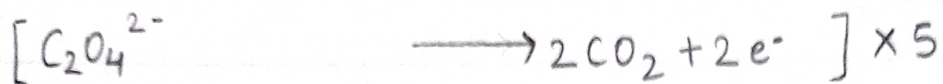


Index

S.No.	Experiment Name	Page No.	Experiment Date	Submission Date	Teacher's Sign./Remarks
1.	To estimate the strength of oxalic acid by titrating it against KMnO_4	1-3	14/12/21		
2.	To estimate the strength of Mohr's salt by titrating it against KMnO_4	4-6	21/12/21		
3.	To estimate the strength of given CuSO_4 solution iodometrically by titrating it against $\text{Na}_2\text{S}_2\text{O}_3$	7-10	28/12/21		
4.	Estimation of Fe^{2+} by titrating it with $\text{K}_2\text{Cr}_2\text{O}_7$ using internal indicator Diphenylamine	11-12	4/1/22		
5.	Estimation of number of water of crystallisation in Mohr's salt by titrating it with KMnO_4	13-14	11/1/22		
6.	a) To recrystallise the given sample of organic acid ^{compound} using water as a solvent.	15-19	18/1/22		
	b) To recrystallise the given sample of organic compound using alcohol as a solvent				
7.	To determine melting point of organic compound using Kjeldahl method & electrical heating apparatus	20-22	1/2/22		
8.	To identify and separate the compounds of a given mixture of two amino acids by ascending paper chromatography & measure R_f value	23-26	8/2/22		



Overall reaction:



EXPERIMENT-1

AIM: To estimate the strength of given Oxalic acid by titrating it with KMnO_4 solution (approx 0.05N)

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. Volumetric Analysis: As we are going to measure volume of 0.05N KMnO_4 solution and applying it to determine the strength of oxalic acid.
2. Redox titration: KMnO_4 undergoes reduction while oxalic acid undergoes oxidation.
3. Reduction & oxidation takes place simultaneously.
4. KMnO_4 is an oxidising agent.
5. Oxalic acid is reducing agent.

KMnO_4 behaves as a good oxidising agent in acidic medium. For this we need to add dil. H_2SO_4 .

Warm the oxalic acid solution.

Mn^{2+} behaves as an auto catalyst.

KMnO_4 behaves as a self indicator.

Procedure:

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

1. Weigh accurately 0.315g of oxalic acid required for preparing 0.05N solution.

2. Transfer to a 100 ml standard volumetric flask.

3. Add 10 ml of dilute H_2SO_4 and after that distilled water to make up the volume.

II. Standardisation of given $KMnO_4$ solution

1. Pipette out standard oxalic acid in a conical flask and then warm the content.

2. Titrate with the $KMnO_4$ solution taken in the burette until the color change to pink.

3. Repeat the titration to get three concordant readings.

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

1. Pipette out given oxalic acid solution in a conical flask (10 ml).

2. Titrate with the $KMnO_4$ solution ^{taken} in the burette until the color change to pink.

3. Repeat the titration to get three concordant readings.

Observation:

1. Record observation of titration of standard oxalic acid in observation table - 1 and observation of titration of given oxalic acid in observation table - 2.

$$N = \frac{\text{wt. of oxalic acid}}{\text{equivalent wt. of oxalic acid}} \times \frac{1000}{\text{volume}}$$

$$0.05 = \frac{\text{wt. of oxalic acid}}{63} \times \frac{1000}{100}$$

$$\text{wt. of oxalic acid} = \frac{0.05 \times 63}{10} = 0.315 \text{ gm}$$

OBSERVATION TABLE-1

Sl No.	Volume of oxalic acid (ml)	Initial Burette readings	Final Burette readings	Vol. of KMnO_4 consumed
1.	10			
2.	10			
3.	10			

OBSERVATION TABLE-2

Sl No.	Volume of oxalic acid (ml)	Initial Burette readings	Final Burette readings	Vol. of KMnO_4 consumed
1.	10			
2.	10			
3.	10			

Calculation:

$$N_1 V_1 (\text{oxalic acid}) = N_2 V_2 (\text{KMnO}_4)$$

$$0.05 \times 10 (\text{oxalic acid}) = N_{(\text{KMnO}_4)} \times \underline{\hspace{2cm}}$$

$$N_{(\text{KMnO}_4)} = \underline{\hspace{2cm}}$$

$$N_1 V_1 (\text{oxalic acid}) = N_2 V_2 (\text{KMnO}_4)$$

$$N_{(\text{oxalic acid})} \times 10 = \underline{\hspace{2cm}} \times \underline{\hspace{2cm}}$$

$$N_{(\text{oxalic acid})} = \underline{\hspace{2cm}}$$

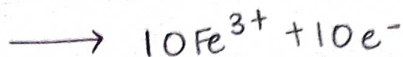
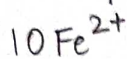
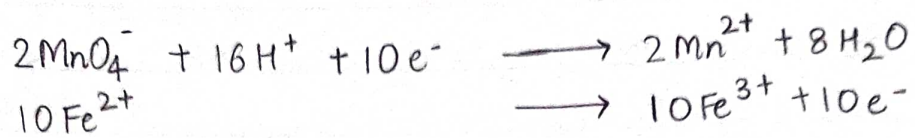
$$\begin{aligned} \text{Strength of oxalic acid} &= N_{(\text{oxalic acid})} \times \text{Equivalent weight} \\ &= \underline{\hspace{2cm}} \times 63 \\ &= \underline{\hspace{2cm}} \text{ g/l} \end{aligned}$$

Result:

The strength of given oxalic acid solution was found to be _____ g/l.

Precautions:

1. Clean all apparatus with distilled water before the starting of experiment.
2. Use dilute H_2SO_4 for acidifying the $KMnO_4$.
3. Do not use rubber cork burette as it can be attacked by $KMnO_4$.
4. Take accurate readings once it reaches the end point and don't go with average readings.
5. The strength of unknown solution should be taken upto two decimal places only.



Overall reaction:



EXPERIMENT-2

AIM: To estimate the strength of given Mohr's salt by titrating it against KMnO_4 (approx 0.05N)

Apparatus required: Burette, Pipette, conical flask, burner, Test tube, weighing balance, funnel, Burette stand, measuring cylinder, measuring flask, watch glass

Chemicals required: Mohr's salt, dilute sulphuric acid, distilled water

Theory:

1. Volumetric Analysis: As we are going to measure volume of 0.05N KMnO_4 and applying it to determine the strength of Mohr's salt.
2. Redox titration: KMnO_4 undergoes reduction while Mohr's salt undergoes oxidation.
3. Reduction & oxidation takes place simultaneously.
4. KMnO_4 is an oxidising agent
5. Mohr's salt is reducing agent.

KMnO_4 behaves as a good oxidising agent in acidic medium. For this we need to add dil. H_2SO_4 .

Mn^{2+} behaves as auto catalyst.

KMnO_4 behaves as a self indicator.

$$N = \frac{\text{wt. of Mohr's salt}}{\text{Equivalent wt. of Mohr's salt}} \times \frac{1000}{\text{volume}}$$

$$0.05 = \frac{\text{wt. of Mohr's salt}}{392} \times \frac{1000}{100}$$

$$\text{wt. of Mohr's salt} = \frac{0.05 \times 392}{10} = 1.96 \text{ gm}$$

OBSERVATION TABLE - 1

Sl No.	Volume of Mohr's salt (ml)	Initial Burette readings	Final Burette readings	Vol. of KMnO_4 used
1	10			
2	10			
3	10			

OBSERVATION TABLE - 2

Sl No.	Volume of Mohr's salt (ml)	Initial Burette readings	Final Burette readings	Vol. of KMnO_4 used
1	10			
2	10			
3	10			

Procedure:

I. Preparation of standard Mohr's salt, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.05N) solution

1. weigh accurately 1.96 gm of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ required for preparing 0.05N solution.
2. Transfer to a 100 ml standard volumetric flask.
3. Add 10 ml of dilute H_2SO_4 and after that distilled water to make up the volume.

II. Standardisation of given KMnO_4 solution:

1. Pipette out standard $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 0.05N solution in a conical flask (10 ml).
2. Add 10 ml of dilute H_2SO_4 to prevent hydrolysis of Mohr's salt.
3. Titrate with the KMnO_4 solution taken in the burette until the colour changes to pink.
4. Repeat the titration to get three concordant readings.

III. Titration of given $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ solution with given KMnO_4 solution:

1. Pipette out given Mohr's salt solution in a conical flask (10 ml).
2. Add 10 ml of dilute H_2SO_4 .
3. Titrate with KMnO_4 solution taken in the burette until the colour changes to pink.
4. Repeat the titration to get three concordant readings.

Observation: Record readings of titration of standard & given Mohr's salt solution in observation table 1 & 2 respectively.

Calculation:

$$N_1 V_1 (\text{Mohr's Salt}) = N_2 V_2 (\text{KMnO}_4)$$

$$0.05 \times 10 (\text{Mohr's salt}) = N_2 \times \text{---} (\text{KMnO}_4)$$

$$N_{\text{KMnO}_4} = \text{---}$$

$$N_1 V_1 (\text{Mohr's salt}) = N_2 V_2 (\text{KMnO}_4)$$

$$N (\text{Mohr's salt}) \times 10 = \text{---} \times \text{---}$$

$$N (\text{Mohr's salt}) = \text{---}$$

$$\text{Strength of Mohr's salt} = N (\text{Mohr's salt}) \times \text{Equivalent weight}$$

$$= \text{---} \times 392$$

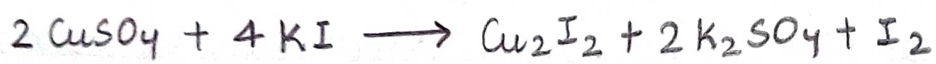
$$= \text{---} \text{ g/l}$$

Result:

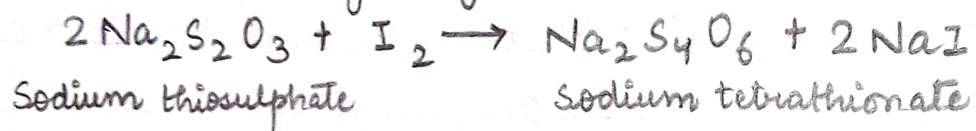
The strength of given Mohr's salt was found to be _____ g/l

Precautions:

1. Clean all apparatus with distilled water before the starting of experiment.
2. Use dil. H_2SO_4 for acidifying the $KMnO_4$.
3. Do not use rubber cork burette as it can be attacked by $KMnO_4$.
4. Take accurate readings once it reaches end point and don't go with average readings.
5. The strength of the unknown solution should be taken upto two decimal places only.



Iodide is getting oxidised to iodine



Expt No. _____

Page No. 7EXPERIMENT-3

AIM: To estimate the strength of given CuSO_4 solution iodometrically by titrating it against $\text{Na}_2\text{S}_2\text{O}_3$ (approx 0.05N)

Apparatus required: Burette, Pipette, Conical flask, Burner, Test tube, weighing balance, funnel, Burette stand, measuring cylinder, measuring flask, watch glass

Chemicals required: Copper sulphate, potassium iodide, sodium thiosulphate, Na_2CO_3 , acetic acid, NH_4CNS and starch indicator

Theory:

1. This is a redox titration
2. This is an iodometric titration.
3. CuSO_4 is oxidising agent
4. $\text{Na}_2\text{S}_2\text{O}_3$ is reducing agent.
5. Starch is an adsorption indicator.

Iodometry - It involves indirect titration where an iodide is made to react to form iodine.

Iodimetry - It involves direct titration wherein iodine is used as a titrant.

6. In the experiment, starch is added at the end of titration and not in beginning, because at the start of titration, concentration of iodine in solution is high and starch readily reacts with iodine

Teacher's Signature :

$$N = \frac{\text{wt. of } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}}{\text{equivalent wt. of } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}} \times \frac{1000}{\text{volume}}$$

$$0.05 = \frac{\text{wt. of } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}}{249.6} \times \frac{1000}{100}$$

$$\begin{aligned} \text{wt. of } \text{CuSO}_4 \cdot 5\text{H}_2\text{O} &= \frac{0.05 \times 249.6}{10} \\ &= 1.248 \text{ g} \end{aligned}$$

and forms starch-iodine complex and thus sodium thiosulphate will not be able to react with CuSO_4 molecules and thus diffused end point may be observed.

7. NH_4CNS is added to avoid premature end point.

8. Na_2CO_3 is added because sodium thiosulphate is unstable under acidic pH conditions; Na_2CO_3 being alkaline in nature makes the solution basic, so that $\text{Na}_2\text{S}_2\text{O}_3$ can react easily.

9. Acetic acid is used to balance pH as excess addition of Na_2CO_3 may cause solution to be highly basic and starch will not be able to react in high basicity, so to maintain the pH, few drops ^{of acetic acid} ~~are~~ are added.

Standardisation of Na_2SO_3

Preparation of standard solution of CuSO_4 .

1. Weigh a clean and dry watch glass.
2. Weigh 1.248g of CuSO_4 in the watch glass.
3. Transfer the weighed ~~to~~ CuSO_4 from the watch glass into the volumetric flask of 100 ml.
4. Add few drops of dil. H_2SO_4 .
5. Add distilled water to make up the volume in volumetric flask.

Procedure for titration

1. Pipette out 10 ml of CuSO_4 solution in a conical flask.

OBSERVATION TABLE - 1

Sl. No.	Volume of CuSO_4 solution (ml)	Initial Burette readings	Final Burette readings	Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ consumed
1.	10			
2.	10			
3.	10			

2. Add a pinch of Na_2CO_3 (solid) and a few drops of acetic acid.
3. Add a pinch of KI solid.
4. Cover the mouth of the conical flask by a watch glass as KI is volatile in nature.
5. Put the flask in dark for 5-7 minutes because iodine is weakly solution and easily lost out of solution because it is volatile. Keeping the solution in dark slows down the loss of iodine from water.
6. The colour of the content in conical flask changes to brown indicating release of I_2 . Add $\text{Na}_2\text{S}_2\text{O}_3$ from burette drop wise.
7. The brown colour changes to yellow (showing that most of iodine is escaped and now starch can be added). Add few drops of starch indicator.
8. Add a pinch of NH_4CNS . The colour of solution will turn blue.
9. Keep on adding $\text{Na}_2\text{S}_2\text{O}_3$ till the blue colour disappears. Note down the readings. This is end point.
10. Repeat above procedure 2-3 times to get concordant readings.

Titration of standard CuSO_4 Vs $\text{Na}_2\text{S}_2\text{O}_3$
Record observation in observation table - 1.

OBSERVATION TABLE - 2

Sl No.	Volume of CuSO_4 solution (ml)	Initial Burette readings	Final Burette readings	Volume of $\text{Na}_2\text{S}_2\text{O}_3$ consumed
1.	10			
2.	10			
3.	10			

Calculation:

1. Normality of $\text{Na}_2\text{S}_2\text{O}_3$ solution using:

$$N_1 V_1 (\text{Na}_2\text{S}_2\text{O}_3) = N_2 V_2 (\text{standard copper sulphate solution})$$

$$0 N_1 \text{ — } (\text{Na}_2\text{S}_2\text{O}_3) = 0.05 \times 10 (\text{standard copper sulphate solution})$$

$$N (\text{Na}_2\text{S}_2\text{O}_3) = \text{ — }$$

2. Normality of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in unknown solution.

$$N_3 V_3 (\text{CuSO}_4 \cdot 5\text{H}_2\text{O}) = N_1 V_1 (\text{Na}_2\text{S}_2\text{O}_3)$$

$$N (\text{CuSO}_4 \cdot 5\text{H}_2\text{O}) \times 10 = \text{ — } \times \text{ — }$$

$$N (\text{CuSO}_4 \cdot 5\text{H}_2\text{O}) = \text{ — }$$

$$\begin{aligned} \text{Strength of } \text{CuSO}_4 \cdot 5\text{H}_2\text{O} &= N (\text{CuSO}_4 \cdot 5\text{H}_2\text{O}) \times \text{Equivalent weight} \\ &= \text{ — } \times 249.6 \\ &= \text{ — } \text{ g/l} \end{aligned}$$

Expt No. _____

Page No. 10

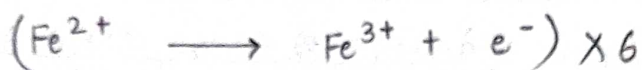
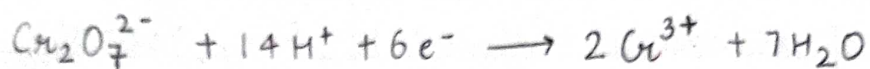
Titration of given CuSO_4 Vs $\text{Na}_2\text{S}_2\text{O}_3$

Record observations in observation table - 2.

Precautions:

1. Clean all apparatus with distilled water before the experiment starts.
2. The strength of unknown solution should be taken upto two decimal places only.
3. Take accurate readings once it reaches the end point and don't go with average readings.

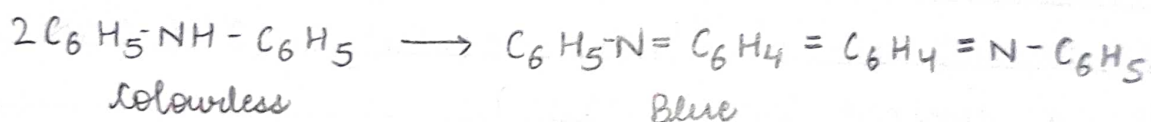
Teacher's Signature :



Overall equation:



Indicator is oxidized by excess dichromate:



Calculation of normality of standard $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$

$$N = \frac{\text{wt. of Mohr's Salt}}{\text{Equivalent wt. of Mohr's Salt}} \times \frac{1000}{\text{volume}}$$

$$\frac{1}{40} = \frac{\text{wt. of Mohr's Salt}}{392.14} \times \frac{1000}{100}$$

$$\text{wt. of Mohr's Salt} = \frac{1 \times 392.14}{40 \times 10} = 0.98035\text{g}$$

EXPERIMENT-4

Aim: Estimation of Fe^{2+} by titrating it with $\text{K}_2\text{Cr}_2\text{O}_7$ using internal indicator Diphenylamine

Apparatus required: Burette, Pipette, Conical Flask, Test tube, weighing balance, funnel, Burette stand, measuring cylinder, volumetric flask, watch glass.

Chemicals required: Mohr's salt, Potassium dichromate, diphenylamine, dilute sulphuric acid, H_3PO_4 , distilled water

Theory:

1. This is a redox titration.
2. $\text{K}_2\text{Cr}_2\text{O}_7$ is an oxidizing agent.
3. Mohr's salt is a reducing agent.
4. Excess dichromate oxidize diphenylamine.

Procedure:

1. Preparation of standard Mohr's salt, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (N/40) solution:
 1. weigh accurately 0.98035 g of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ required for preparing N/40 solution.
 2. Transfer to a 100 ml standard volumetric flask.
 3. Add 10 ml of dilute H_2SO_4 and after that distilled water to make up the volume.

OBSERVATION TABLE - 1

Sl No.	Volume of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ solution (ml)	Initial Burette readings	Final Burette readings	Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed (ml)

OBSERVATION TABLE - 2

Sl No.	Volume of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ solution (ml)	Initial Burette readings	Final Burette readings	Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution consumed (ml)

Calculations:

1. Calculation of normality of $\text{K}_2\text{Cr}_2\text{O}_7$ solution using:

$$N_1 V_1 (\text{K}_2\text{Cr}_2\text{O}_7) = N_2 V_2 (\text{std. } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O})$$

$$N_1 (\text{K}_2\text{Cr}_2\text{O}_7) = \frac{1}{40} \times 10 (\text{std. } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O})$$

$$N_1 = \underline{\hspace{2cm}}$$

2. Calculation of normality of Fe^{3+} in unknown solution using:

$$N_3 V_3 (\text{unknown } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) = N_1 V_1 (\text{K}_2\text{Cr}_2\text{O}_7)$$

$$\underline{\hspace{2cm}} \times 10 (\text{unknown } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) = \underline{\hspace{2cm}} \times \underline{\hspace{2cm}} (\text{K}_2\text{Cr}_2\text{O}_7)$$

$$N_3 = \underline{\hspace{2cm}}$$

3. Strength of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O} = N_3 \times \text{eq. wt of } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$
 $= \underline{\hspace{2cm}} \times 392.14 = \underline{\hspace{2cm}}$

- II. Standardization of given $K_2Cr_2O_7$ ($\approx N/40$) solution:
1. Pipette out standard $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ ($N/40$) solution in a conical flask (10 ml).
 2. Add 10 ml of dilute H_2SO_4 , 2 ml of dilute H_3PO_4 and 2-3 drops of diphenylamine.
 3. Titrate with the $K_2Cr_2O_7$ solution taken in the burette until the color change to blue.
 4. Repeat the titration to get three concordant readings.

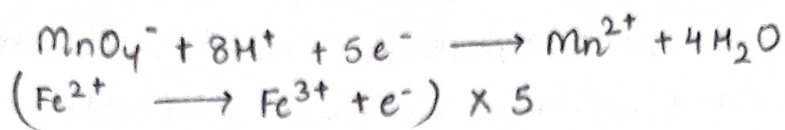
- III. Titration of given $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution with given $K_2Cr_2O_7$ solution:
1. Pipette out given $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ solution in a conical flask.
 2. Add 10 ml of dilute H_2SO_4 , 2 ml dilute H_3PO_4 and 2-3 drops of diphenylamine.
 3. Titrate with the $K_2Cr_2O_7$ solution taken in the burette until the color change to blue.
 4. Repeat the titration to get three concordant readings.

Observation:

Record the result of titration of standard Mohr's salt solution in observation table-1 and given Mohr's salt solution in observation table-2.

Precautions:

1. Clean all apparatus before starting experiment with distilled water.
2. The strength of unknown solution be taken upto 2 decimal places.
3. Take accurate readings.



Overall reaction:



Calculation of Normality of standard $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$

$$N = \frac{\text{wt. of Mohr's salt}}{\text{Equivalent wt. of Mohr's salt}} \times \frac{1000}{\text{volume}}$$

$$\frac{1}{40} = \frac{\text{wt. of Mohr's salt}}{392.14} \times \frac{1000}{100}$$

$$\text{wt. of Mohr's salt} = \frac{1 \times 392.14}{40 \times 10} = 0.98035\text{g}$$

Expt No. 5

Page No. 13

EXPERIMENT-5

AIM: Estimation of number of water of crystallization in Mohr's salt by titrating it with KMnO_4 .

Apparatus required: Burette, Pipette, conical Flask, Test tube, weighing balance, funnel, Burette stand, measuring cylinder, volumetric flask, watch glass.

Chemicals required: Mohr's salt, KMnO_4 , dilute H_2SO_4 , distilled water.

Theory:

1. It is a redox titration.
2. KMnO_4 is strong oxidizing agent.
3. Mohr's salt is a reducing agent.
4. KMnO_4 behaves as a good oxidizing agent in acidic medium.
5. Mn^{2+} behaves as auto catalyst.
6. KMnO_4 acts as a self-indicator.
7. Colour change is colourless to pink.

Procedure:

1. Preparation of standard Mohr's salt, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$ (N/40) solution
1. weigh accurately 0.98035 g of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$ required for preparing N/40 solution.

Teacher's Signature :

OBSERVATION TABLE - 1

Sl. No.	Volume of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$ solution (ml)	Burette readings		Volume of KMnO_4 solution consumed (ml)
		Initial	Final	
1.	10			
2.	10			
3.	10			

OBSERVATION TABLE - 2

Sl. No.	Volume of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$ solution (ml)	Burette readings		Volume of KMnO_4 solution consumed (ml)
		Initial	Final	
1.	10			
2.	10			
3.	10			

Calculation:

1. Calculation of normality of KMnO_4 solution using:

$$N_1 V_1 (\text{KMnO}_4) = N_2 V_2 (\text{std. } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O})$$

$$N_1 \times \text{---} (\text{KMnO}_4) = \frac{1}{40} \times 10 (\text{std. } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}) \Rightarrow N_1 = \text{---}$$

2. Calculation of normality of given $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$

$$N_3 V_3 (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O} = N_1 V_1 (\text{KMnO}_4)$$

$$N_3 \times 10 (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O} = \text{---} \times \text{---} (\text{KMnO}_4) \Rightarrow N_3 = \text{---}$$

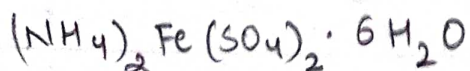
$$\begin{aligned} \text{Strength of } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O} &= N_3 \times \text{eq. wt. of } (\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O} \\ &= \text{---} \times \text{---} = \text{---} \text{ L} \end{aligned}$$

Strength of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$ is given

$$2 \text{ g/L} = N_3 \times (284 + 18x)$$

From here, we can calculate the value of x

It will come out to be 6



2. Transfer to a 100ml standard volumetric flask.
3. Add 10 ml of dilute H_2SO_4 and after that distilled water to make up the volume.

II. Standardisation of given $KMnO_4$ ($\approx N/40$) solution:

1. Pipette out standard $(NH_4)_2Fe(SO_4)_2 \cdot xH_2O$ ($N/40$) solution in a conical flask (10 ml).
2. Add 10 ml of dilute H_2SO_4 .
3. Titrate with the $KMnO_4$ solution taken in the burette until the color changes to pink.
4. Repeat the titration to get three concordant readings.

III. Titration of given $(NH_4)_2Fe(SO_4)_2 \cdot xH_2O$ solution with given $KMnO_4$ solution:

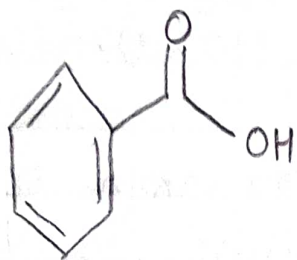
1. Pipette out given $(NH_4)_2Fe(SO_4)_2 \cdot xH_2O$ solution in a conical flask.
2. Add 10 ml of dilute H_2SO_4 .
3. Titrate with the $KMnO_4$ solution taken in the burette until the color changes to pink.
4. Repeat the titration to get three concordant readings.

Observation:

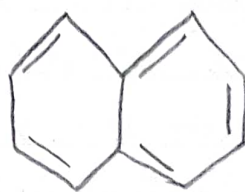
Record the result of titration of standard and given Mohr's salt in observation table 1 & 2 respectively.

Precautions:

1. Clean all apparatus before starting experiment with water.
2. The strength of unknown solution be taken upto 2 decimal places.
3. Take accurate readings.



Benzoic acid



Naphthalene

Experiment - 6

- AIM: (a) To recrystallise the given sample of organic compound using water as a solvent.
- (b) To recrystallise the given sample of organic compound using alcohol as a solvent.

Theory:

Recrystallisation is the primary method for purifying solid organic compounds obtained from natural sources or from reaction mixtures almost always contain impurities.

The impurities may include some combination of insoluble, soluble or coloured impurities. To obtain a pure compound these impurities must be removed. Each is removed in a separate step in the recrystallisation process.

To understand the recrystallization process, solubility behaviour must first be considered. It is often stated that "like dissolve like". More correctly it should be stated that compounds having similar structures, behaviour or features will be soluble in one another. Some obvious structural features that may affect solubility include polarity and ability to form hydrogen bond. For eg- a compound having just few carbons and an alcohol functional group would be expected to be soluble in solvent that has few carbons and an alcohol functional group or in some other polar solvents, and to be less soluble in non-polar solvents.

Conversely, an alkane be expected to show the opposite solubility behaviour. In most cases, though it is not as simple as this. If for example a compound has lots of carbon and hydrogen and just one alcohol group, the solubility will be dominated more by alkyl part of molecule than by alcohol part, and the compound will show a solubility more like that of an alkane. For known compounds it is useful to consider the structure of compound when choosing a recrystallisation solvent. Usually, however, the structure of compound may not be known so the solvent must be chosen by carrying out solubility test.

Recrystallization is a very important technique often employed for the purification of solids. It is based on the fact that all organic solids are more soluble in hot than in cold solvents, so that the solid dissolved on heating can be obtained back on cooling.

The following steps are performed for purification by crystallisation.

- (a) Selection of a solvent or mixture of solvents.
- (b) Dissolution of the solute in required amount of the hot solvent to obtain a saturated solution.
- (c) Decolorisation (charcoalisation) for removal of coloured impurities.
- (d) Crystallisation, filtration and drying of the solid.

a) Selection of a solvent

The solvent chosen for recrystallisation should be such that the solid to be purified is insoluble or nearly insoluble in the cold and fairly soluble in the hot solvent. The boiling point of the solvent should be lower than the melting point of the solid so that it can be removed during the drying of the solid.

Procedure

For selection of the solvent: Take ~50 mg of the solid in a clean dry tube and add about 0.5-1 ml of the solvent, stir well. If the solid is soluble at room temperature, the solvent cannot be used for crystallisation. If the solid is insoluble, heat the test tube in a hot water bath or directly on a flame. If the solid dissolves in the boiling solvent and crystals appear on cooling, the solvent is suitable for recrystallisation.

If a pure (single) solvent is found unsuitable, a mixture of solvents may be used, the two solvents should be miscible with each other eg. water-ethanol or ethylacetate-petroleum ether.

Dissolve the compound in the hot solvent (A) in which it is freely soluble and add drop-wise the solvent (B) in which the compound is insoluble till a turbid solution is obtained. Add drop-wise the solvent (A) to get back a clear solution and allow the solution to cool undisturbed for the separation of

crystals.

b) **Dissolution of the solute in the required amount of solvent:** After selecting a suitable solvent for crystallization, take the impure solid (~200mg) in a clean test tube or a conical flask (never use a beaker for recrystallisation). Add a few millimetres of the solvent and heat the solution on a water bath with constant stirring or swirling (do not heat inflammable liquids directly on a flame). If the solid does not dissolve, add a few more millimetres of the solvent and heat again (do not add excess of solvent). If near the end of the dissolution process, it appears that an additional amount of the solvent is not dissolving any more of the solid, then this small amount of undissolved solid is likely to be an insoluble impurity. Do not add any more solvent but filter the hot solution through a fluted filter paper and allow the filtrate to stand undisturbed for crystallisation.

c) **Decolourisation:** If the resultant solution obtained above is highly coloured, add a small amount of charcoal, boil the solution for some more time (the coloured impurities are adsorbed on the surface of charcoal), filter the hot solution through a fluted filter paper and allow the filtrate to cool for the separation of crystals.

d) Crystallisation

Do not cool the solution obtained above rapidly, allow it to slowly cool to room temperature. If no crystals form, cool the solution in an icebath. Scratch the sides of the tube with a glass rod, just above the surface of the solution to initiate crystallisation or add a very small amount of solid compound to induce crystal formation.

If an oil separates on cooling: (1) either scratch it against the sides of the tube (or flask) to solidify, (2) add a few drops of the solvent, heat, allow to solidify, or (3) add a few crystals of the solute to initiate crystal formation.

e) Filtration and drying etc.: Filter the crystals by vacuum filtration and collect the mother liquor in a clean dry flask or a filter tube. Cool the mother liquor in an ice bath or concentrate and cool to obtain the second crop of the crystals. Do not mix the two crops until their purity has been confirmed by melting point determination. Transfer the crystals from the filter funnel on to a filter paper and air dry or dry in an air oven maintained at a much lower temperature than the melting point of the solid or by keeping it in vacuum desiccator.

Experiment - 7

Aim: To determine melting point of organic compound using Kjeldahl method and electrical heating apparatus

Theory

Melting point - The temperature at which solid and liquid states are in equilibrium

Because of the limitations of methods we use, we determine range of melting point as highly sensitive apparatus are not there.

We determine melting point because:

- Melting point are used to characterise organic and inorganic crystalline compounds.
- Purity check - Using depression in melting point on increasing impurity, we can check purity of a substance

Procedure:Electrical heating Apparatus

1. Fill a capillary tube (sealed at one end) with sample whose melting point is to be determined by thrusting the open end into sample several times and tapping the solid end on the table till tube contain $\frac{1}{4}$ th - $\frac{1}{5}$ th level of powdered sample.
2. Place the thermometer in the packet for thermometer and filled capillary tube in small packet meant for it.

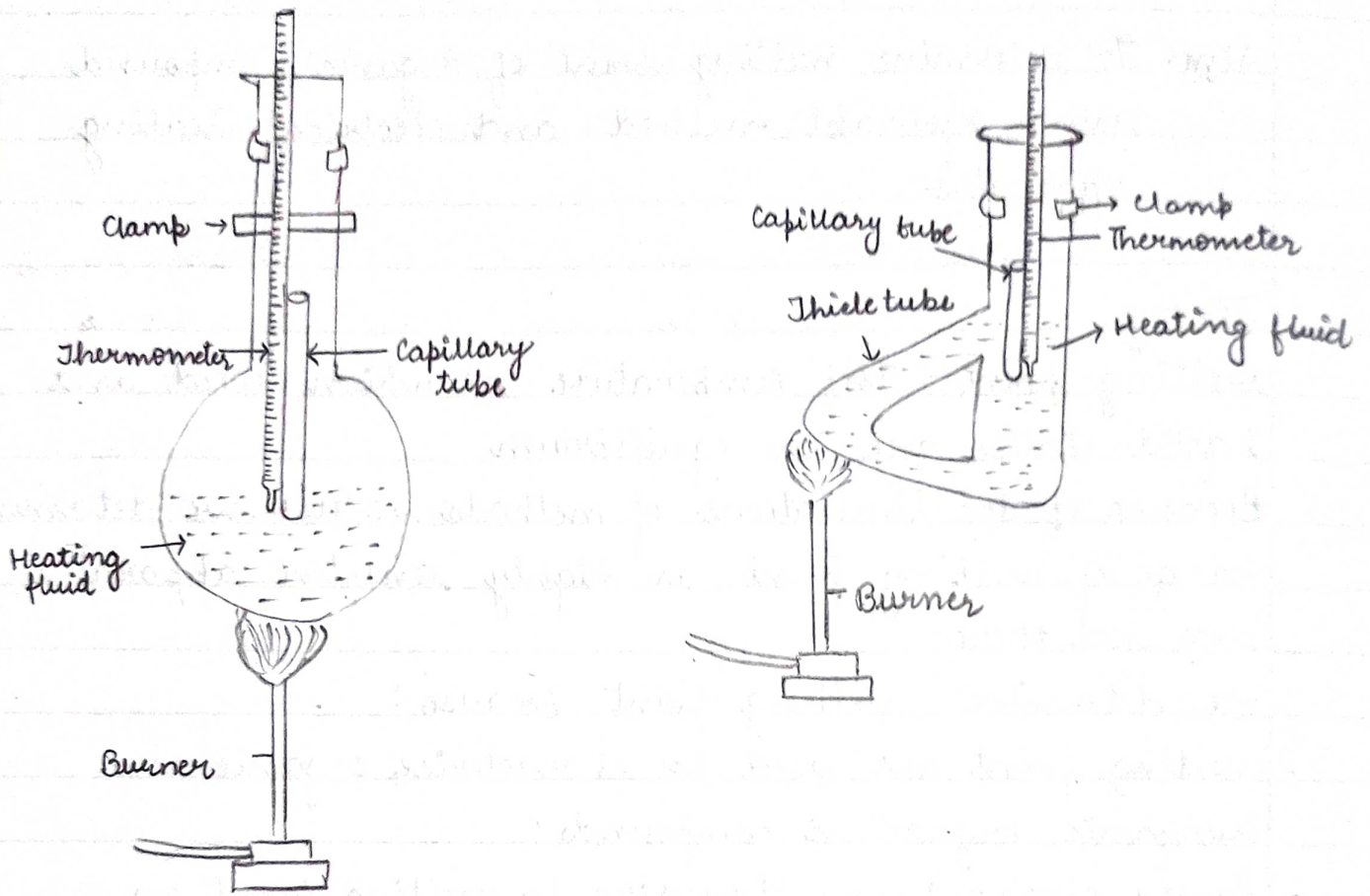


Fig: Kjeldahl Apparatus

3. The capillary tube is observed through it.

Kjeldahl method

The most common method to heat small amount of substance in a capillary tube attached to a thermometer which is immersed in a suitable liquid bath to determine the temperature at which it melts.

- Take a small thin capillary tube, which is made of soda glass (weak glass). Close one end of the tube by heating. On heating, it becomes red hot and automatically the capillary tube closes from one end.
- Now fill the powdered organic compound in the capillary tube.
- Fill the organic compound to 0.5-1 cm in capillary tube. About $\frac{1}{4}$ - $\frac{1}{5}$ th of the level is filled with organic compound.
- This capillary must be filled upto 1 cm and organic compound should be closely packed to avoid any air gaps between (by tapping on table).
- This capillary tube is attached to thermometer by applying 1 drop of H_2SO_4 or paraffin oil on outer wall of capillary tube [H_2SO_4 and paraffin oil has high temperature stability. These compounds provide equal heating to both capillary tube and thermometer).
- We can also use a rubber or thread to attach capillary tube with thermometer.

- The thermometer and capillary tube is inserted into the Kjeldahl flask in which H_2SO_4 bath is used.
- Flask is heated with low flame by holding the burner in hand rotating it over flask.
- We have to observe state of organic compound.
- Check the temperature at which the organic compound start to convert into liquid. Note this temperature.
- And after it we have to slow heat (increase $1^\circ C/min$) the compound till this ^{organic compound} melts into liquid completely.
- This gives the melting point range.
- We must try to $2^\circ C$ deviation in our answer, not more than that.

Result:

The melting point of given organic compound is determined.

Precautions:

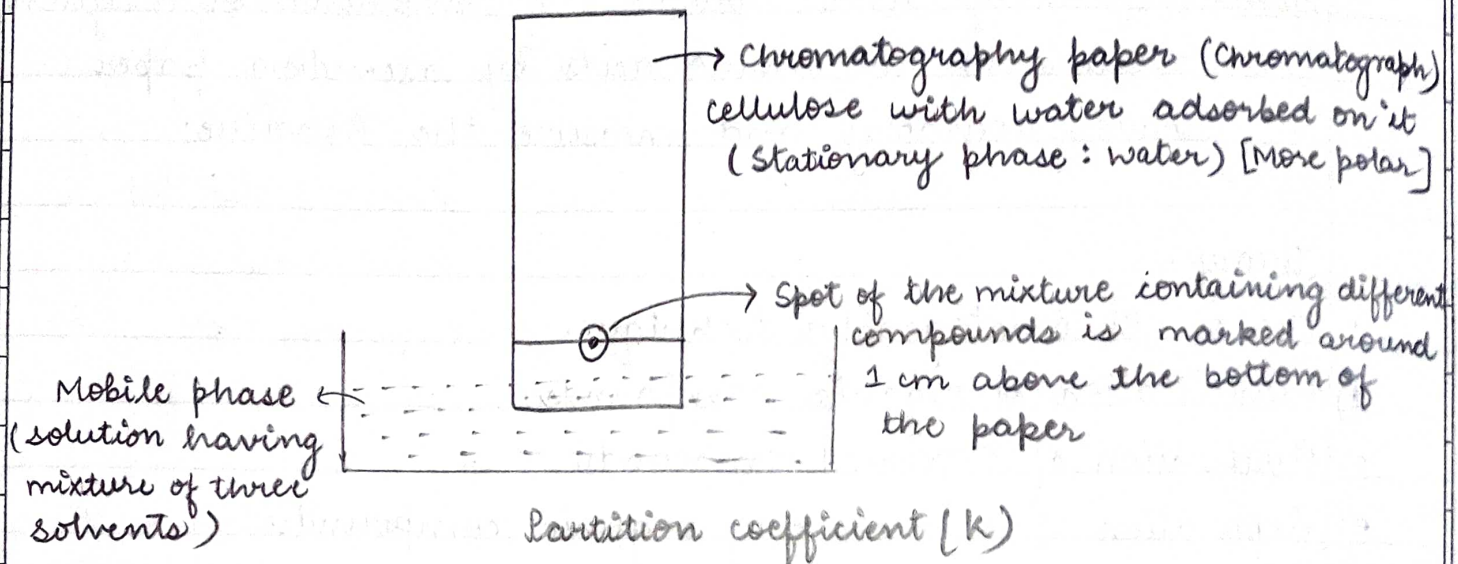
- * Thermometer ^{& capillary tube} must not touch walls of Kjeldahl flask.
- * Burner must be used carefully.
- * H_2SO_4 must be used carefully.

Experiment-8

Aim: To identify and separate the compounds of a given mixture of two amino acids by ascending paper chromatography and measure the R_f value.

Theory:

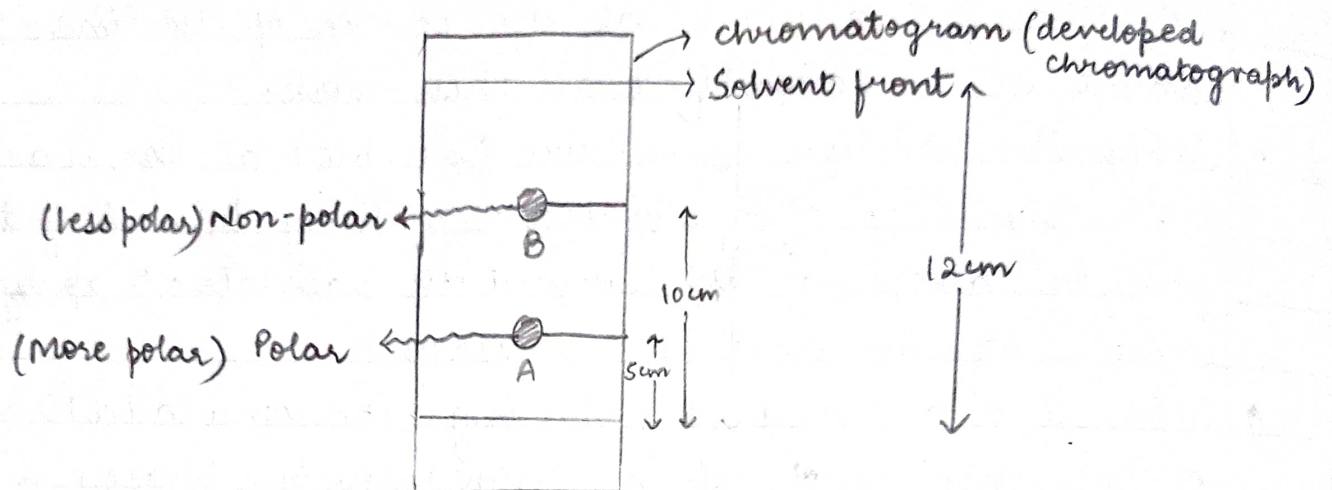
- Use of Chromatography technique
 - 1) Identification of organic compounds
 - 2) Purification of organic compounds
 - 3) Separation of a mixture of organic compounds
- Basic principle behind chromatography experiment
The components of the mixture is distributed (partitioned) along these two phases -
 - 1) Stationary phase (fixed phase)
 - 2) Mobile phase
- Different types of chromatography
 - 1) Adsorption Chromatography
Stationary phase (SP) = solid
Mobile phase (MP) = liquid
For eg- Thin layer chromatography (TLC), Column Chromatography
 - 2) Partition Chromatography
Stationary phase (SP) = liquid
Mobile phase (MP) = solid
For eg- Paper Chromatography



BAW
 ↓
 Butanol Acetic Acid Water
 (less polar)

Procedure

1. Cut the chromatographic paper strip into the required size.
2. Draw a line with pencil at about 1cm from one end and put a mark at the centre of the line. The sample is to be applied at this mark.
3. Apply the solution of mixture (sample) at the mark with the help of a pointed capillary tube. Dry the part by allowing the solvent to evaporate. Reapply the sample at the mark if necessary.
4. Take a clean and dry boiling tube and add 10 ml of the mixture of solvent (BAW) [mobile phase] in it. Suspend the spotted and dried paper strip in the boiling tube containing mobile phase very carefully with the upper end pinned to the cork and the lower end touching the BAW solution.
[Note :- The paper strip should be vertical in the boiling tube and it should not touch the walls of boiling tube.]
5. The spot should always be above the mobile phase. Allow the mobile phase to rise along the paper and wait till the mobile phase (solvent front) reaches near the upper end of the paper.
6. Remove the paper strip carefully from the boiling tube and mark the solvent front with the help of a pencil.
7. Dry the strip until the acetic acid odour from the strip disappears.
8. Treat the strip with a visualizing agent by a spraying



Limitless quantity

$$R_f(A) = \frac{5 \text{ cm}}{12 \text{ cm}} = 0.416$$

$$R_f(B) = \frac{10 \text{ cm}}{12 \text{ cm}} = 0.833$$

$$R_f(B) > R_f(A)$$

More polar (polar) compound will have less R_f value than less polar (non polar)

Expt No. _____

bottle. Heat the strip in the oven until the coloured zones of amino acids are seen. Encircle the coloured zones and mark the centre of each zone.

B. Calculate the R_f value of each component of the mixture.

For making mixture of amino acids - Take 25 mg of each amino acid and dissolve it in 50 ml of water to make 50 ml solution.

Partition Coefficient (K)

$K = \frac{C_s}{C_m}$ → concentration of compound in stationary phase
 C_m → concentration of compound in mobile phase

suppose A and B are two components and $C_{s(A)} > C_{s(B)}$

$$K_A > K_B$$

Polar compound will have higher K in paper chromatography. Polar compounds will have more affinity towards stationary phase. It will dissolve more in it leading to higher concentration of compound in stationary phase.

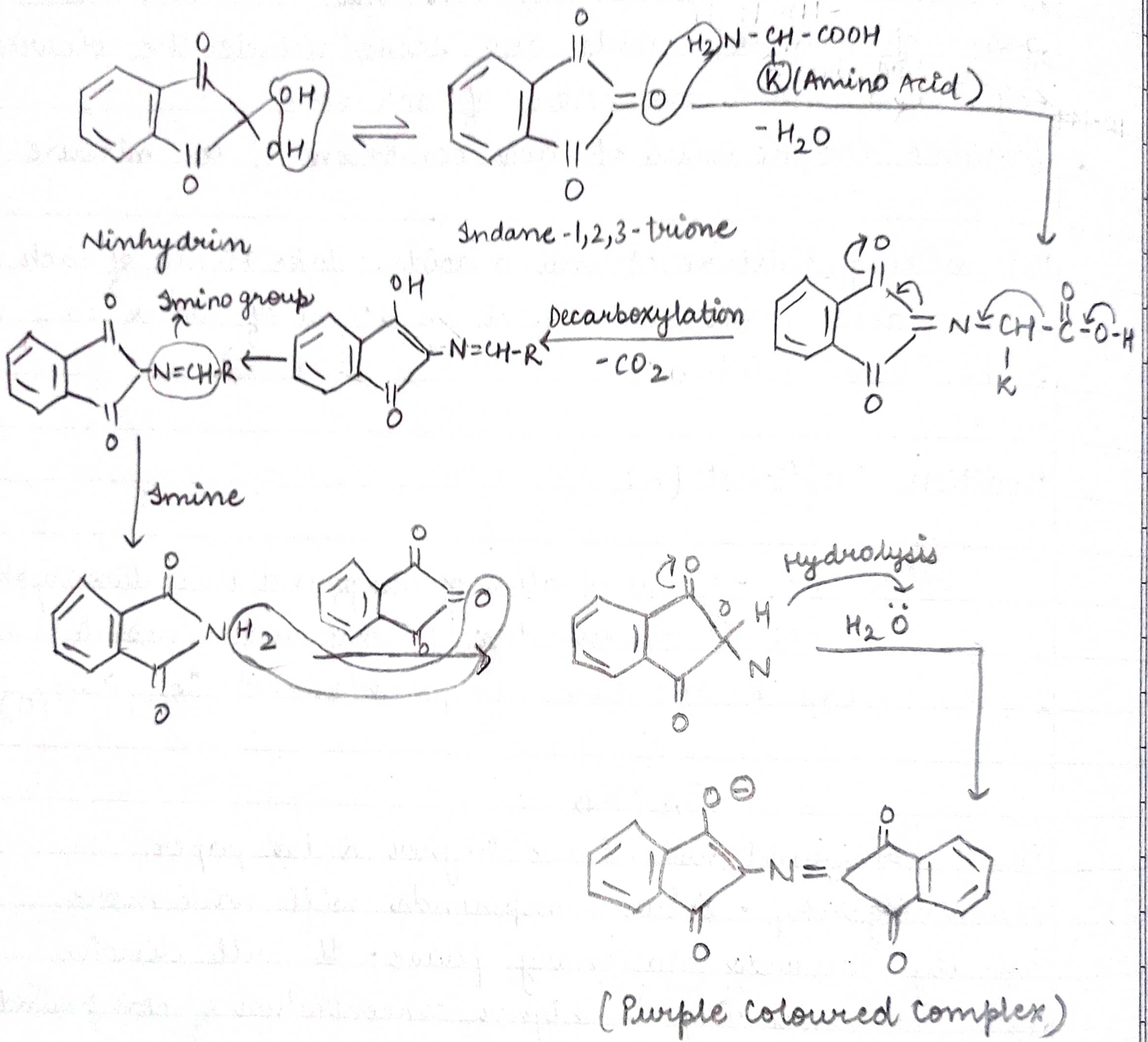
Polar compound will travel slowly on paper strip in comparison to non-polar compound.

Retention factor (R_f)

$$\text{Retention factor } (R_f) = \frac{\text{Distance travelled by compound}}{\text{Distance travelled by solvent front}}$$

Teacher's Signature :

Reaction of Ninhydrin with Amino Acids (except proline)



More polar compound will have less R_f than less polar (non-polar) compound.

Visualizing Agent

Amino acids are colourless. So to make the spots coloured, nin-hydrin solution is sprayed on chromatogram.

Nin-hydrin reacts with amino acids and make them purple (violet).

Proline gives yellow colour with nin-hydrin solution.

Result

The mixture has been separated by ascending paper chromatography.

Precautions

- Wear glasses while using nin-hydrin solution
- Butanol is inflammable, make sure that there are no open flames.
- Handle chromatogram with care.

Experiment - 9

Aim: To determine the boiling point of given organic liquid compound by simple distillation method

Apparatus required: Burner, tripod stand, wire gauze, burette stand, clamp, round bottom flask, thermometer, stand, liebig's condenser, adapter, receiving flask

Chemicals required: Given organic liquid compound

Theory

Boiling point: The temperature at which vapour pressure of a liquid becomes equal to the atmospheric pressure or simply the temperature at which liquid starts to boil.

Boiling point depends upon the following factors:

- 1) Hydrogen bonding - Compounds with hydrogen bonds will have higher boiling points than similarly sized molecules which do not have H-bonds.
- 2) Molecular weight - Higher the molecular weight, higher the boiling point.
- 3) Polarity - Greater the polarity, higher the boiling point.
- 4) Branching - With increase in branching, there is a decrease in boiling point.

Boiling point basically depends on forces of attraction

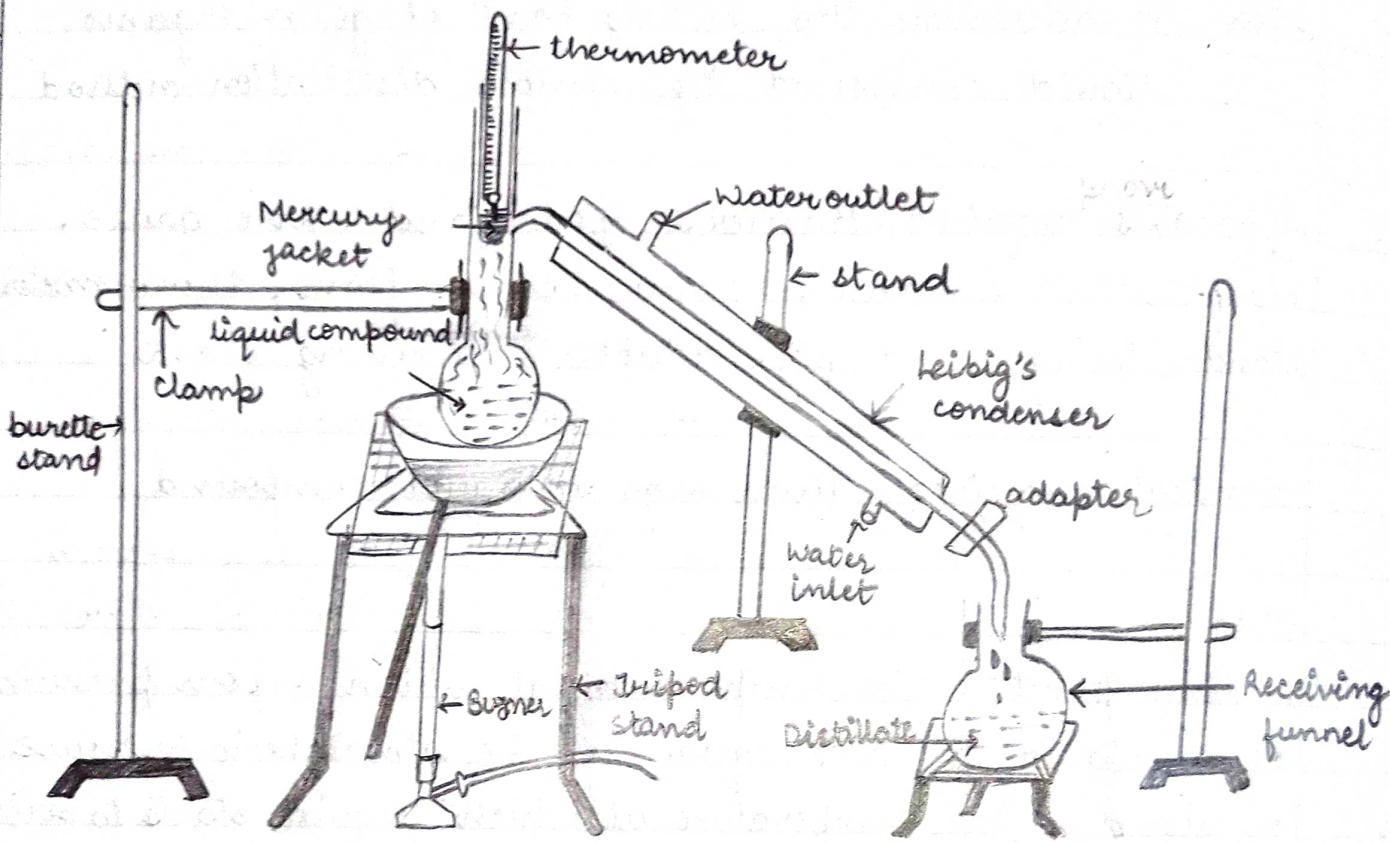


Fig: Distillation

taking place between molecules

Simple distillation: A method of separating mixtures based on differences in their volatilities in a boiling liquid mixture

A simple distillation apparatus consists of a boiling flask (round bottom flask) attached to a condenser and a receiving flask.

The distillation method of boiling point determination measures the temperature of the vapours above the liquid. Since these vapours are in equilibrium with the boiling liquid, they are the same temperature as the boiling liquid.

Procedure

Take the given liquid compound in a dry round bottom flask. Add a few pieces of pumice stones to avoid bumping (adding pumice stone gives a lot of surface area for bubbles to form and release the energy gradually as if bubbles are not formed, then the liquid will superheat and can possibly explode) and set up the distillation apparatus. The distillation flask is fitted to a water condenser and an adaptor is fitted to the water condenser to collect the distillate in the reservoir. Place the thermometer in the mercury jacket. Heat the liquid gently at a uniform rate. Discard the first fraction (2-3 ml) of distillate.

Continue the heating slowly and steadily. Note that temperature at which given liquid boils (constant temperature) and collect the distillate in the reservoir.

Results:

The boiling point of the given organic liquid was determined. (literature value of boiling point: _____ °C)

Precautions:

- All components of the distillation apparatus should be placed on a suitable stand and should be checked for cracks before their prior use.
- Lubricant such as grease can be applied carefully at the appropriate connections or joints.
- The inlet and outlet pipes of the condenser must be tight and should be connected in proper order.
- The distillation flask should be a round bottom for ensuring uniform and smooth boiling.
- Boiling chips or anti bumping granules should be added to the liquid in the distillation flask before heating.