils at a temperature lower than the boiling point of either pure component, allowing the compound to distil over at a temperature below 100 °C without decomposition.

3. The distillate, a mixture of water and the organic compound, separates into two layers on standing, which can be separated using a separating funnel.

4. The organic layer is then dried using an anhydrous drying agent to obtain the pure compound.

Procedure

Procedure

1. Take the given organic compound/mixture in a round-bottom flask fitted with a delivery tube connected to a steam source (steam generator) on one side.

2. Connect the other outlet of the flask to a condenser, with the lower end of the condenser leading to a receiver flask.

3. Pass steam into the flask containing the compound while gently heating the flask to prevent excessive condensation inside it.

4. Continue passing steam until no more oily droplets (organic compound) are observed in the distillate and only clear water distils over.

5. Collect the distillate (a mixture of water and the organic compound) in the receiver.

6. Transfer the distillate to a separating funnel, allow the layers to separate, and run off the lower/upper organic layer as applicable.

7. Dry the separated organic layer over anhydrous sodium sulphate and note its volume/weight.

Observation Table

Observation Table: Steam distillation

Sl. No.	Volume/Weight taken	Temperature reading (°C)	Volume/Weight of distillate collected	Observation
1				
2				

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Sl. No.	Volume/Weight taken	Temperature reading (°C)	Volume/Weight of distillate collected	Observation
3				

Calculation

Weight/volume of impure compound taken = _____

Weight/volume of pure compound recovered after steam distillation = _____

Percentage recovery = (Amount recovered / Amount taken) x 100 = _____ %

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. On what principle does steam distillation work?

Ans. When two immiscible liquids are heated together, the mixture boils at a temperature lower than the boiling point of either pure liquid, since the total vapour pressure is the sum of the individual vapour pressures (Dalton's law of partial pressures).

Q2. What type of compounds are purified by steam distillation?

Ans. Compounds that are steam-volatile, immiscible (or only sparingly miscible) with water, and which might decompose if distilled alone at their normal (high) boiling point.

Q3. Why does the compound distil over at a temperature below 100 °C?

Ans. Because the combined vapour pressure of water and the organic compound reaches atmospheric pressure (causing boiling) at a temperature lower than the boiling point of either component alone.

Q4. How is the organic layer separated from the water layer in the distillate?

Ans. Using a separating funnel, exploiting the immiscibility and density difference between the organic compound and water.

Q5. Why is the organic layer dried over anhydrous sodium sulphate after separation?

Ans. To remove any trace/dissolved water remaining in the organic layer, giving a purer, dry product.

Teacher's Signature: _____

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EXPERIMENT - 20

Aim

To determine the coefficient of viscosity of the given liquid relative to water using Ostwald's viscometer.

Apparatus Required

Ostwald's viscometer, stop-watch, pipette, thermometer, clamp stand, specific gravity bottle.

Chemicals Required

Given liquid (experimental liquid), distilled water (reference liquid).

Theory

1. Viscosity is the internal resistance offered by a liquid to its own flow.

2. Ostwald's viscometer measures viscosity by comparing the time taken by equal volumes of the experimental liquid and a reference liquid (water) to flow through the same capillary tube under their own gravity, from a fixed upper mark to a fixed lower mark.

3. According to Poiseuille's equation, for a given viscometer, the coefficient of viscosity is related to the density and flow time of the liquid: $\eta_1/\eta_2 = (\rho_1 \times t_1) / (\rho_2 \times t_2)$

4. Since water is used as the reference liquid, the viscosity of the given liquid can be determined relative to that of water, given the densities and flow times of both liquids.

Procedure

Procedure

1. Clean and dry the Ostwald's viscometer thoroughly.

2. Pipette out a fixed volume of distilled water into the viscometer through the wider limb.

3. Suck the liquid up the capillary limb above the upper mark using a suction bulb, then allow it to flow down freely.

4. Note the time taken (using a stop-watch) for the liquid meniscus to pass from the upper mark to the lower mark; repeat to get concordant readings.

5. Empty, clean, and dry the viscometer; repeat the same procedure with the given experimental liquid, using the same volume.

6. Determine the densities of water and the given liquid using a specific gravity bottle.

7. Maintain the same temperature for both sets of measurements (using a constant temperature bath if available).

Observation Table

Observation Table: Flow times and densities

Sl. No.	Liquid	Density, ρ (g/mL)	Flow time, t (sec) (Trial 1/2/3)	Mean flow time (sec)
1				
2				

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Calculation

η(liquid) / η(water) = [ρ(liquid) x t(liquid)] / [ρ(water) x t(water)]

η(liquid) = η(water) x [ρ(liquid) x t(liquid)] / [ρ(water) x t(water)]

(Coefficient of viscosity of water at room temperature, η(water), taken from standard tables)

Coefficient of viscosity of given liquid, η(liquid) = _____ poise/centipoise

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is viscosity?

Ans. Viscosity is the internal frictional resistance offered by adjacent layers of a liquid to relative flow/motion.

Q2. What is the basic principle of Ostwald's viscometer?

Ans. It compares the time taken for a fixed volume of the experimental liquid and a reference liquid (water) to flow through the same capillary under gravity, relating this to their relative viscosities and densities (Poiseuille's equation).

Q3. Why must the viscometer be perfectly vertical and at constant temperature during the experiment?

Ans. Flow time depends on the hydrostatic head (hence verticality) and viscosity is highly temperature-sensitive, so both must be controlled for accurate, reproducible results.

Q4. Why is water used as the reference liquid?

Ans. Because the viscosity of water at various temperatures is accurately known from standard tables, allowing the unknown liquid's viscosity to be calculated relative to it.

Q5. What are the units of viscosity?

Ans. Poise (CGS unit) or Pascal-second (SI unit); viscosity is commonly reported in centipoise (cP) for common liquids.

Teacher's Signature: _____

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EXPERIMENT - 21

Aim

To determine the surface tension of the given liquid relative to water using a Stalagmometer (drop number method).

Apparatus Required

Stalagmometer, clamp stand, beaker, specific gravity bottle, rubber tubing with pinch cock.

Chemicals Required

Given liquid (experimental liquid), distilled water (reference liquid).

Theory

1. Surface tension is the property of a liquid surface which makes it behave like a stretched elastic membrane, tending to minimise its surface area.

2. The Stalagmometer method (drop-weight/drop-number method) is based on the principle that the weight of a drop of liquid falling from a capillary tube is directly related to the surface tension of the liquid: the formation of a drop is a balance between the surface tension force holding the drop and the gravitational force pulling it down.

3. By counting the number of drops formed by a fixed volume of liquid flowing from the stalagmometer, and comparing it with the number of drops of water (reference liquid) for the same volume, the surface tension of the liquid can be calculated relative to water.

4. Formula: $\gamma_1/\gamma_2 = (\rho_1 \times n_2) / (\rho_2 \times n_1)$, where n_1 and n_2 are the number of drops of liquid and water respectively.

Procedure

Procedure

1. Clean and dry the stalagmometer thoroughly.

2. Suck distilled water into the stalagmometer above the upper mark using the attached rubber tubing.

3. Allow the water to flow down drop by drop and count the number of drops formed between the upper and lower marks.

4. Repeat for concordant readings.

5. Empty, rinse, and dry the stalagmometer; repeat the same procedure with the given experimental liquid, counting the number of drops for the same volume (between the same two marks).

6. Determine the densities of water and the given liquid using a specific gravity bottle.

7. Ensure both measurements are taken at the same room temperature.

Observation Table

Observation Table: Number of drops and densities

Sl. No.	Liquid	Density, ρ (g/mL)	No. of drops, n (Trial 1/2/3)	Mean no. of drops
1				
2				

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Calculation

γ(liquid) / γ(water) = [ρ(liquid) x n(water)] / [ρ(water) x n(liquid)]

γ(liquid) = γ(water) x [ρ(liquid) x n(water)] / [ρ(water) x n(liquid)]

(Surface tension of water at room temperature, γ(water), taken from standard tables, in dynes/cm)

Surface tension of given liquid, γ(liquid) = _____ dynes/cm

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is surface tension?

Ans. Surface tension is the force acting per unit length along the surface of a liquid, tending to minimise the surface area, caused by unbalanced cohesive forces on surface molecules.

Q2. What is the principle of the drop-number method?

Ans. The size (and hence number) of drops formed from a fixed volume of liquid flowing from a capillary depends on the liquid's surface tension and density; comparing the drop count with that of water gives the relative surface tension.

Q3. Why should the stalagmometer tip be free from grease or dirt?

Ans. Contamination changes the wetting behaviour and drop formation at the tip, leading to inconsistent, inaccurate drop counts.

Q4. Why does surface tension decrease with an increase in temperature?

Ans. Increased temperature increases the kinetic energy of molecules, weakening the intermolecular cohesive forces responsible for surface tension.

Q5. In what units is surface tension expressed?

Ans. Dynes per centimetre (CGS) or Newtons per metre (SI).

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EXPERIMENT - 22

Aim

To determine the pH of the given solutions using a digital pH meter and to study the variation of pH with concentration/dilution.

Apparatus Required

Digital pH meter with glass electrode, beakers, glass rod, wash bottle, buffer solutions (pH 4, 7, 9) for calibration.

Chemicals Required

Given solutions (e.g., HCl/CH_3COOH/NaOH of varying concentrations), standard buffer solutions, distilled water.

Theory

1. pH is a measure of the hydrogen ion concentration in a solution, defined as pH = -log10[H^+].

2. A pH meter works on the principle of measuring the potential difference developed between a glass electrode (sensitive to H^+ ion activity) and a reference electrode (usually combined as a combination electrode) when dipped in the test solution; this potential is converted and displayed directly as the pH value.

3. The pH meter must be calibrated using standard buffer solutions of known pH (e.g., pH 4.0 and pH 7.0) before use, to ensure accurate readings.

4. The pH of strong acids/bases is close to the theoretical value calculated from their concentration, while weak acids/bases show a pH higher/lower than expected due to partial ionisation.

Procedure

Procedure

1. Switch on the pH meter and allow it to warm up/stabilise.

2. Calibrate the pH meter using standard buffer solutions of pH 4.0 and pH 7.0 (or pH 9.0), washing the electrode with distilled water between each buffer.

3. Rinse the glass electrode thoroughly with distilled water and blot gently (do not rub) before each measurement.

4. Dip the electrode into the given solution, allow the reading to stabilise, and note the pH value displayed.

5. Repeat the measurement for each of the given solutions (and for a series of dilutions, if required), rinsing the electrode with distilled water between each measurement.

6. Record the pH of each solution and plot a graph of pH versus concentration/dilution if required.

Observation Table

Observation Table: pH of given solutions

Sl. No.	Solution	Concentration	Observed pH
1			
2			
3			
4			

Calculation

Theoretical pH of strong acid solution of concentration C = -log10(C)

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Theoretical pH of strong base solution of concentration C = 14 + log10(C)

Observed pH (from pH meter) = _____

Comparison of observed and theoretical pH values confirms whether the given solute is a strong or weak electrolyte.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What does pH measure?

Ans. pH measures the hydrogen ion concentration (activity) in a solution; pH = -log10[H^+].

Q2. Why must a pH meter be calibrated before use?

Ans. To correct for any drift or variation in the glass electrode's response, ensuring the readings obtained for unknown solutions are accurate, by comparing against solutions of precisely known pH (buffers).

Q3. Why does a weak acid show a higher pH than a strong acid of the same concentration?

Ans. A weak acid ionises only partially, producing fewer H^+ ions in solution compared to a strong acid of the same concentration, which ionises almost completely.

Q4. Why should the glass electrode be rinsed with distilled water between measurements?

Ans. To avoid cross-contamination between solutions, which could alter the reading of the next solution tested.

Q5. What is the pH range of a neutral solution at 25 °C?

Ans. pH = 7.

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EXPERIMENT - 23

Aim

To determine the strength of the given HCl (strong acid) solution by conductometric titration against standard NaOH solution.

Apparatus Required

Conductivity meter with conductivity cell, burette, beaker, magnetic stirrer, burette stand.

Chemicals Required

Given HCl solution, standard NaOH solution, distilled water.

Theory

1. Conductometric titration is based on measuring the change in electrical conductance of a solution as a titrant is added, rather than relying on a chemical indicator.

2. During the titration of a strong acid (HCl) with a strong base (NaOH), as NaOH is added, highly mobile H^+ ions are progressively replaced by less mobile Na^+ ions, so the conductance decreases up to the equivalence point.

3. Beyond the equivalence point, further addition of NaOH introduces free OH^- ions (highly mobile) into the solution, causing the conductance to increase sharply.

4. A graph of conductance versus volume of NaOH added shows two straight lines intersecting at a minimum, which corresponds to the equivalence point of the titration.

Procedure

Procedure

1. Pipette out a fixed volume (e.g., 20 mL) of the given HCl solution into a beaker and dilute if necessary.

2. Dip the conductivity cell into the beaker and note the initial conductance reading.

3. Add standard NaOH solution from the burette in small increments (e.g., 0.5 or 1 mL at a time), stirring well after each addition.

4. Note the conductance after each addition, continuing well beyond the expected equivalence point.

5. Plot a graph of conductance (Y-axis) against volume of NaOH added (X-axis).

6. Extrapolate the two linear portions of the graph (decreasing and increasing) to find the point of intersection, which gives the volume of NaOH at the equivalence point.

Observation Table

Observation Table: Conductance vs. Volume of NaOH added

Sl. No.	Volume of NaOH added (mL)	Conductance reading (mho/S)
1		
2		
3		
4		
5		
6		
7		
8		

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Calculation

From the graph, volume of NaOH at the equivalence point (intersection of the two lines) = V(eq) mL

N_1V_1 (HCl) = N_2V_2 (NaOH)

N(HCl) x V(HCl) = N(NaOH) x V(eq)

N(HCl) = [N(NaOH) x V(eq)] / V(HCl)

Strength of HCl = N(HCl) x Equivalent weight of HCl (36.5) g/L

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is the advantage of conductometric titration over a normal indicator-based titration?

Ans. It can be used for coloured or turbid solutions where indicator colour changes are hard to observe, and it gives a more objective, graphical determination of the equivalence point.

Q2. Why does conductance decrease initially when NaOH is added to HCl?

Ans. The highly mobile H^+ ions are progressively replaced by the much less mobile Na^+ ions as the neutralisation reaction proceeds, reducing overall conductance.

Q3. Why does conductance increase sharply after the equivalence point?

Ans. Beyond the equivalence point, excess NaOH adds free, highly mobile OH^- ions to the solution, which sharply increases the conductance.

Q4. How is the equivalence point determined graphically?

Ans. By plotting conductance against volume of titrant added; the two linear portions of the graph are extrapolated, and their point of intersection gives the equivalence point.

Q5. Why is the solution not stirred magnetically before each reading skipped?

Ans. Stirring (not skipping) is essential — thorough mixing after each addition ensures a uniform solution before each conductance reading is recorded.

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EXPERIMENT - 24

Aim

To verify Beer-Lambert's law for the given coloured solution using a photoelectric colorimeter and to determine the unknown concentration of a sample solution.

Apparatus Required

Photoelectric colorimeter, cuvettes/test tubes, volumetric flasks, pipette, beakers.

Chemicals Required

Given coloured solution (e.g., $CuSO_4$/$KMnO_4$ solution), distilled water.

Theory

1. Beer-Lambert's law states that the absorbance (optical density) of a solution is directly proportional to its concentration and the path length of light through the solution, at a fixed wavelength: A = epsilon x c x l, where A is absorbance, epsilon is the molar extinction coefficient, c is concentration, and l is path length.

2. A colorimeter measures the absorbance (or percentage transmittance) of a coloured solution at a selected wavelength (using a suitable colour filter) by comparing the intensity of light transmitted through the solution with that through a blank (solvent).

3. By preparing a series of solutions of known concentration and measuring their absorbance, a calibration (standard) graph of absorbance versus concentration is obtained, which should be a straight line passing through the origin, verifying Beer-Lambert's law.

4. The concentration of an unknown solution of the same substance can then be determined by measuring its absorbance and reading the corresponding concentration from the calibration graph.

Procedure

Procedure

1. Prepare a series of standard solutions of the given coloured substance of known, increasing concentrations (e.g., by diluting a stock solution) using volumetric flasks.

2. Switch on the colorimeter and select the appropriate wavelength/filter for the colour of the solution (complementary to the colour of the solution).

3. Set the absorbance to zero using a blank (distilled water) in the cuvette.

4. Measure and record the absorbance of each standard solution, from the lowest to the highest concentration.

5. Plot a graph of absorbance (Y-axis) against concentration (X-axis); verify that it is a straight line passing through the origin.

6. Measure the absorbance of the given unknown solution under identical conditions.

7. Determine the concentration of the unknown solution from the calibration graph.

Observation Table

Observation Table: Absorbance of standard solutions

Sl. No.	Concentration (mol/L or g/L)	Absorbance / Optical Density
1		
2		
3		

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Sl. No.	Concentration (mol/L or g/L)	Absorbance / Optical Density
4		
5		

Calculation

From the calibration graph (absorbance vs. concentration), the graph is linear and passes through the origin, verifying Beer-Lambert's law.

Absorbance of unknown solution = A(unknown) = _____

Corresponding concentration from graph = C(unknown) = _____ mol/L (or g/L)

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. State Beer-Lambert's law.

Ans. The absorbance of a solution is directly proportional to its concentration and the path length of light through it: A = epsilon x c x l.

Q2. Why should the calibration graph pass through the origin?

Ans. At zero concentration, there is no absorbing species, so absorbance should theoretically be zero, giving a straight line through the origin if Beer-Lambert's law is obeyed.

Q3. Why is a specific colour filter/wavelength chosen for a particular solution?

Ans. The filter of the complementary colour to the solution allows maximum absorption of light by the solution, giving maximum sensitivity and accuracy in absorbance measurement.

Q4. What is meant by a 'blank' in colorimetry?

Ans. A blank is the pure solvent (without the coloured solute), used to set the absorbance/transmittance reference (zero absorbance / 100% transmittance) before measuring sample solutions.

Q5. What are the possible reasons for deviation from Beer-Lambert's law at high concentrations?

Ans. At high concentrations, solute-solute interactions, changes in refractive index, or association/dissociation equilibria can cause the absorbance-concentration relationship to deviate from linearity.

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EXPERIMENT - 25

Aim

To determine the critical solution temperature (CST) of the phenol-water system and to study the effect of impurity (NaCl) on the CST.

Apparatus Required

Boiling tube, thermometer, water bath, beaker, burner, wire gauze, stirrer, clamp stand.

Chemicals Required

Phenol, distilled water, sodium chloride (NaCl).

Theory

1. Phenol and water are partially miscible liquids at room temperature, forming two distinct layers; on heating, their mutual solubility increases, and above a particular temperature they become completely miscible in all proportions.

2. This temperature, above which the two liquids become completely miscible for all compositions, is called the Critical Solution Temperature (CST) or upper consolute temperature.

3. A mixture of phenol and water, when heated, becomes a single clear homogeneous phase at the temperature at which it just turns clear (disappearance of turbidity); on cooling, turbidity reappears at the same temperature.

4. By preparing mixtures of phenol and water of different compositions and noting the temperature at which each becomes clear (and again turbid on cooling), a graph of temperature versus composition is plotted; the maximum point on this curve gives the CST and the corresponding critical composition.

5. Addition of a soluble impurity (like NaCl) generally raises the CST, while addition of a partially soluble impurity may lower it.

Procedure

Procedure

1. Prepare a series of mixtures of phenol and water of different known compositions (percentage by weight) in separate boiling tubes.

2. Take one such mixture in a boiling tube fitted with a thermometer and stirrer, and place it in a water bath.

3. Heat the mixture slowly with continuous stirring, noting the temperature at which the turbid mixture just becomes clear (miscibility temperature).

4. Allow the mixture to cool slowly with stirring and note the temperature at which turbidity just reappears.

5. Take the mean of the two temperatures (heating and cooling) as the miscibility temperature for that composition.

6. Repeat the above steps for all the prepared compositions of phenol-water mixtures.

7. Plot a graph of miscibility temperature (Y-axis) against the percentage composition of phenol (X-axis); the maximum point of the smooth curve obtained gives the CST and the critical composition.

8. Repeat the experiment by adding a small, fixed amount of NaCl to each mixture and determine the new CST to study the effect of the impurity.

Observation Table

Observation Table: Miscibility temperature vs. composition of phenol-water mixtures

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Sl. No.	% Composition of Phenol	Temp. on heating (°C)	Temp. on cooling (°C)	Mean Temp. (°C)
1				
2				
3				
4				
5				
6				

Calculation

Plot the graph of Miscibility temperature (Y-axis) vs. % composition of phenol (X-axis).

From the maximum point of the curve:

Critical Solution Temperature (CST) of phenol-water system = _____ °C

Critical composition (% phenol) at CST = _____ %

CST of phenol-water-NaCl system (with impurity) = _____ °C

Conclusion: Addition of NaCl (soluble impurity) raises/lowers the CST of the phenol-water system.

Result: The above practical was performed successfully and the required value/observation was determined (see calculation above).

Viva-Voce Questions

Q1. What is Critical Solution Temperature (CST)?

Ans. It is the temperature above which two partially miscible liquids become completely miscible in all proportions (also called the upper consolute temperature).

Q2. Why does turbidity appear/disappear at the CST?

Ans. Below the CST, the two liquids are only partially miscible, so a mixture forms two separate phases (turbid); above CST, they are fully miscible into one homogeneous (clear) phase.

Q3. What is the critical composition?

Ans. It is the specific percentage composition of the two-component mixture at which the miscibility (consolute) temperature is at its maximum — i.e., the composition corresponding to the peak of the temperature-composition curve.

Q4. How does adding NaOH (a soluble impurity) affect the CST of phenol-water?

Ans. It generally raises the CST, since the dissolved impurity alters the mutual solubility behaviour of phenol and water.

Q5. Why is the mean of heating and cooling temperatures taken for each composition?

Ans. To minimise experimental error due to thermal lag (delay in heat transfer), since the observed clearing temperature on heating and the turbidity-reappearance temperature on cooling may differ slightly.

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