Std. 12th

CHEMISTRY

PRACTICAL HANDBOOK

Instruction for Students :

1. L.H.S. means Left Hand Side (Blank page of practical record) and R.H.S. means Right Hand Side (line page of practical record)

2. L.H.S. page of each and every experiment should be written by pencil only.

3. R.H.S. page of each and every experiment should be written by blue/black pen.

4. Diagrams should be drawn neatly and should be properly labelled.

5. Graphs will be drawn on separate graph paper after noting observations on performing experiment.

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PART A : CHEMICAL KINETICS

Experiment No. 1

Study of the effect of concentration on the rate of reaction between sodium thiosulphate and hydrochloric acid.

Aim : To study the effect of concentration hangs on the rate of a reaction between Sodium thiosulphate ($Na_2S_2O_3$) and Hydrochloric Acid (HCl)

Requirements: (i) **Apparatus :** Two burettes (50mL), Pipette (10mL) ,four 100 mL conical flasks, stop watch, thick white paper with a cross mark.
(ii) **Chemicals :** 0.1 M $Na_2S_2O_3$, 1M HCl , distilled water.

Diagram :

Fig. 1 : Reaction between $Na_2S_2O_3$ and HCl
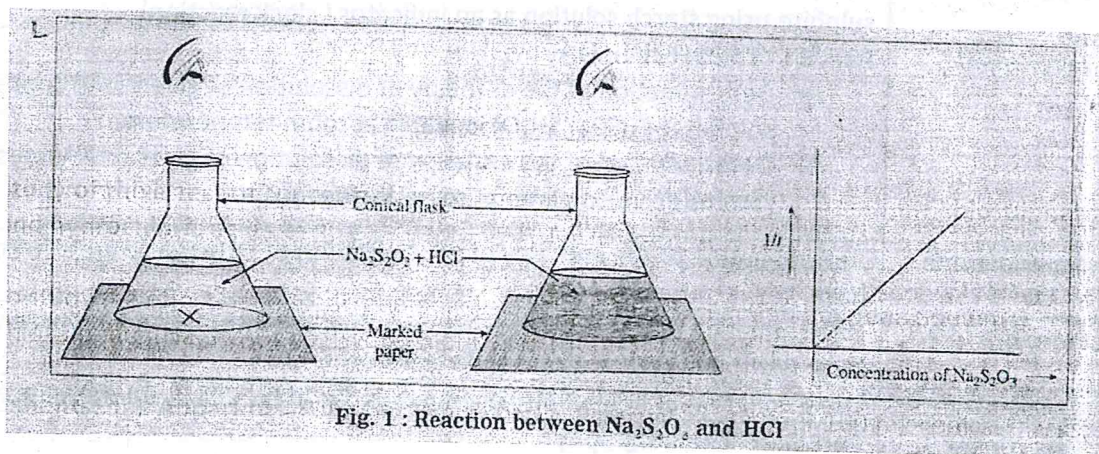
Observation table :

Conical flask	Volume of 0.1 M $Na_2S_2O_3$ (mL)	Volume of distilled water (mL)	Total volume (mL)	Volume of HCl (mL)	Conc. Of $Na_2S_2O_3$ Solution 'C' (M)	Time required For the mark to become just invisible t s	l/t s^{-1}
A	10	40	50	10	0.02		
B	20	30	50	10	0.04		
C	30	20	50	10	0.06		
D	40	10	50	10	0.08		

Graph : Plot a graph of l/t against the concentration 'C' which will be a straight line.

Conclusion : The rate of reaction increases with the increase in concentration of $Na_2S_2O_3$

PART A : CHEMICAL KINETICS

Experiment No. 1

Study of the effect of concentration on the rate of reaction between thiosulphate and hydrochloric acid

Aim : To study the effect of concentration Changes on the rate of a reaction between Sodium thiosulphate ($Na_2S_2O_3$) and Hydrochloric Acid (HCl)

Requirements: (i) **Apparatus :** Two burettes (50mL), Pipette (10mL), four 100 mL conical flasks, stop watch, thick white paper with a cross mark.
(ii) **Chemicals :** 0.1 M $Na_2S_2O_3$, 1M HCl, distilled water.

Chemical Reaction :

$$Na_2S_2O_3 + 2HCl \rightarrow 2\ N_aCl + H_2O + SO_2 + S\downarrow$$
$$\text{(aq)} \qquad \text{(aq)} \qquad \text{(aq)} \quad \text{(l)} \quad \text{(g)} \quad \text{(s)}$$
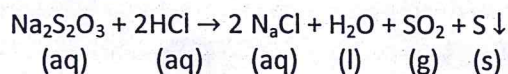
Procedure :
1. Wash all the glass apparatus with water and label washed conical flasks as A,B,C and D.
2. Wash burette with $Na_2S_2O_3$ solution. Fill the burette with $Na_2S_2O_3$ solution and adjust zero level after Removing air bubbles from the nozzle.
3. Wash another burette with water and fill it with distilled water And adjust the zero level.
4. Add $Na_2S_2O_3$ and water in different conical flasks as mentioned in The following table by burettes.
5. Add 10mL 1MHCl to the flask A kept on a marked paper and swirl the solution and start the stopwatch. The solution will start becoming turbid Observe the mark on the paper by viewing it from the top of the conical flask.
6. Stop the stopwatch when the mark on the paper becomes invisible and the formation of sulphur is complete and record the time.
7. Repeat the same procedure for remaining flasks B,C and D stepwise and record the time observation in the tabular form .

Graph : Plot a graph of l/t against the concentration 'C' which will be a straight line.

Conclusion : The rate of reaction increases with the increase in concentration of $Na_2S_2O_3$

Experiment No. 2 :

Study of the effect of temperature change on the rate of reaction between sodium thiosulphate and hydrochloric acid

Aim : To study the effect of temperature change on the rate of the reaction between sodium Thiosulphate ($Na_2S_2O_3$) and hydrochloric acid (HCl).

Requirements: (i) Apparatus : 250 mL conical flasks, measuring cylinder ,stopwatch, Thermometer, thermostat (or water bath) ,burner,wire gauze, Tripod stand, marked   asbestos sheet.
       (ii) Chemicals : 0.1 M $Na_2S_2O_3$, 1 M HCl, distilled water.

Diagram : -

Observations :
Volume of 0.1 M $Na_2S_2O_3$ = 50 mL
Volume of 1M HCl   = 10 mL

Observation Table :

Sr . No.	Temperature ^0C	1M HCl (mL)	Time required for the mark to be invisible t s	l/t s^{-1}
1	t = ... ^{0} C	10		
2	t + 10 = ... ^{0} C	10		
3	t + 20 =............... ^{0} C	10		
4	t + 30 =............... ^{0} C	10		
5	t + 40 =............... ^{0} C	10		
6	t + 50 =............... ^{0} C	10		

Graph : Plot a graph of l/t against the temperature (T^0C). It will be a straight line.

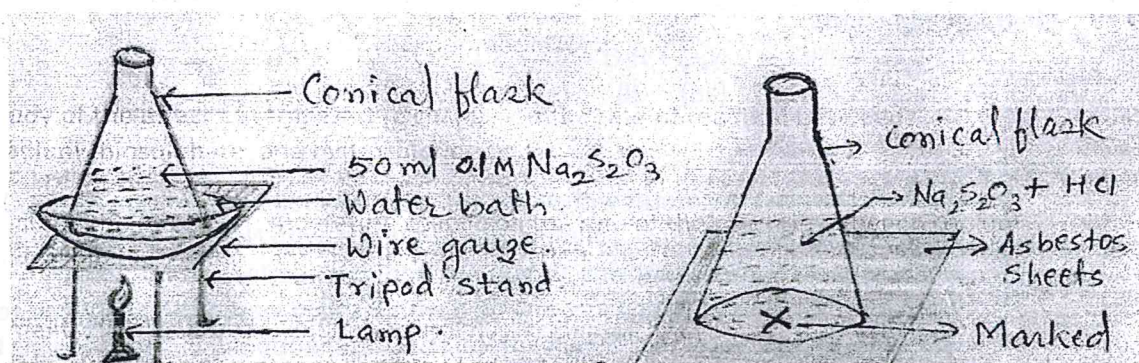**Conclusion :** Rate of reaction between $Na_2S_2O_3$ and HCl increases as the temperature increase

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Experiment No. 2 :

Study of the effect of temperature change on the rate of reaction between sodium thiosulphate and hydrochloric acid

Aim : To study the effect of temperature change on the rate of the reaction between sodium Thiosulphate ($Na_2S_2O_3$) and hydrochloric acid (HCl).

Requirements: (i) Apparatus : 250 mL conical flasks, measuring cylinder ,stopwatch, thermometer, thermostat (or water bath) ,burner, wire gauze, Tripod stand, marked asbestos sheet.
(ii) Chemicals : 0.1 M $Na_2S_2O_3$, 1 M HCl, distilled water.

Chemical Reaction :

$$Na_2S_2O_3 + 2HCl \rightarrow 2\ N_aCl + H_2O + SO_2 + S \downarrow$$
$$\text{(aq)} \qquad \text{(aq)} \qquad \text{(aq)} \quad \text{(l)} \quad \text{(g)} \quad \text{(s)}$$

Procedure :
1) Wash all the glass apparatus with water.
2) Take 50 mL of 0.1-M $Na_2S_2O_3$ solution in a clean conical flask using a measuring cylinder. Place the flask on a marked asbestos sheet in a water bath at room temperature . Note the temperature of sodium Thiosulphate solution (t^0C).
3) Add 10 mL of 1 M HCl to the flask, swirl the solution and stopwatch immediately. The solution will start becoming turbid. Observe the Mark on the asbestos sheet by viewing it from the top of the flask.
4) Stop the stopwatch when the mark on the asbestos sheet just becomes Invisible and the formation of sulphur is complete, record the time.
5) Take 50 mL of 0.1 M $Na_2S_2O_3$ solution in another clean conical flask. Place the flask on a marked asbestos sheet in a water bath. Heat the Water bath using a burner to increase the temperature of the solution by 10^0C [i.e. (t + 10) ^0C]. Maintain the temperature by reducing the heating of water bath. (Using a thermostat gives better results.)
Add 10 mL of M HCl to the flask, swirl the solution ,keep it on yhe marked asbestos sheet and start the stopwatch.
6) The solution will start becoming turbid. Observe the mark on the asbestos sheet by viewing it from the top of the flask.
7) Stop the stopwatch when the mark just becomes invisible and the formation of sulphur is complete. Record the time.
8) Repeat the procedure for the temperatures (t+20) ^0C,(t+30)^0C , (t+40) ^0C,and (t+50) ^0C Record the observation

Precautions:
1) Use of thermostat gives better result .
2) Record the time accurately.

Graph : Plot a graph of against the temperature. It will be a straight line.

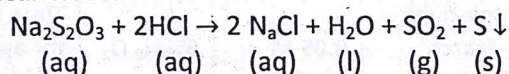**Conclusion :** Rate of reaction between $Na_2S_2O_3$ and HCl increases as the temperature increases.

Experiment No. 3

Study of the reaction rate of the reaction between iodide ions and hydrogen peroxide at different concentration of iodide ions

Aim : To study the rate of the reaction between iodide ions (I-) and hydrogen peroxide ($H_2 O_2$)
Using different concentrations of iodide ions.

Requirements : (i) Apparatus : Four conical flasks (250 mL) , burette, measuring cylinder, stopwatch.

(ii) Chemicals : 6% $H_2 O_2$, 0.05 M $Na_2S_2O_3$,0.1 M KI , 2.5 M $H_2 SO_4$, freshly prepared starch solution.

Observations:

Conical flask	Volume of solutions						Time required for appearance Of blue colour t s
	0.1M KI mL	2.5 M $H_2 SO_4$ mL	Distilled Water mL	Starch Solution mL	0.05 M $Na_2S_2O_3$ mL	6% $H_2 O_2$ mL	
A	20	10	70	5	10	10	
B	30	10	60	5	10	10	
C	50	10	40	5	10	10	
D	70	10	20	5	10	10	

Conclusion : The reaction reaches completion earlier with increase in the concentration of KI.
Hence, the rate of reaction increases as the concentration of iodide ions increases.

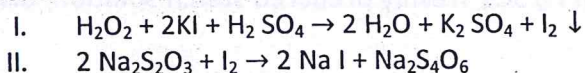**Graph :** Plot a graph of t s against the concentration of KI .

Experiment No. 3

Study of the reaction rate of the reaction between iodide ions and hydrogen peroxide at different concentration of iodide ions

Aim : To study the rate of the reaction between iodide ions (I^-) and hydrogen peroxide (H_2O_2) Using different concentrations of iodide ions.

Requirements : (i) **Apparatus :** Four conical flasks (250 mL) , burette, measuring cylinder, stopwatch.

(ii) **Chemicals :** 6% H_2O_2 , 0.05 M $Na_2S_2O_3$, 0.1 M KI , 2.5 M H_2SO_4, freshly prepared starch solution.

Chemical Reaction :

I. $H_2O_2 + 2KI + H_2SO_4 \rightarrow 2 H_2O + K_2SO_4 + I_2 \downarrow$

II. $2 Na_2S_2O_3 + I_2 \rightarrow 2 Na I + Na_2S_4O_6$

Procedure:

1) Take four clean conical flasks and label them as A,B,C and D.
2) Take 20 ml , 30 ml , 50 mL and 70 mL of 0.1M KI in the flasks A,B,C,D respectively.
3) Add 10 ml of 2.5 M H_2SO_4 to each flasks.
4) Add 70 ml , 60 ml ,40 ml and 20mL distilled water to the flasks A,B,C,D respectively, to make total volume equal to 100 mL in each flask.
5) Add 5mL of freshly prepared starch solution to each flask.
6) Add 10mL of 0.05 M $Na_2S_2O_3$ solution to each flask.
7) Add 10 mL of 6% H_2O_2 solution to the flask 'A' using burette. Shake well and start the stopwatch immediately. Stop the stopwatch when blue colour just appears in the Flask and record the time. (This blue colour arises due to the reaction of starch with I_2 after the complete consumption of $Na_2S_2O_3$)
8) Repeat the procedure for the flasks B,C and D stepwise. Record the observations.

Precautions:

1) Use freshly prepared solution.
2) Note the time accurately.

Conclusion : The reaction reaches completion earlier with increase in the concentration of KI. Hence, the rate of reaction increases as the concentration of iodide ions increases.

Experiment No. 4

Study of the reaction rate of reaction between potassium iodate and sodium sulphite using starch solution as an indicator (clock reaction)

Aim: To study the reaction rate of a reaction between potassium iodate (KIO_3) and sodium sulphite (Na_2SO_3) using starch solution as an indicator, (clock reaction).

Requirements: (i)**Apparatus:** Four conical flask (250 mL) ,burette (50 mL) measuring cylinder, pipettes (25 mL and 10 mL), stopwatch.

(ii) **Chemicals :** 0.1 M KIO_3, 0.01 M Na_2SO_3 (freshly prepared), 2M $H_2 SO_4$, freshly prepared starch solution, distilled water.

Observation Table :

Conical flask	Volume of solutions					Time required for blue colour to appear (t) s
	0.1M KIO_3 mL	2.5 M $H_2 SO_4$ mL	Distilled Water mL	Starch Solution mL	0.01 M $Na_2S_2O_3$ mL	
A	10	10	80	5	25	
B	20	10	70	5	25	
C	30	10	60	5	25	
D	40	10	50	5	25	

Conclusion : Time required for appearance of blue colour decreases with increase in the concentration Of potassium iodated (KIO_3) solution ,hence the rate of reaction increases with the increase in concentration of KIO_3

Graph :- Plot a graph of time (t s) against the concentration of KIO_3
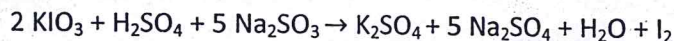
Experiment No. 4

Study of the reaction rate of reaction between potassium iodate and sodium sulphite using starch solution as an indicator (clock reaction)

Aim: To study the reaction rate of a reaction between potassium iodate (KIO_3) and sodium sulphite (Na_2SO_3) using starch solution as an indicator, (clock reaction).

Requirements: (i) **Apparatus:** Four conical flask (250 mL),burette (50 mL), measuring cylinder, pipettes (25 mL and 10 mL), stopwatch.

(ii) **Chemicals :** 0.1 M KIO_3 0.01 M Na_2SO_3 (freshly prepared), 2M H_2SO_4 freshly prepared starch solution, distilled water.

Chemical Reaction :

$$2 KIO_3 + H_2SO_4 + 5 Na_2SO_3 \rightarrow K_2SO_4 + 5 Na_2SO_4 + H_2O + I_2$$

Procedure :

1) Take four clean conical flask and label them as A,B,C and D.
2) Wash burette with water. Rinse it with 0.1 M KIO_3 Fill the burette with 0.1 M KIO_3 solution and adjust the solution up to zero mark after removing air bubbles from the nozzle.
3) Take 10 mL , 20 mL 30 mL and of 0.1 M KIO_3 solution in the flasks A,B,C,D respectively.
4) Add 10 mL of 2M H_2SO_4 to each flask using a measuring cylinder.
5) Add 80 mL, 70 mL , 60 mL and 50mL of distilled water in flasks A,B,C ,D respectively.
6) Add 5mL of freshly prepared starch solution to each flask.
7) Wash 25 mL pipette with water . Rinse it with 0.01 M Na_2SO_3 solution.
8) Add 25mL of 0.01 M Na_2SO_3 solution to flask 'A' Swirl the solution and start stopwatch immediately. Stop the stopwatch immediately when blue colour just appears indicating the completion of the reaction. Record the time.
9) Repeat the procedure for flasks B,C and D stepwise. Record the observations.

Precautions:

1) Use freshly prepared Na_2SO_3 solution as on exposure to air it gets oxidized to Na_2SO_4
2) Use freshly prepared starch solution .
3) Note the time accurately.

Conclusion : Time required for appearance of blue colour decreases with increase in thrconcentration Of potassium iodate (KIO_3) solution ,hence the rate of reaction increases with the increase in concentration of KIO_3.

PART B : THERMOCHEMISTRY

Experiment No. 5 (A)

Determination of the enthalpy of dissolution Of given solid in water at room temperature

Aim : To determine the enthalpy of dissolution of given solid copper Sulphate ($CuSO_4 5H_2O$) in water at room temperature.

Requirements: (i) Apparatus: Polythene bottle with cork having two holes, stirrer, measuring cylinder, thermometer(least count 0.1 ^0C).
(ii) Chemicals : $CuSO_4 \cdot 5H_2O$, distilled water.

Diagram : -
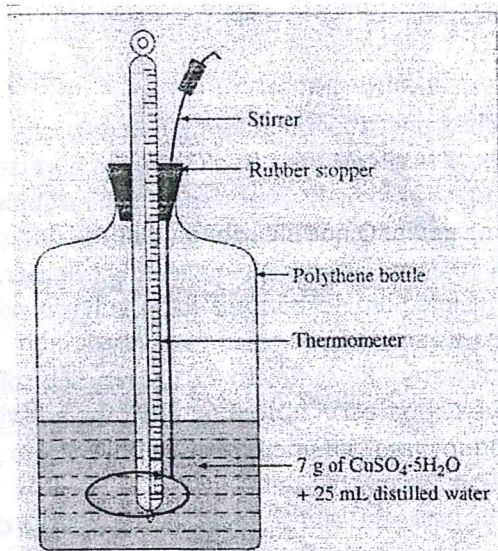
Fig. 2 : Heat of dissolution of $CuSO_4$

Observations:
1) Volume of water taken = 25 mL (25 grams)
2) Weight of $CuSO_4 5H_2O$ taken =   7.0 grams
3) Initial temperature of water = t_1 = --------- ^0C
4) Final temperature of the solution = t_2 = -------- ^0C
5) Water equivalent of the polythene bottle = q (in gram) (It is given.)

q = Specific heat x weight of polythene bottle .
q= .... g

Calculations:
Heat absorbed evolved in the reaction = Heat lost / gained by water + Heat lost / gained by polythene bottle.

Heat lost / gained by water = Mass of water x specific heat x change in temperature
= 25 g x 4.184 J g^{-1} $^0C^{-1}$ x ($t_1 - t_2$) ^0C J
=

Heat lost / gained by polythene bottle = water equivalent x specific heat x change in temperature
= q g x 4.184 J g^{-1} $^0C^{-1}$ x ($t_1 - t_2$) ^0C J
=

Heat absorbed / evolved in the reaction = [25 x 4.184 x ($t_1 - t_2$)] + [q x 4.184 x ($t_1 - t_2$)]
Q = 4.184 x ($t_1 - t_2$) x (25 + q) = ... J

Experiment No. 5 (A)

Determination of the enthalpy of dissolution Of given solid in water at room temperature
(A)Enthalpy of dissolution of $CuSO_4 5H_2O$

Aim : To determine the enthalpy of dissolution of given solid copper Sulphate ($CuSO_4 5H_2O$) in water at room temperature.

Requirements : i)**Apparatus :** Polythene bottle with cork having two holes ,stirrer, measuring cylinder, thermometer(least count $0.1\ ^0C$).

ii) **Chemicals :** $CuSO_4\ 5H_2O$, distilled water.

Procedure:

1) Wash polythene bottle and measuring cylinder with water.
2) Take 25 mL of distilled water in the polythene bottle and Note its temperature (t_1).
3) Now take 7.0 g of finely powdered $CuSO_4.\ 5H_2O$ (accurately weighed) and add it to this water in one lot and stir well.
4) Insert the thermometer in the solution. The temperature of the solution changes. Note the constant temperature (t_2).

Result : The heat of solution of $CuSO_4 5H_2O$, $\Delta\ H$ = ... KJ Mol $^{-1}$

Q is the heat absorbed / evolved when (7.0) g of $CuSO_4 \cdot 5H_2O$ are dissolved in water , hence when 1 mole of $CuSO_4 \cdot 5H_2O$ (Molar mass = 249 g mol^{-1}) is dissolved in water , the hens absorbed / evolved will be

$$\Delta H = \frac{Q \times 249}{7} = \dots J = \dots KJ$$

Heat of solution of $CuSO_4 \cdot 5H_2O =$ $\Delta H = \dots$ KJ mol^{-1}

Result : The heat of solution of $CuSO_4 \cdot 5H_2O =$ $\Delta H = \dots$ KJ mol^{-1}

Experiment No. 5 (B)

Enthalphy of dissolution of KNO_3

Aim : To determine the enthalpy of dissolution of potassium nitrate (KNO_3) in water at room temperature.

Requirements : i)**Apparatus :** Polythene bottle , stirrer , measuring cylinder , thermometer (least count 0.1 ^0C).

ii) **Chemicals:** KNO_3, distilled water .

Diagram :-

Fig. Heat of dissolution of KNO_3
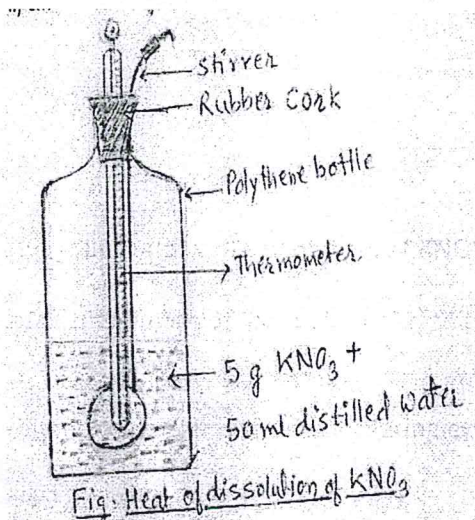
Observations :

1) Volume of water taken = 50 mL (50 grams)
2) Weight of KNO_3 taken = 5.0 grams
3) Initial temperature of water = t_1 = ... ^0C
4) Final temperature of the solution = t_2 = ... ^0C
5) Water equivalent of the polythene bottle = q (in gram) (It is given)

q = Specific heat x weight of polythene bottle .

q= .... g

Calculations :

Heat absorbed /evolved in the reaction=Heat lost/gained by water + heat lost or gained by the polythene bottle

Heat lost / gained by water = Mass of water x specific heat x change in temperature

= 50 g x 4.184 J g^{-1} $^0C^{-1}$ x ($t_1 - t_2$) J

Heat lost / gained by polythene bottle = water equivalent x sp . heat of water x change in temperature

= q g x 4.184 J g^{-1} $^0C^{-1}$ x ($t_1 - t_2$) J ^0C

=

Heat absorbed / evolved in the reaction = [50 x 4.184 x ($t_1 - t_2$)] + [q x 4.184 x ($t_1 - t_2$) J

=

Q = 4.184 x (50 + q) x (t_1 - t_2) = ... J

Q is the heat absorbed / evolved when (5.0) g of KNO_3 are dissolved in water, hence when 1 mole Of KNO_3 (Molar mass = 101 g mol^{-1}) is dissolved in water, the heat absorbed / evolved

Will be $\dfrac{Q \times 101}{5}$ = ... J = ... kJ .

Heat of solution of KNO_3, Δ H = ... kJ mol^{-1}

Result : The heat of solution of KNO_3, Δ H = ... KJ Mol^{-1}

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Experiment No. 5 (B)

Enthalphy of dissolution of KNO_3

Aim : To determine the enthalpy of dissolution of potassium nitrate (KNO_3) in water at room temperature.

Requirements : **i)Apparatus :** Polythene bottle , stirrer , measuring cylinder , thermometer
(least count 0.1 ^0C).
ii) Chemicals: KNO_3 distilled water .

Procedure :
1) Wash polythene bottle and measuring cylinder with water.
2) Take 50 mL of distilled water in the polythene bottle and note its temperature (t_1).
3) Now , take 5 grams of finely powdered potassium nitrate (accurately weighed) and Add it to this water in one lot and stir well
4) Insert the thermometer in the solution . The temperature of the solution changes Note
5) The constant temperature (t_2).

Result : The heat of solution of KNO_3, Δ H = ...KJ Mol^{-1}

Experiment No. 6

Determination of the enthalpy of neutralization of Strong acid and strong base

Aim : To determine the heat of neutralization of a strong acid and a strong base.

Requirements: i) Apparatus : 500 mL polythene bottle with stirrer, 100 mL measuring cylinder, 250 mL beaker , thermometer .

ii) Chemicals : 0.2 M NaOH solution and 0.2 M HCL solution.

Diagram :-
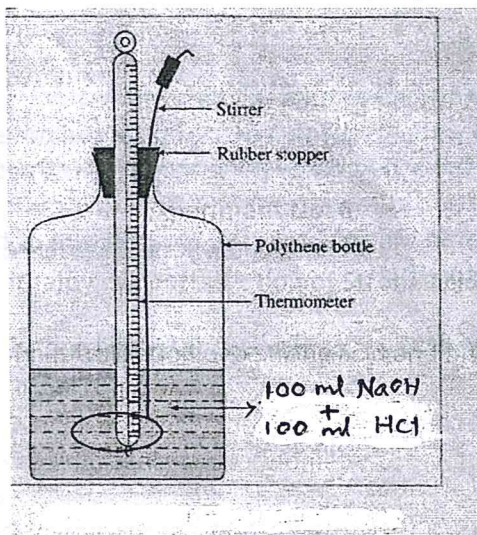
Observations :

1. Initial temperature of the NaOH solution = t_1 (in ^0C)= .... ^0C
2. Initial temperature of the HCl solution = t_2 (in ^0C) = ... ^0C
3. Initial temperature of the mixture = $\dfrac{t_1 + t_2}{2}$ = ... ^0C
4. Final temperature of the mixture = t_3 (in ^0C) = ... ^0C
5. Rise in temperature(Δt) = $\left[t_3 - \left(\dfrac{t1+t2}{2} \right) \right] ^0C$ = ... ^0C
6. Volume of NaOH solution = 100 mL
7. Volume of HCl solution = 100 mL

   Total volume of the solution = 200 mL

   If the density of the solution is assumed to be 1 g mL^{-1} than mass of solution = 200 g.

   Water equivalent of the polythene bottle = q (in gram) (It is given.)

   q = Specific heat x weight of polythene bottle .

   q = ... g

Calculations :

1. Heat evolved in the neutralization (Q) = Heat absorbed by the solution + Heat absorbed by the polythene bottle
2. Heat absorbed by the polythene bottle = Water equivalent x specific heat x rise in temperature.

   = q g x   4.184 J g^{-1} ^0C x Δt ^0C J

   =

Experiment No. 6

Determination of the enthalpy of neutralization of Strong acid and strong base

Aim : To determine the heat of neutralization of a strong acid and a strong base.

Requirements: i) Apparatus : 500 mL polythene bottle with stirrer, 100 mL measuring cylinder, 250 mL beaker , thermometer ($0\ ^0C - 100\ ^0C$ range , with $0.1\ ^0C$ least count).

ii)Chemicals : 0.2 M NaOH solution and 0.2 M HCL solution.

Chemical Reaction :-

$$NaOH_{(aq)} + HCl_{(aq)} \Longrightarrow NaCl_{(aq)} + H_2O_{(l)}\ \Delta H = -57.36\ kJ\ mol^{-1}$$

Procedure :

1. Wash polythene bottle with water and dry it . Wash measuring Cylinder with water.
2. Measure 100 mL of the 0.2 M NaOH solution using the measuring cylinder and transfer the solution completely to
3. The polythene bottle . (Drain the measuring cylinder in the bottle).
3. Insert the thermometer in the NaOH solution and note the constant temperature (t_1).
4. Wash and dry the measuring cylinder . Measure 100 mL of 0.2 M HCl solution using the measuring cylinder and transfer the solution completely into a 250 mL beaker.
5. Insert the thermometer in the HCl solution and note the constant temperature (t_2).
6. Pour the HCl solution from the beaker into the NaOH solution in the polythene bottle and stir the solution using the stirrer.
7. Insert the thermometer in the solution (mixture) and note the maximum temperature attained by the solution (t_3)

Result : Heat of neutralization of HCl , ΔH = ... kJ mol^{-1}

3. Heat absorbed by the mixture = Mass x sp. Heat x rise in temperature

$$= 200 \quad g \quad x \quad 4.184 \ J \ g^{-1} \ ^0C \ x \, \Delta t \ ^0C \ J$$
$$=$$

4. Heat evolved in the neutralization = [200 x 4.184 x Δt] + [q x 4.184 x Δt]
$$=$$
$$Q = 4.184 \ x \quad \Delta t \ x \ (\ 200 + q \ ) = ... \ J$$

5. Heat evolved in the neutralization of 100 mL of 0.2 M HCl = Q = ... J
6. Heat evolved in the neutralization of 1000 mL of 1 M HCl = $\dfrac{Q \ x \ 1000}{100 \times 0.2}$ = ... J

$$\Delta H = ... \ kJ \ mol^-$$

Result : Heat of neutralization of HCl , Δ H = ... kJ mol$^-$

Experiment No. 7

Determination of the enthalpy change during the interaction (hydrogen bond formation) Between acetone and chloroform

Aim : To determine the enthalpy change during the interaction (hydrogen bond formation) Between acetone and chloroform.

Requirements : i) Apparatus : Polythene bottle , Rubber cork, measuring cylinder , thermometer

ii) **Chemicals :** Acetone and chloroform.

Diagram : -
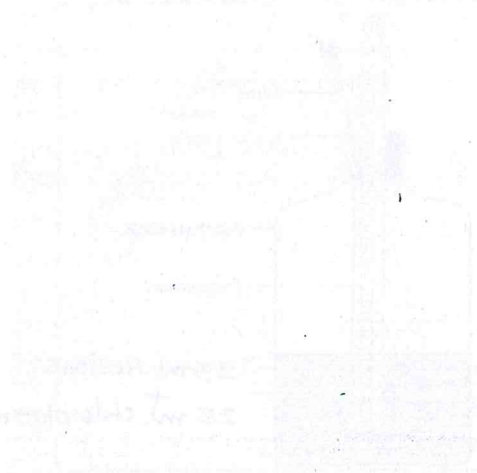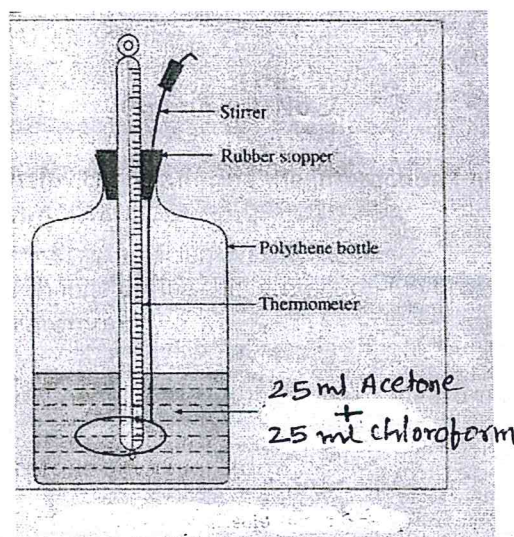
Observations:
1) Initial temperature of 25 mL acetone t_1 ^0C = ... ^0C
2) Initial temperature of 25 mL chloroform t_2 ^0C =............. ^0C

3) Initial temperature of 50 mL mixture $\dfrac{(t_1 + t_2)}{2}$ =............. ^0C

4) Final temperature of the mixture t_3 ^0C = ... ^0C

5) Rise in temperature of mixture $(\Delta t) = \left[t_3 - \left(\dfrac{t1+t2}{2}\right) \right]$ ^0C = ... ^0C

6) Water equivalent of polythene bottle ,   q g (given) =
     q = Specific heat x weight of polythene bottle .
     q= .... g

7) Density of acetone   0.787 g / cm ^{3}
8) Density of chloroform   1.499 g / cm^3
9) Specific heat of acetone   2.18 J / g
10) Specific heat of chloroform   0.96 J / g

Calculations :
1. Heat evolved in the reaction = Heat absorbed by the mixture +
                   Heat absorbed by the polythene bottle

Experiment No.7

### Determination of the enthalpy change during the interaction (hydrogen bond formation) Between acetone and chloroform

Aim : To determine the enthalpy change during the interaction (hydrogen bond formation)
Between acetone and chloroform.

Requirements : i) **Apparatus :** Polythene bottle with cork having two holes
(for stirrer and thermometer) ,measuring
ii) **Chemicals :** Acetone and chloroform.

Chemical Reaction :
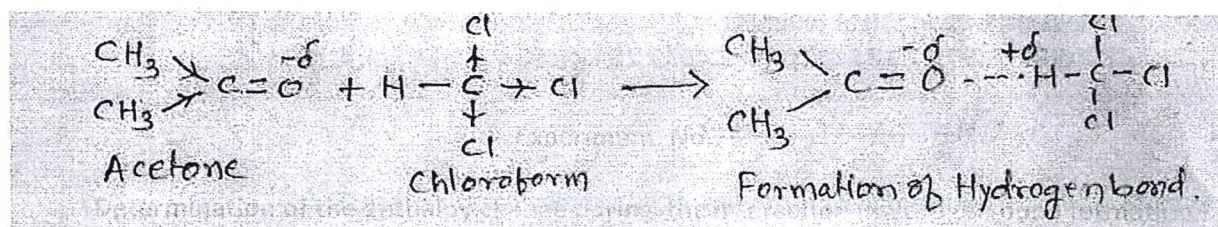
Procedure :

1. Take a clean , dry measuring cylinder and measure 25 mL of acetone and transfer it to a clean , dry polythene bottle , Cork the polythene bottle.
2. Pass the thermometer through the hole of the cork into the polythene bottle so that the bulb of the thermometer is immersed in the solution. Record the steady temperature as t_1 ^{0}C.
3. Take a clean , dry measuring cylinder and measure 25 mL of chloroform.
4. Place the thermometer into 25 mL chloroform and record the steady temperature of 25 mL of chloroform as t_2 ^{0}C.
5. Insert the thermometer and stirrer through the holes of the cork of polythene bottle.
6. Open the cork of the polythene bottle and immediately pour chloroform from measuring cylinder into the bottle. Cork the bottle and stir the mixture.
7. Record the maximum constant temperature of the mixture as t_3 ^{0}C.
8. Record the observation in tabular form.

Precautions:

1. Check the thermometer before starting the experiment . The mercury column must be continuous.
2. Thermometer should be immersed in the solution so that its bulb does not touch the bottom or side of polythene bottle.
3. Stir the solution and record the temperature carefully.

Result : Enthalpy change due to formation of intermolecular hydrogen bonds , ΔH = ……. kJ

2. Heat absorbed by polythene bottle = Water equivalent x specific heat x rise in temperature.

$$= q \ g \times 4.184 \quad J g^{-1} \ {}^{\circ}C^{-1} \ \times \Delta t \ {}^{\circ}C \quad J$$
$$=$$

3. Heat absorbed by the mixture = Mass x sp. Heat x rise in temperature

= Mass x sp. Heat x rise in temperature of acetone+ Mass x sp. Heat x rise in temperature of chloroform.

$$= \left[\ (0.787 \times 25) \quad g \ cm^{-3} \ cm^3 \times 2.18 \ J g^{-1} \ {}^{\circ}C^{-1} \times \Delta \ t \ {}^{\circ}C \ \right]$$

$$+ \left[\ (1.499 \times 25) \quad g \ cm^{-3} \ cm^3 \times 0.96 \ J g^{-1} \ {}^{\circ}C^{-1} \times \Delta \ t \ {}^{\circ}C \right] J$$

$$=$$

Heat evolved in the reaction Q =

$$= [\ (0.787 \times 25) \times 2.18 \times \Delta t] + [\ (1.499 \times 25) \times 0.96 \times \Delta t] + [\ qx \ 4.184 \times \Delta t \ ] J$$

$$\Delta \ H = \ldots\ldots\ldots J = \ldots\ldots\ldots kJ$$

Result : Enthalpy change due to formation of intermolecular hydrogen bonds , $\Delta H = \ldots\ldots kJ$

Experiment No. 8

Determination of heat of displacement of Cu From CuSO₄ Solution by zinc

Aim : To determine heat of displacement (enthalpy change) of copper (Cu) from copper sulphate ($CuSO_4$) solution by zinc (Zn) .

Requirements : (i) **Apparatus :** 100 mL polythene bottle , thermometer ($0^0C - 100\,^0C$ range with $0.1\,^0C$ least count)   , 50 mL of measuring cylinder.
(ii) **Chemicals :** 0.2 M copper sulphate solution , zinc powder.

Diagram:-

Stirrer
Rubber stopper
Polythene bottle
Thermometer

50 mL 0.2 M CuSO₄ Solution
+
1.0 g Zn powder.
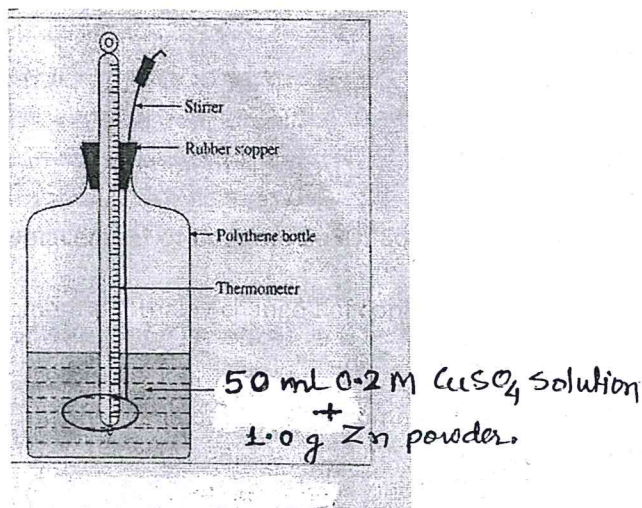
Observation :

1.   Initial temperature of the CuSO₄ solution = t_1 (in ^0C) = ... ^0C
2.   Final temperature of the CuSO₄ solution = t_2 (in ^0C) =.............. ^0C
3.   Rise in the temperature of the = ($t_2 - t_1$) (in ^0C)   =.............. ^0C
4.   Water equivalent of the polythene bottle = q (in gram) (given) =
        q = Specific heat x weight of polythene bottle .
        q= .... g

Calculations:

1.   Heat evolved in the reaction = Heat absorbed by the solution +
                Heat absorbed by the polythene bottle
2.   Heat absorbed by the solution = Mass of solution x specific heat x rise in temperature .
        [If the density of the solution is assumed to be 1 g mL
            Then mass of solution =50 g .
3.   Heat absorbed by the solution = Mass x sp. Heat x rise in temperature
        = 50 g x 4.184 J g^{-1} ^0C x ($t_2 - t_1$) ^0C J
        =
4.   Heat absorbed by the polythene bottle = water equivalent x sp. Heat x rise in temperature
        =q g x   4.184   J g^{-1} ^0C x ($t_2 - t_1$) ^0C J
        =
5.   Heat evolved in the reaction = [50 x 4.184 x ($t_2 - t_1$) + [q x 4.184 x ($t_2 - t_1$]
        =
        Q = 4.184 x ($t_2 - t_1$) x (50 + q)
   = ... J

Experiment No. 8

Determination of heat of displacement of Cu From $CuSO_4$ Solution by zinc

Aim : To determine heat of displacement (enthalpy change) of copper (Cu) from copper sulphate ($CuSO_4$) solution by zinc (Zn) .

Requirements : i) Apparatus : 100 mL polythene bottle , thermometer (0^0C – $100\,^0C$ range with $0\,^0C$ least count) , 50 mL of measuring cylinder.
      ii) Chemicals : 0.2 M copper sulphate solution , zinc powder.

Chemical Reaction :

$$CuSO_4 + Zn \rightarrow ZnSO_4 + Cu \qquad \textbf{OR} \qquad Cu^{+2} + Zn \rightarrow Zn^{2+} + Cu$$
$$\text{(s)} \qquad\qquad \text{(s)} \qquad\qquad\qquad \text{(aq)} \qquad \text{(aq)} \quad \text{(s)}$$

Procedure :

1. Wash polythene bottle and measuring cylinder with water.
2. Measure 50 mL of 0.2 M $CuSO_4$ solution using a measuring cylinder and transfer it to the polythene bottle.
3. Insert the thermometer in the solution and accurately note the temperature (t_1)
4. Add about 1.0 g of the zinc power to the solution and shake the bottle to mix the powder thoroughly . Shake till the blue colour of the solution vanishes completely.
5. Keep the thermometer inserted in the solution and record the maximum temperature attained by the solution (t_2).

Result : Heat of displacement of copper by zinc , $\Delta H =$ ………. $KJ\,mol^{-1}$

6. Heat evolved in the displacement of copper (1 g atom) from
   1000 mL of 1.0 M CuSO$_4$ solution = $\dfrac{Q \times 1000}{50 \times 0.2}$ = ... J

   Heat of reaction (ΔH) = ... K J mol^{-1}

Result : Heat of displacement of copper by zinc , ΔH = ... K J mol^{-1}

PART D : CHROMATOGRAPHY

Experiment No. 9

Separation of coloured components from a mixture of inks

Aim : To separate the coloured components present in the mixture of red ink and blue ink by ascending paper chromatography and determine their R_f values.

Requirements : i) Apparatus : A glass jar with a lid , capillary , chromatography paper , pencil.

ii) Chemicals : Mixture of equal volume of red ink and blue ink ; 50% alcohol.

Diagram : -

Fig. 4 : Chromatography paper suspended in a glass jar

Observation table : -

Spot on the Chromatogram	Distance travelled By the spot Cm	Distance travelled by The solvent front Cm	R_f value
Red spot	OA = ………. cm	10 cm	OA / 10 =………. cm
Blue spot	OB =………. cm	10 cm	OB / 10 =………. cm

Result :

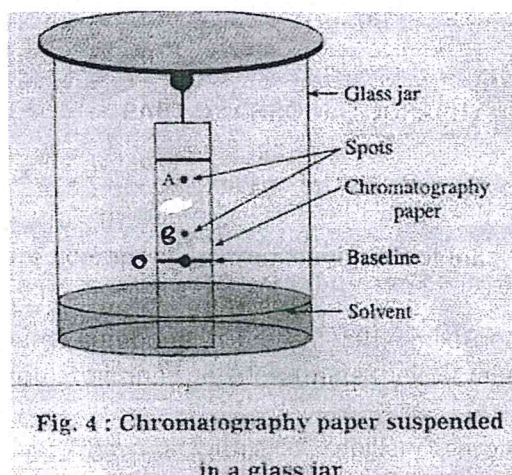| R_f value of red spot	………. cm
R_f value of blue spot	………. cm

PART D : CHROMATOGRAPHY

Experiment No. 9

Separation of coloured components from a mixture of inks

Aim : To separate the coloured components present in the mixture of red ink and blue ink by ascending paper chromatography and determine their R_f values.

Requirements : i) Apparatus : A glass jar with a lid , capillary , chromatography paper , pencil.

ii) Chemicals : Mixture of equal volume of red ink and blue ink ; 50% alcohol.

Procedure :

1. Take about 10 mL of 50 % alcohol (eluent) in a glass jar . (Its level should be 1 cm from the bottom of the jar) . Cover it with its lid and leave it for 30 minutes for saturation the jar.
2. Cut a strip of chromatography paper in a proper size , (at least 14 cm or according to the size of the jar)
3. Draw a line by pencil parallel to one edge of the paper , 2 cm above the edge. This is the base line.
4. Draw another line at a distance of 10 cm from the base line . This is solvent front.
5. Using a capillary , Place a drop of the given mixture of inks at the centre of the base line. Allow it to dry . Successively put 2 – 3 drops at the same point and allow to dry them.
6. Suspend the paper strip in the jar such that the lower end of the strip carrying the apot, just dips in the solvent about a centimetre height . Cover the jar .
7. When the solvent (eluent) reaches the upper line (solvent front) remove the paper and dry it . Mark the spots.
8. Measure the distance of spots from the base line . Record the observations .
9. Calculate the R , value for each spot using the formula ,

$$R_f \; = \; \frac{\text{Distance travelled by the spot}}{\text{Distance travelled by the solvent front}}$$

Precautions :

- Chromatography paper strip should remain erect when suspended in the jar .
- Let the spots dry properly before placing the paper in the jar .
- Do not disturb the jar during the experiment .

Result :

R_f value of red spot	.... cm
R_f value of blue spot	.... cm

Experiment No. 10

Separation of coloured components present in the mixture of extract of coloured flowers and leaves ascending paper chromatography.

Aim : To separate coloured components present in red flower , green and yellow leaves by ascending paper chromatography and determine their R_f values .

Requirements : i) Apparatus : Chromatography paper , glass cylinder (glass jar) with a lid , capillary tube , pencil.

ii) Chemicals : A mixture of extract of red flower (red rose) , green leaves and yellow leaves and 50 % alcohol .

Diagram :-

Fig. 4 : Chromatography paper suspended in a glass jar

Observation table :

Spot on the Chromatogram	Distance travelled By the spot Cm	Distance travelled By the solvent front Cm	R_f value
Red	OA =.......... cm	10 cm	OA /10 =.......... cm
Green	OB =.......... cm	10 cm	OB / 10 =.......... cm
Yellow	OC =.......... cm	10 cm	OC / 10 =.......... cm

Result :

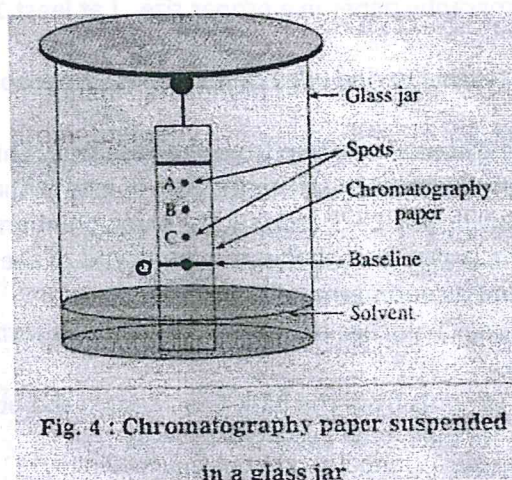| R_f value for red colour	.... cm
R_f value for green colour	.... cm
R_f value for yellow colour	.... cm

Experiment No. 10

Separation of coloured components present in the mixture of extract of coloured flowers and leaves ascending paper chromatography.

Aim : To separate coloured components present in red flower , green and yellow leaves by ascending paper chromatography and determine their R_f values .

Requirements : i) Apparatus : Chromatography paper , glass cylinder (glass jar) with a lid , capillary tube , pencil.

ii) Chemicals : A mixture of extract of red flower (red rose) , green leaves and yellow leaves and 50 % alcohol .

Procedure :

1. Take about 10 mL Of 50 % alcohol which is used as an eluent in a clean glass jar (The quantity of eluent depends upon the capacity of the jar . Its level should be about 1 cm from the bottom of the jar) . Cover it with the lid and leave for about 30 minutes for saturation of the jar .
2. Cut a paper strip of chromatography paper according to the size of the glass jar (about 14 cm) .
3. Draw a line using a pencil which is parallel to one edge of the paper strip and 2 cm above the edge . This line is called base line.
4. Draw another line at a distance of 10 cm from the base line . This line is called solvent front .
5. Using a capillary tube a drop of the given mixture (of the extract of flower and leaves) at the centre of the base line . Allow it to dry . Successively put 2 – 3 drops the same spot and allow to dry .
6. Suspend the chromatography paper strip in the jar containing the solvent (eluent) such that the lower edge of the paper dips in the solvent but the base line is above the solvent level.
7. Cover the glass jar.
8. Solvent in the jar starts rising up the paper strip . Remove the chromatography paper strip from the jar when the solvent rises up to the second line that is drawn at a distance of 10 cm from the base line.
9. Dry the chromatography paper strip. Mark the spots developed .
10. Measure the distance of the coloured spots from the base line and record the observations.
11. Calculate R_f values for each spot using the formula,
    i. Distance travelled by the spot
    ii. R_f = Distance travelled by the solvent front

Precautions :

1. Allow the spot of the given mixture to dry properly before suspending the paper strip in the jar.
2. Chromatography paper strip should remain erect when suspended in the jar.
3. Do not disturb the jar during the experiment.

Result :

R_f value for red colour	.... cm
R_f value for green colour	.... cm
R_f value for yellow colour	.... cm

Experiment No.11

Separation of Co^{2+} and Ni^{2+} ions present in the given mixture by ascending paper chromatography and determination of their R_f values.

Aim : To separate Co^{2+} and Ni^{2+} ions present in the given mixture by using ascending paper chromatography and determine their R_f values .

Requirements : i) Apparatus : Chromatography paper , capillary tube , glass cylinder or glass jar with a lid , beaker , pencil .

ii) Chemicals : Solution containing mixture of Co^{2+} and Ni^{2+} ions, acetone , concentrated HCl and distilled water, Concentrated aqueous ammonia solution , acid spray .

Diagram : -
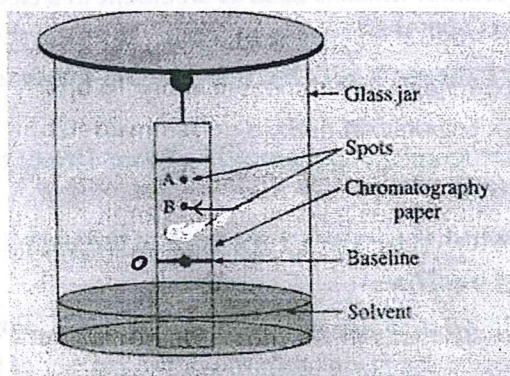
Fig. 1 : Chromatography paper suspended in a glass jar

Observation and result table :

Spot on the Chromatogram	Distance travelled by the sport cm	Distance travelled by The solvent front Cm	R_f value
Yellow orange (Co^{2+})	OA = ... cm	10 cm	OA / 10 =......... cm
Blue purple (Ni^{2+})	OB =......... cm	10 cm	OB / 10 =......... cm

Result :

R_f value for Co^2	.... cm
R_f value for Ni^{2+}	.... cm

Experiment No. 11

Separation of Co^{2+} and Ni^{2+} ions present in the given mixture by ascending paper chromatography and determination of their R_f values.

Aim : To separate Co^{2+} and Ni^{2+} ions present in the given mixture by using ascending paper chromatography and determine their R_f values .

Requirements : i) Apparatus : Chromatography paper , capillary tube , glass cylinder or glass jar with a lid , beaker , pencil .

ii) Chemicals : Solution containing mixture of Co^{2+} and Ni^{2+} ions, acetone , concentrated HCl and distilled water, Concentrated aqueous ammonia solution , acid spray .

Procedure :

1. Prepare the eluent by mixing 90 parts (by volume) acetone , 5 parts concentrated HCl , 5 parts distilled water . Take about 10 mL of the eluent in a clean jar . Cover it with the lid and leave for about 30 minutes for saturation of the glass jar.
2. Cut a paper strip of chromatography paper according to the size of the glass jar, (about 14 cm).
3. Draw a line parallel to one edge of the paper strip and 2 cm above the edge . This is the base line .
4. Draw another line at a distance of 10 cm from the base line.
5. Using a capillary tube , put a drop of the given mixture of Co^{2+} and Ni^{2+} ions at the centre of the base line . Allow the spot to dry . Successively put 2-3 drops at the same spot and allow to dry .
6. Suspend the chromatography paper strip in the jar containing the solvent such that the lower edge of the paper dips in the solvent but the base line is above the solvent level Cover the jar with lid .
7. Solvent in the jar starts rising . Remove the chromatography paper from the jar when the solvent rises up to the second line that is drawn at a distance of 10 cm from the base line.
8. Dry the paper and place it in a beaker containing aqueous ammonia for two minutes.
9. Remove the paper from the beaker and spray it with rabbinic acid . Dry it .
10. Mark the spots developed .Blue or purple spot of Ni^{2+} ions and yellow orange spot is of Co^{2+} ions .
11. Measure the distance of the spots from the base line record the observations .
    Calculate R_f value for each spot using the formula :

$$R_f = \frac{\text{Distance travelled by the spot}}{\text{Distance travelled by the solvent front}}$$

Precautions :

1. Allow the spot to dry properly before putting the chromatography paper in the jar .
2. Chromatography paper strip should remain erect when suspended in the jar .
3. Do not disturb the during the experiment .

Result :

R_f value for Co^2	.... cm
R_f value for Ni^{2+}	.... cm

PART E : PREPARATION OF INORGANIC COMPOUNDS

Experiment No. 12

Preparation of ferrous ammonium sulphate (Mohr 's salt)

Aim : To prepare a pure sample of ferrous ammonium sulphate (Mohr's salt) .

Requirements : i) Apparatus : Beakers , glass rod , evaporating dish , wire gauze , burner , tripod stand , wash bottle , test tube , pair of tongs, asbestos sheet ,funnel , filter papers , watch glass, chemical balance .

ii) Chemicals : $FeSO_4 . 7H_2O$, $(NH_4)_2 SO_4$, dilute $H_2 SO_4$, alcohol , distilled water .

Diagram : -

(a) Preparing clear solution of the given sample

(b) Filtering the solution for removing suspended impurities

(c) Heating the solution for crystallisation

(d) Cooling the solution at room temperature

Observations :

Mass of $FeSO_4.7H_2O$	1.0 g
Mass of $(NH_4)_2 SO_4$	0.5 g

Result :

Colour of crystals	
Shape of crystals	
Yield of Mohr's salt	.... g

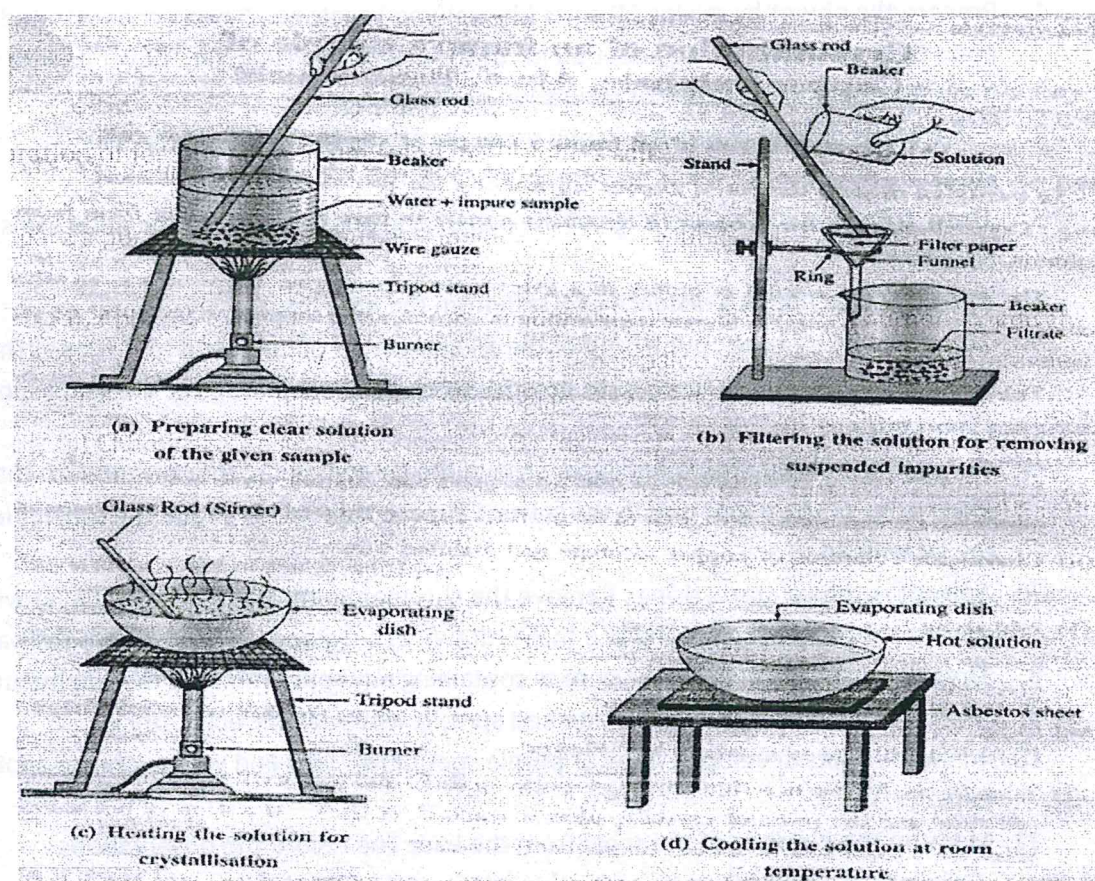## PART E : PREPARATION OF INORGANIC COMPOUNDS

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Requirements : i) Apparatus : Beakers , glass rod , evaporating dish , wire gauze , burner , tripod stand , wash bottle , test tube , pair of tongs, asbestos sheet ,funnel , filter papers , watch glass, chemical balance .

ii) Chemicals : $FeSO_4 . 7H_2O$, $(NH_4)_2 SO_4$, dilute $H_2 SO_4$, alcohol , distilled water .

Chemical Reaction :
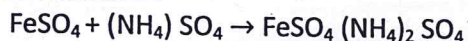
$$FeSO_4 + (NH_4) SO_4 \rightarrow FeSO_4 (NH_4)_2 SO_4$$
$$\text{1 mol} \qquad \text{1 mol}$$

When solution containing ferrous ammonium sulphate is concentrated , it crystallises.

$$FeSO_4 (NH_4)_2 SO_4 + 6H_2O \xrightarrow[\text{(crystallisation)}]{\text{concentrate}} FeSO_4 (NH_4)_2 SO_4 \, 6H_2O$$
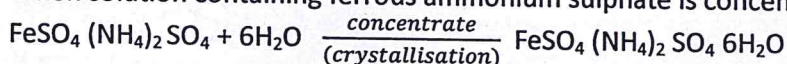
The crystals are washed with alcohol to obtain the salt in pure from.

Procedure :

1. Weigh accurately 1.0 g $FeSO_4.7H_2 O$ and 0.5 g ($NH_4)_2 SO_4$ separately on watch glass using a chemical balance. Transfer both the salt in a clean beaker and add 1-2 mL of dilute sulphuric acid.
2. Take 10 mL of water in another clean beaker and boil it
3. Add the hot water to the beaker containing the salt and dilute $H_2 SO_4$ slowly .Dissolve the contents of the beaker by stirring with a glass rod .
4. Filter the hot solution through a funnel lined with filter paper to remove insoluble impurities . Collect the filtrate in a clean beaker .
5. Transfer the filtrate containing Mohr's salt in an evaporating dish and heat the solution till the point of crystallization is achieved .(Check the crystallization point by taking the solution on glass rod . It solidifies on cooling at the crystallization point .)
6. Allow the solution to cool slowly by keeping the evaporating dish on asbestos sheet , The crystals of ferrous ammonium sulphate are obtained .
7. Separate the crystals from mother liquor by filtering . Collect the crystals on filter paper.
8. Wash the crystals with a small quantity of alcohol using a wash bottle.
9. Dry the crystals by gently pressing them between filter papers.
10. Weigh the crystals using a chemical balance and find the yield . Record the observations.

Precautions:

1. Dissolve the salts , ferrous sulphate and ammonium sulphate completely .
2. Cool the solution in the evaporating dish slowly without disturbing it .

Result :

Colour of crystals	
Shape of crystals	
Yield of Mohr's salt	.... g

Experiment No. 13

Preparation of potash alum

**Aim :** To prepare a pure sample of potash alum , $K_2 SO_4 . Al_2 (SO_4)_3 .24H_2 O$.

Requirements : i)Apparatus : Beakers , glass rod , evaporating dish , wire gauze , burner , tripod stand , wash bottle, test tube, pair of tongs, asbestos sheet , funnel, filter papers, watch glass , chemical balance.

ii)Chemicals : $K_2 SO_4$, $Al_2 (SO_2)_3 . 18H_2O$, dilute H_2SO_4 acid , ice –cold water , distilled water .

Diagram : -

(a) Preparing clear solution of the given sample

(b) Filtering the solution for removing suspended impurities

(c) Heating the solution for crystallisation

(d) Cooling the solution at room temperature

Observations:

Mass of $K_2 SO_4$	1.0 g
Mass of $Al_2 (SO_4)_3.18H_2O$	4.0 g

Result :

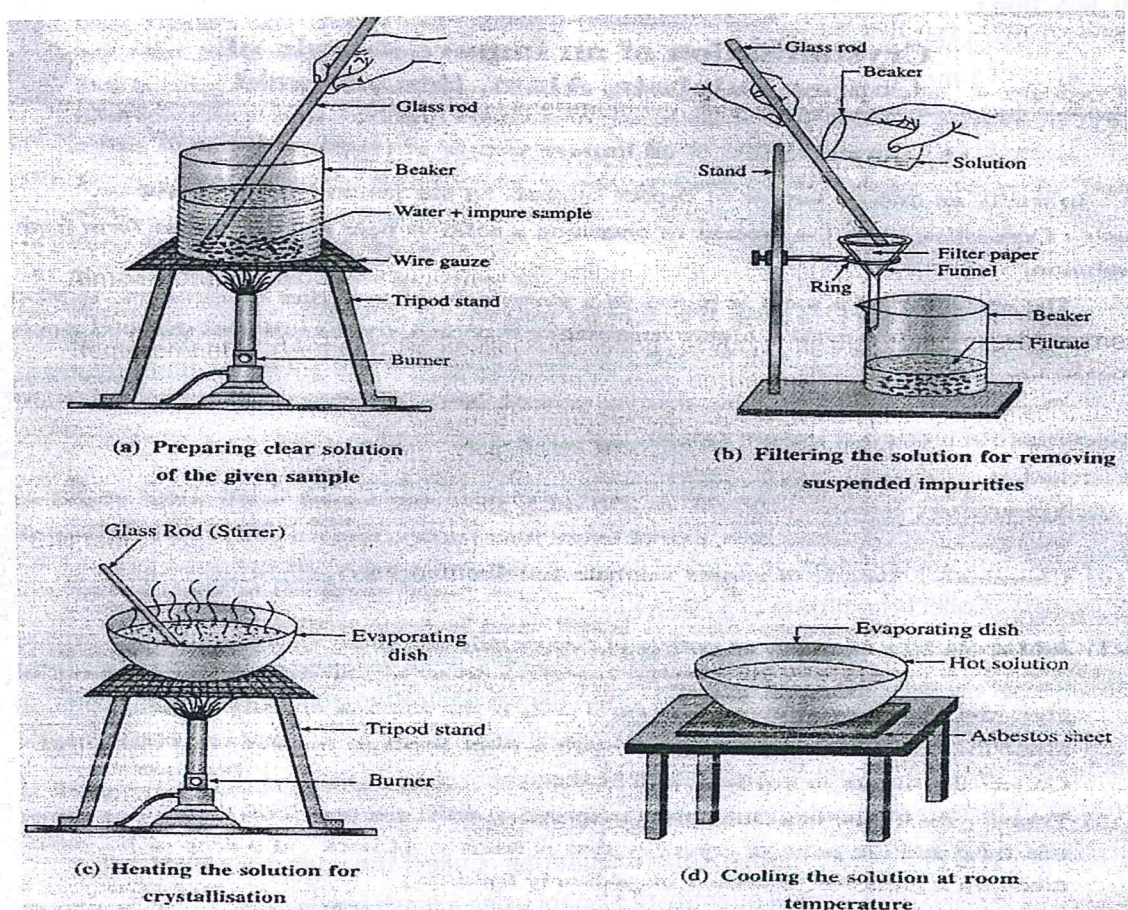| Colour of crystals	
Shape of crystals	
Yield of alum	……….gm

Experiment No. 13

Preparation of potash alum

Aim : To prepare a pure sample of potash alum , $K_2 SO_4 . Al_2 (SO_4)_3 .24H_2 O$.

Requirements : i)Apparatus : Beakers , glass rod , evaporating dish , wire gauze , burner , tripod stand , wash bottle, test tube, pair of tongs, asbestos sheet, funnel, filter papers, watch glass , chemical balance.
ii)Chemicals : $K_2 SO_4$, $Al_2 (SO_4)_3 . 18H_2O$, dilute H_2SO_4 acid , ice –cold water , distilled water.

Chemical Reaction :

$$K_2SO_4 + Al_2(SO_4)_3 \cdot 18\, H_2O + 6\, H_2O \rightarrow K_2SO_4 \cdot Al_2 (SO_4)_3 \cdot 24\, H_2O$$
$$\text{Crystals of potash Alum.}$$
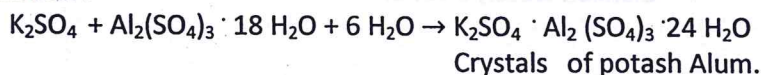
Procedure :

1. Weigh accurately 1.0 g 0f $K_2 SO_4$ and dissolve in 20 ml of distilled water in a beaker by stirring with a glass rod .
2. Weigh accurately 4.0 of $Al_2 (SO_4)_3 . 18H_2O$ and dissolve it in 20 mL of distilled water and 2 mL dilute $H_2 SO_4$. Dissolve the salt by heating for about 5 minutes with constant stirring .
3. Mix the two solutions and filter the hot solution to remove insoluble impurities .Collect. the filtrate in a clean beaker .
4. Transfer the solution to an evaporating dish and heat the solution using wire gauze till the point of Crystallization.
a. (Check the crystallization point by talking the solution on a glass rod . It solidifies on cooling at the crystallization point .)
5. Allow the solution to cool slowly by keeping the evaporating dish on asbestos sheet. The crystals of alum are obtained.
6. Separate the crystals from mother liquor by filtering . Collect the crystals on filter paper.
7. Wash the crystals with small quantity of ice-cold water using a wash bottle.
8. Dry the crystals gently by pressing them in folds of filter paper.
9. Find the yield of alum using a chemical balance . Record the observations.

Precautions :

1. Dissolve the salts completely.
2. Cool the solution of evaporating dish slowly without disturbing it .

Result :

Color of crystals	
Shape of crystals	
Yield of alum	.... g

Experiment No. 14

Preparation of potassium trioxalatoferrate (III).

Aim : To prepare a pure sample of potassium trioxalatoferrate (III) .

Requirements : i) Apparatus : Beakers, glass rod , evaporating dish, tripod stand, wire gauze, sand bath, pair of tongs, asbestos sheet, burner, funnel, filter papers, wash bottle, watch glass, measuring cylinder.

ii) Chemicals : $FeCl_3$, KOH, $H_2C_2O_4$ (oxalic acid) . $K_2C_2O_4 . H_2O$ (potassium oxalate) , distilled water, alcohol .

Diagram :-

(a) Preparing clear solution of the given sample

(b) Filtering the solution for removing suspended impurities

(c) Heating the solution for crystallisation

(d) Cooling the solution at room temperature

Observations :

Mass of KOH	1.15 g
Mass of $FeCl_3$	1.00 g
Mass of hydrated Oxalic acid	1.15 g
Mass of hydrated Potassium oxalate	1.60 g

Result :

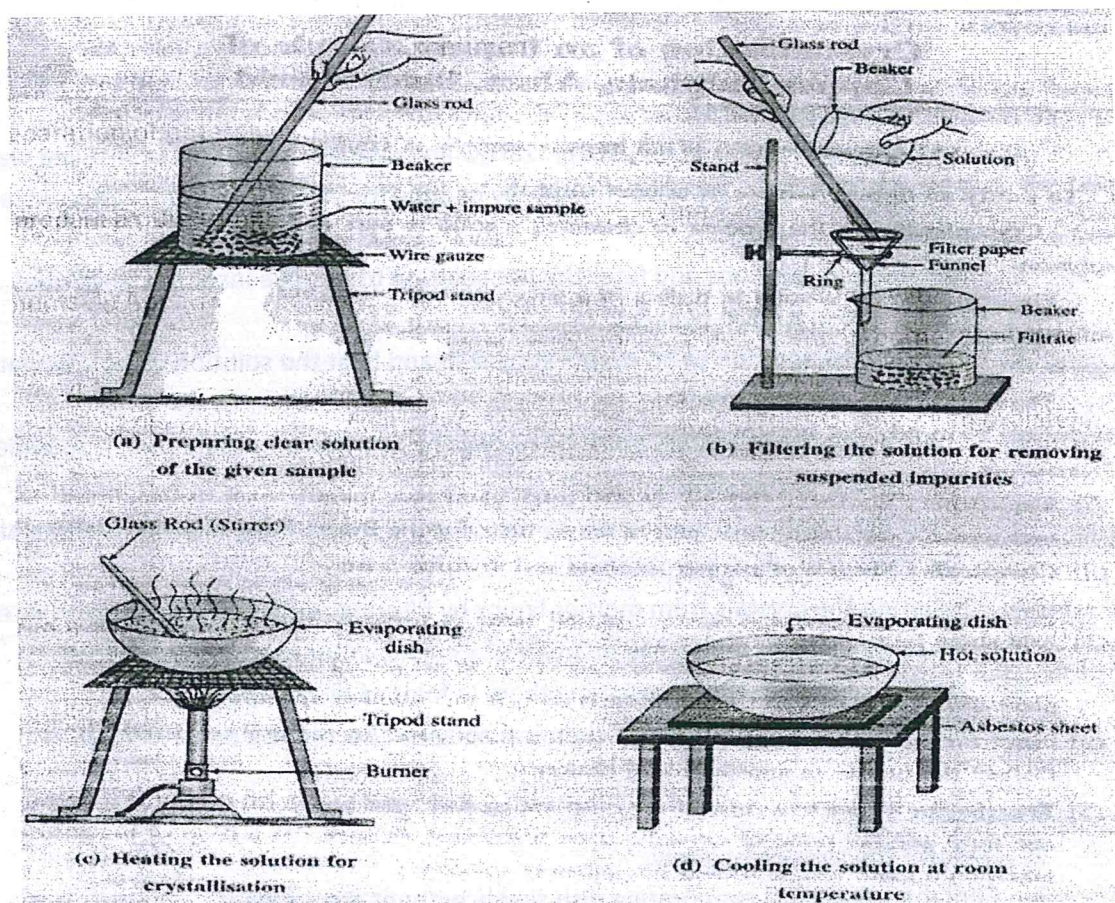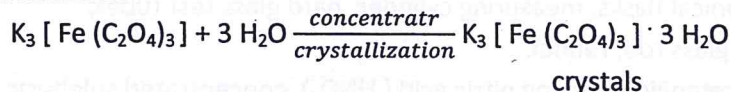| Colour of crystals	
Yield of crystals	.... g

Experiment No. 14

Preparation of potassium trioxalatoferrate (III).

Aim : To prepare a pure sample of potassium trioxalatoferrate (III) .

Requirements : i) Apparatus : Beakers, glass rod , evaporating dish, tripod stand, wire gauze , sand bath, pair of tongs, asbestos sheet, burner, funnel, filter papers, wash bottle, watch glass, measuring cylinder.

ii) Chemicals : $FeCl_3$, KOH, $H_2 C_2 O_4 . K_2 C_2 O_4 . H_2O$, distilled water, alcohol .

Chemical Reaction :-

$$K_3 [Fe (C_2O_4)_3] + 3 H_2O \xrightarrow[crystallization]{concentratr} K_3 [Fe (C_2O_4)_3] \cdot 3 H_2O$$

crystals

Procedure :

1. Weigh 1.15 g hydrated oxalic acid ($H_2 C_2 O_4 .2H_2 O$) and 1.6 g hydrated potassium oxalate ($K_2 C_2 O_4. H_2 O$) separately on watch glass . Transfer oxalic acid and potassium oxalate to a clean beaker . Add 30 mL of distilled water using a measuring cylinder .Stir with a glass rod and dissolve the solid. Warm the solution in the beaker to obtain a clear solution.

2. Weigh 1.15 g KOH and transfer the solid to another clean beaker . Add 15 mL of distilled water Stir with a glass rod till it dissolves.

3. Weigh 1.0 g of anhydrous $FeCl_3$ and dissolve in 15 mL distilled water by stirring with a glass rod .

4. Transfer KOH solution to the $FeCl_3$ solution taking small quantity of KOH at a time . Stir continuously . Fe (OH)$_3$ precipitates out.

5. Filler the precipitate using filter paper and funnel.

6. Collect the precipitate, $(Fe(OH)_3$ on a filter paper. Wash the precipitate using small amount of distilled water from a wash bottle.

7. Add this precipitate of $Fe(OH)_3$ to the clear solution of mixture of oxalic acid and potassium oxalate (prepared in step 1) . Stir with a glass rod to dissolve the precipitate.

8. Filter the solution through funnel with filter paper to remove insoluble impurities.

9. Transfer the filtrate to an evaporating dish . Concentrate the solution by heating the evaporating dish on a sand bath till the crystallization point is reached. (Check by taking the solution on a glass rod . It solidifies on cooling at the crystallization point.)

10. Allow the solution to cool slowly by keeping the evaporating dish on asbestos sheet, Crystals of the complex are formed.

11. Separate the crystals from mother liquor by filtering . Collect the crystals on filter paper.

12. Wash the crystals with small amount of ethyl alcohol using a wash bottle.

13. Dry the crystals by gently pressing them between the filter papers . Weigh the crystals and obtain yield of the complex.

Precautions:

1. Dissolve various solids completely.

2. Cool the solution in evaporating dish slowly.

Result :

Colour of crystals	
Yield of crystals	...... g

PART F : PREPARTION OF ORGANIC COMPOUNDS

Experiment No. 15

Preparation of p- nitro acetanilide from acetanilide

Aim : To prepare a pure sample of p-nitro acetanilide from acetanilide.

Requirements : i) Apparatus : Conical flasks, measuring cylinder, hard glass test tubes,
glass rod, funnel.

ii) Chemicals : Acetanilide, fuming nitric acid (HNO_3), concentrated sulphuric
acid (H_2SO_4), glacial acetic acid (CH_3COOH), methylated spirit or
ethyl alcohol, freezing mixture (mixture of ice and salt), crushed
ice.

Diagram : -

Observations and result :

• Colour of crystal	
• Yield of p- nitro acetanilide	.... g

PART F : PREPARTION OF ORGANIC COMPOUNDS
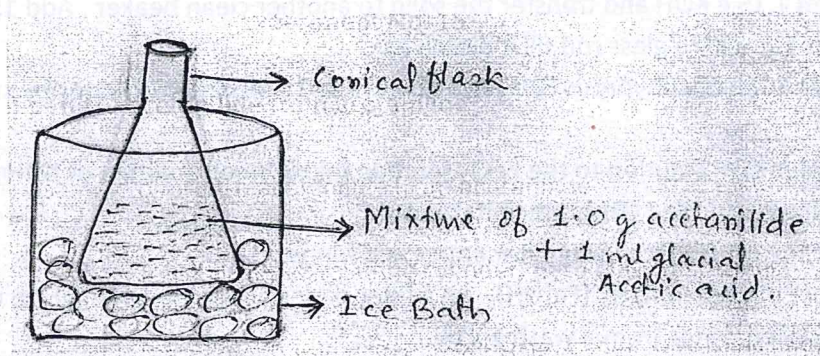
Experiment No. 15

Preparation of p- nitroacetanilide from acetanilide

Aim : To prepare a pure sample of p-nitroacetanilide from acetanilide.

Requirements : i)Apparatus : Conical flasks, measuring cylinder, hard glass test tubes, glass rod, funnel.

ii) **Chemicals :** Acetanilide, fuming nitric acid (HNO_3), concentrated sulphuric acid (H_2SO_4), glacial acetic acid (CH_3COOH), methylated spirit or ethyl alcohol, freezing mixture (mixture of ice and salt), crushed ice.

Chemical reaction : -
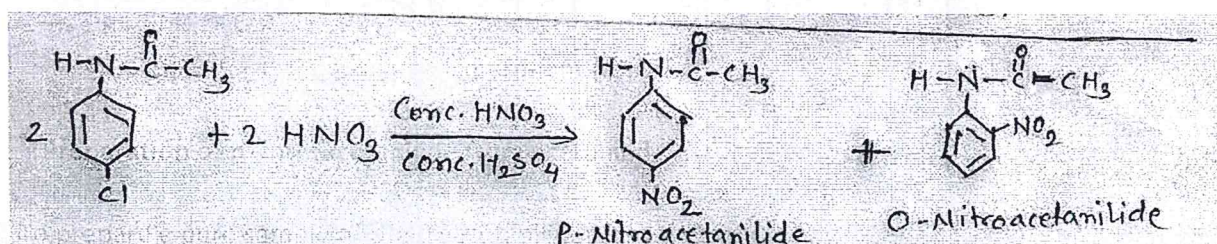
Procedure:

1. Dissolve 1.0 g acetanilide in 1 mL of glacial acetic acid in a conical flask with stirring Keep the flask in a freezing mixture.
2. Prepare nitrating mixture as follows : In a hard glass test tube take 1 mL of fuming nitric acid and cool it by keeping it in a freezing mixture. Add 1 mL of concentrated sulphuric acid drop wise , with constant stirring, while keeping the test tube in the freezing mixtures.
3. Add 1 mL of concentrated sulphuric acid drop wise to the conical flask while keeping it in the freezing mixture.
4. Add nitrating mixture from the test tube drop wise to the solution in the conical flask with constant stirring and keeping the flask in the freezing mixture.
5. Cool the flask for 15 minutes by keeping it in the freezing mixture.
6. Remove the flask from the freezing mixture and keep it outside for 30 minutes.
7. Pour the reaction mixture from the conical flask to a beaker containing crushed ice and stir it.
8. A mixture of p-nitro acetanilide and o-nitroacetanilide crystals is formed.
9. Filter and wash the solid with cold water.
10. Recrystallize the product by adding a small quantity of methylated spirit or ethyl alcohol to the product . Warm and filter . On cooling , crystals of p-nitrocetanilide are obtained while o-nitrocetanilide being soluble remains in the mother liquor.
11. Filter the crystal and dry them by pressing gently in the folds of filter paper.
12. Collect the dry crystals on a dry watch glass and weigh.

Precautions : Do not inhale fuming nitric acid.

Observations and result :

• Colour of crystal	
• Yield of p- nitroacetanilide	.... g

Experiment No. 16

Preparation of 2 – naphthol aniline dye

Aim : To prepare 2 naphthol anline dye.

Requirements: i) Apparatus : Conical flask, beaker, measuring cylinder, glass rod, funnel
hardglass test tubes , freezing mixture , ice bath.

ii) Chemicals : Anline, sodium nitrite ($NaNO_2$) , concentrated hydrochloric acid
2-naphthol, 10% NaOH, distilled water.

Diagram : -

Observations and result:

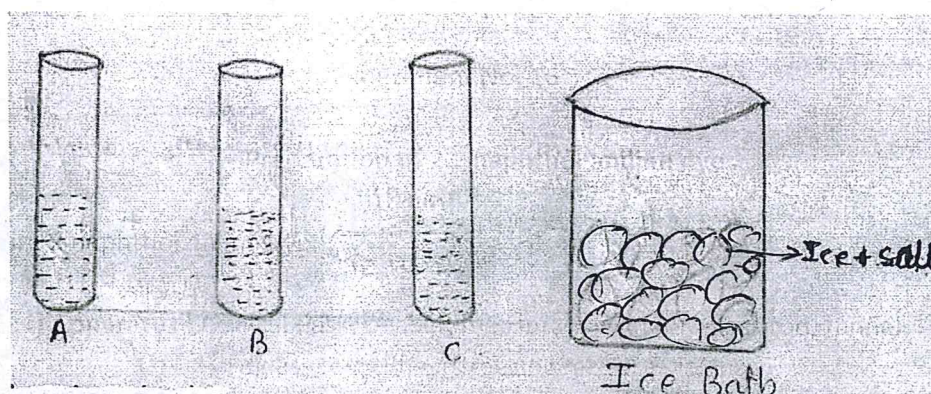| Colour of the dye	
Mass of 2-naphthol anline dye	.... g

Experiment No. 16

Preparation of 2 – naphthol aniline dye

Aim : To prepare 2 naphthol aniline dye.

Requirements: i) Apparatus : Conical flask, beaker, measuring cylinder, glass rod, funnel hard glass test tubes ,freezing mixture , ice bath.

ii) Chemicals : Aniline, sodium nitrite ($NaNO_2$) , concentrated hydrochloric acid 2-naphthol, 10% NaOH, distilled water.

Chemical Reaction : -
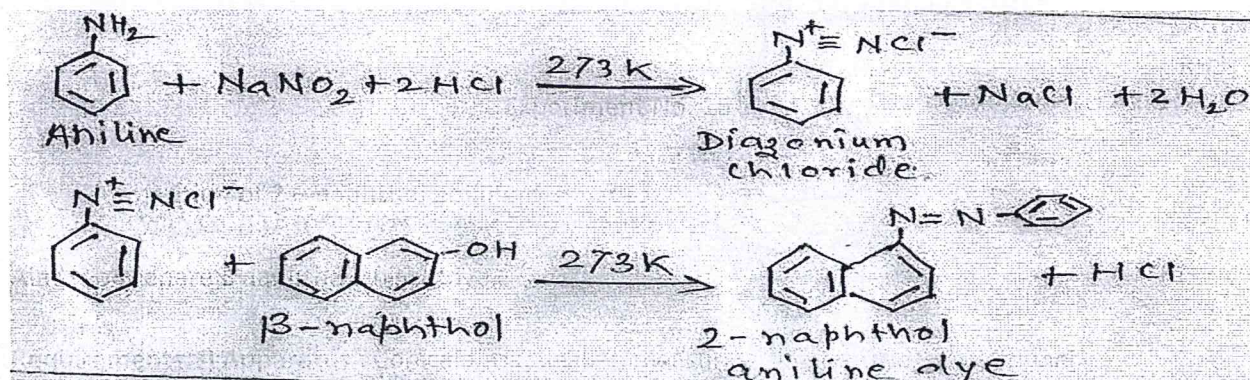
Procedure:

1. Take three hard glass test tubes and label them as A,B and C.
2. Take 1 mL aniline, 2.5 mL concentrated HCl and 5 mL distilled water in test tube A. Cool the test tube in ice bath.
3. In test tube B , take 1.0 g $NaNO_2$ and dissolve it in 5 mL of distilled water, cool the test tube in the ice bath.
4. Dissolve 2.0 g β -naphthol in 15 mL of 10 % NaOH solution in test tube 'C' . Cool the tube in ice bath .The solution of β-naphthol in NaOH is ready.
5. Add $NaNO_2$ solution slowly from test tube 'B' to test tube 'A' containing aniline and concentrated HCl,so that diazonium salt is formed. Add this reaction mixture slowly to the solution of β -naphthol in NaOH (in test tube C) with constant stirring . An orange red dye (benzeneazo-2-naphthol) is formed.
6. Keep this reaction mixture in freezing mixture for 30 minutes . Crystals of the dye are obtained.
7. Filter and wash the crystals with water.
8. Dry the crystals with filter paper, transfer to a watch glass and weigh the crystals.

Observations and result:

Colour of the dye	
Mass of 2-naphthol aniline dye	.... g

Experiment No. 17

Preparation of phthalic anhydride

Aim : To prepare phthalic anhydride from phthalic acid.

Requirements : i) Apparatus : Evaporating dish, funnel, filter paper , wire gauze , tripod stand ,burner, watch glass.

ii) Chemicals: Phthalic acid.

Diagram : -

→ Inverted Funnel
→ filter paper having holes
→ 2.0gm phthalic acid
→ wire gauge
→ Tripod stand
→ Burner

Observations and result :

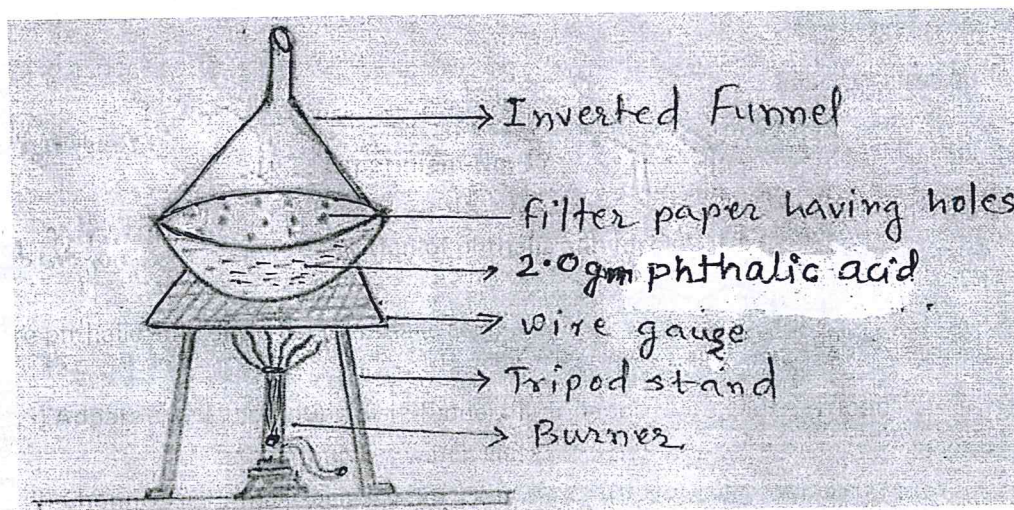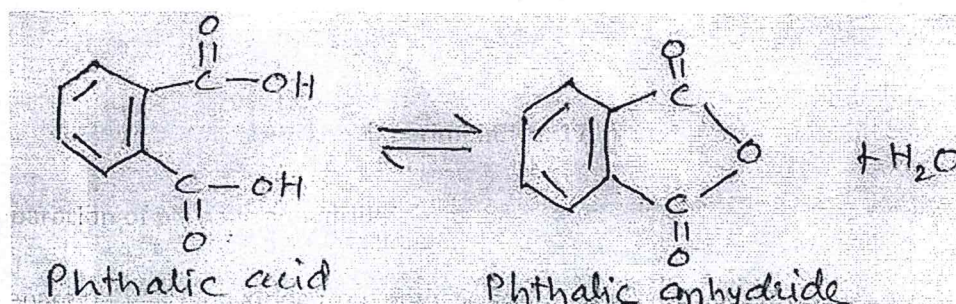| Colour of crystals	
Shape of crystals	
Yield of phthalic anhydride	.... g

Experiment No. 17

Preparation of phthalic anhydride

Aim : To prepare phthalic anhydride from phthalic acid.

Requirements : i) Apparatus : Evaporating dish, funnel, filter paper , wire gauze , tripod stand ,burner, watch glass.

ii) **Chemicals:** Phthalic acid.

Chemical Reaction : -

Prpcedure :

1. Take 2.0 g of phthalic acid in an evaporating dish and cover it with filter paper having several holes.
2. Cover the evaporating dish and filter paper with an inverted funnel.
3. Heat the evaporating dish slowly by using wire gauze and tripod stand.
4. Crystals of phthalic anhydride are deposited on the filter paper.
5. Collect the crystals on a watch glass and weigh.

Precautions: Heat the evaporating dish solely using low of the burner.

Observations and result :

Colour of crystals	
Shape of crystals	
Yield of phthalic anhydride	.... g

Experiment No. 18

Preparation of succinic anhydride

Aim : To prepare succinic anhydride from succinic acid.

Requirements : i) Apparatus : Evaporating dish, filter paper, funnel, wire gauze, tripod stand, burner, watch glass.

ii) Chemicals: succinic acid , acetic anhydride.

Diagram :-

- → Inverted Funnel
- Filter paper having holes
- → 2·0 gm succinic acid + 1 ml acetic acid .
- → wire gauge
- → Tripod stand
- → Burner

Observations and result :

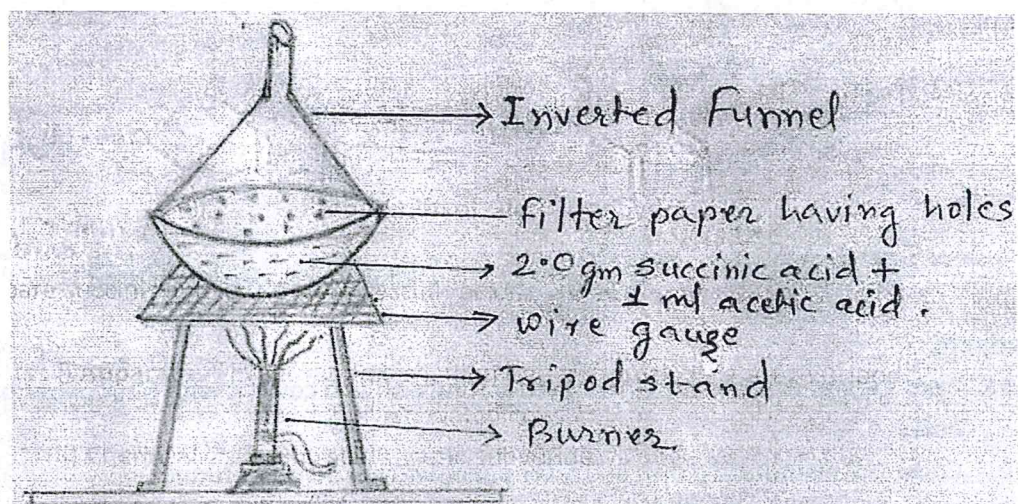|	
• Colour of crystal	
• Shape of crystals	
• Yield of succinic anhydride	.... g

Experiment No. 18

Preparation of succinic anhydride

Aim : To prepare succinic anhydride from succinic acid.

Requirements : i) Apparatus : Evaporating dish, filter paper, funnel, wire gauze, tripod stand, burner, watch glass.

ii) Chemicals: succinic acid , acetic anhydride.

Chemical Reaction : -

Succinic acid + Acetic anhydride → (heat) → Succinic anhydride + 2 CH₃COOH (Acetic acid)
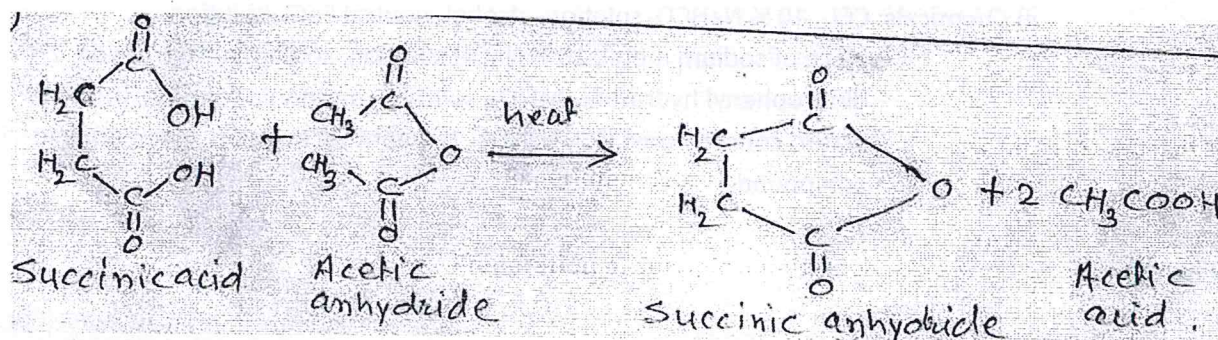
Procedure :

1. Take 2.0 g succinic acid and 1 mL of acetic anhydride in an evaporating dish.
2. Cover the evaporating dish with filter paper having several holes and invert a funnel over it so as to cover the evaporating dish.
3. Heat the evaporating dish slowly by using wire gauze and tripod stand.
4. Crystals of succinic anhydride are deposited on the filter paper.
5. Collect the crystals on a watch glass and weigh them.

Precautions : Heat the evaporating dish slowly using a low flame of the burner.

Observations and result :

• Colour of crystal	
• Shape of crystals	
• Yield of succinic anhydride	.... g

PART G : TESTS FOR FUNCTIONAL GROUPS
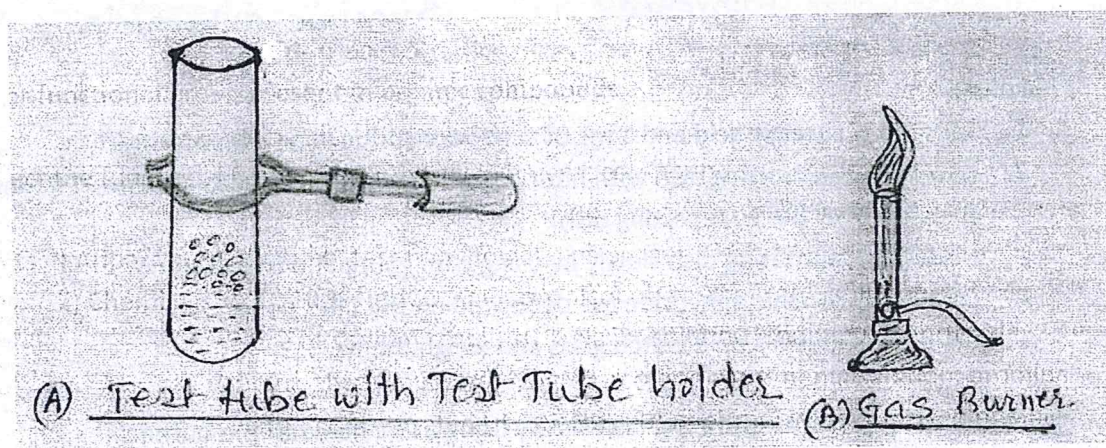
Experiment 19 (A)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) **Chemicals:** CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner.

Result : 1) The functional group of the given compound is ....
2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS

Experiment 19 (A)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

   ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected .
Use organic substance directly.

Test	Observation	Inference
1)Substance + saturated $NaHCO_3$	Effervescence of CO_2	Carboxyl group present -C-OH ‖ O

Reaction :

$$R-C-OH + NaHCO_3 \longrightarrow R-C-O-Na + H_2O + CO_2 \uparrow$$

(with O above each C as $\overset{O}{\overset{\|}{}}$)

Precaution : Be careful while using organic compounds and sodium metal.

Result : 1) The functional group of the given compound is ....

   2) The structure formula of the functional group is ....

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PART G : TESTS FOR FUNCTIONAL GROUPS
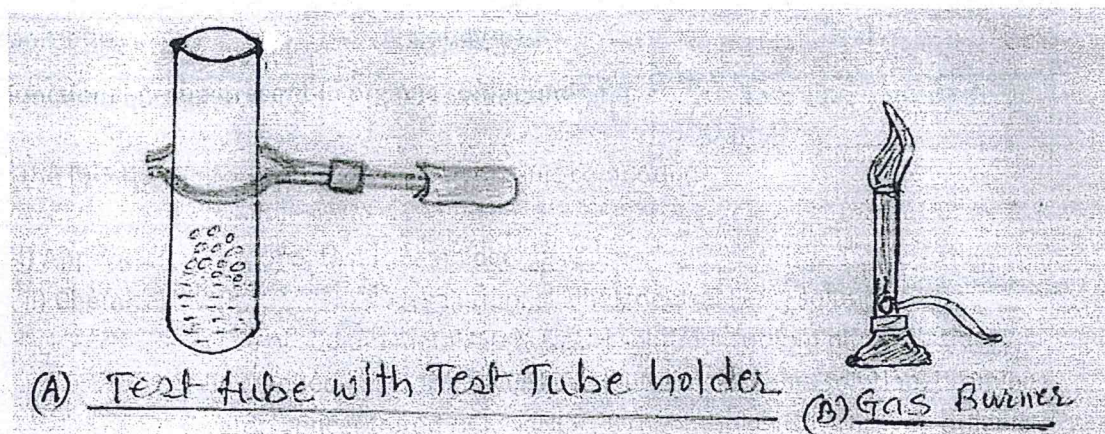
Experiment 19 (B)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) **Chemicals:** CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner

Result : 1) The functional group of the given compound is ....
2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS

Experiment 19 (B)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

　　　　　　　ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected .
　　　　　　Use organic substance directly.

Test	Observation	Inference
1)Substance + saturated 　　　　　　　$NaHCO_3$	No Effervescence of CO_2	Carboxyl group absent $\begin{array}{c} -C-OH \\ \parallel \\ O \end{array}$
2) Substance (in a dry test tube) + a small piece of sodium metal	Effervescence of H_2	Alcoholic hydroxyl group – OH Present

Reaction :

$$2 R - OH + 2 Na \longrightarrow 2 R-O-Na + H. \uparrow$$

Precaution : Be careful while using organic compounds and sodium metal.

Result :　1) The functional group of the given compound is ....
　　　　　2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS
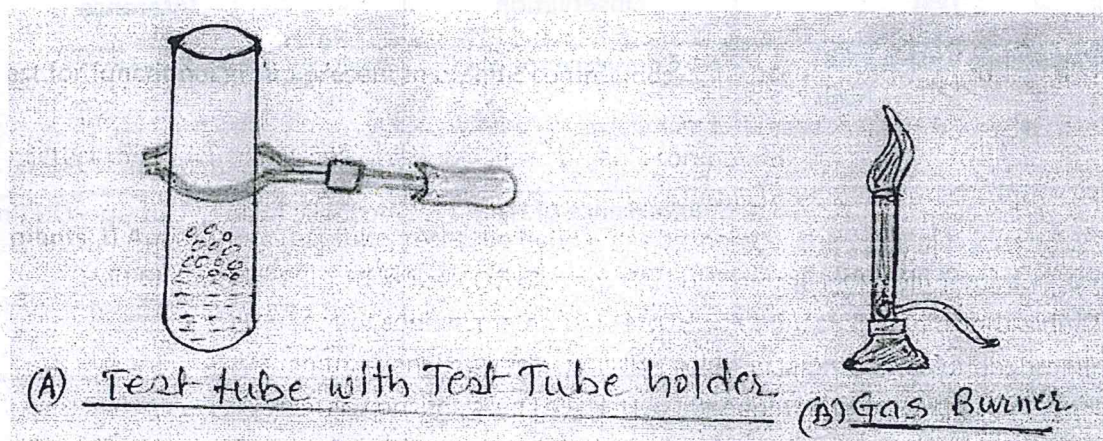
Experiment 19 (C)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner

Result : 1) The functional group of the given compound is ....

2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS

Experiment 19 (C)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

        ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected .
        Use organic substance directly.

Test	Observation	Inference
1)Substance + saturated $NaHCO_3$	No Effervescence of CO_2	Carboxyl group absent -C-OH II O
2) Substance (in a dry test tube) + a small piece of sodium metal	No Effervescence of H_2	Alcoholic hydroxyl group – OH absent
3)Substance + alcohol + neutral $FeCl_3$	Violet or bluish green colouration	Phenolic group Ar – OH group Is present
Reactions : $6\ C_6H_5\ OH\ + FeCl_3\ \longrightarrow [\ Fe\ (\ OC_6\ H_5\ )_6\ ]^{3-} + HCL + 3\ H^+$		

Precaution : Be careful while using organic compounds and sodium metal.

Result : 1) The functional group of the given compound is ....
          2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS
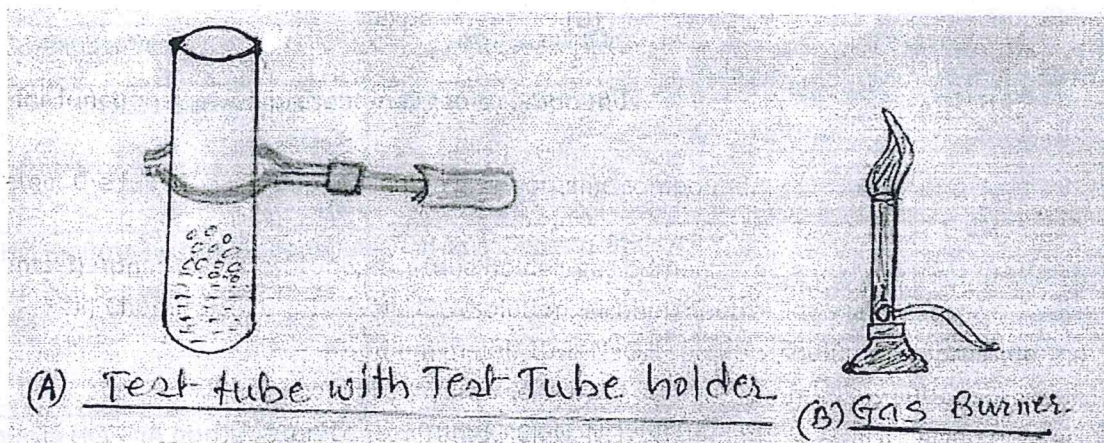
Experiment 19 (D)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

             ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner.

Result : 1) The functional group of the given compound is ....
            2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS

Experiment 19 (D)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected . Use organic substance directly.

Test	Observation	Inference
1)Substance + saturated $NaHCO_3$	No Effervescence of CO_2	Carboxyl group absent -C-OH II O
2) Substance (in a dry test tube) + a small piece of sodium metal	No Effervescence of H_2	Alcoholic hydroxyl group – OH absent
3)Substance + alcohol + neutral $FeCl_3$	No Violet or bluish green colouration	Phenolic group Ar – OH group Is absent
4)Substance + CCl_4, dissolve + Bromine water drop wise	Brown colour of solution Disappears	Unsaturation $>C = C<$) or ($-- C \equiv C--$) present

Reaction :-

$>C = C < + Br_2 \longrightarrow$

$$\underset{\text{colourless}}{-\overset{\overset{Br}{|}}{C}-\overset{\overset{Br}{|}}{C}-}$$

Alkene   Bromin

(ii) $-- C \equiv C -- + 2 Br_2 \longrightarrow$

$$\underset{\text{colourless}}{-\overset{\overset{Br}{|}}{\underset{\underset{Br}{|}}{C}}-\overset{\overset{Br}{|}}{\underset{\underset{Br}{|}}{C}}-}$$

Alkyne   Bromin

Precaution : Be careful while using organic compounds and sodium metal.

Result : 1) The functional group of the given compound is ....

2) The structure formula of the functional group is ....

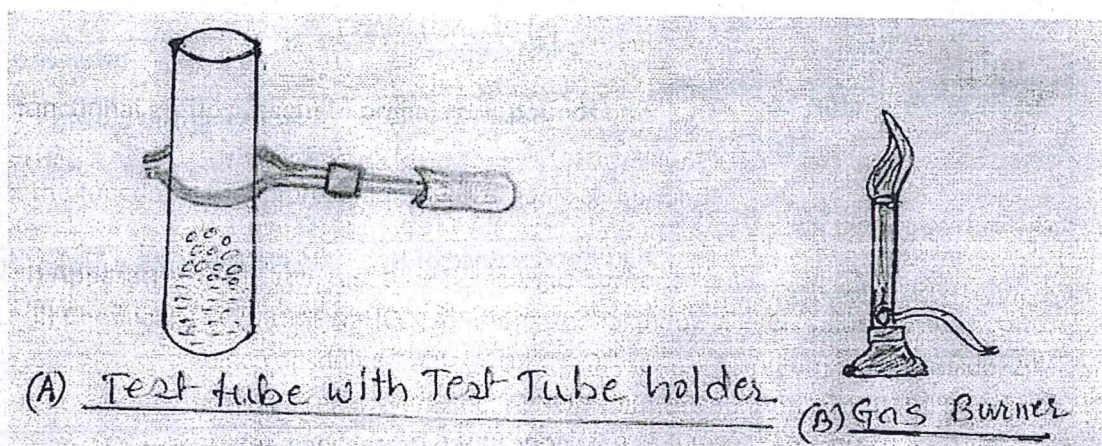## PART G : TESTS FOR FUNCTIONAL GROUPS

Experiment 19 (E)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

      ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner

Result : 1) The functional group of the given compound is ....
           2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS
Experiment 19 (E)
Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: **i) Apparatus :** Test tube, test tube holder, gas burner.

ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected .
Use organic substance directly.

Test	Observation	Inference
1) Substance + saturated NaHCO₃	No Effervescence of CO_2	Carboxyl group absent $-\overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}}-OH$
2) Substance (in a dry test tube) + a small piece of sodium metal	No Effervescence of H_2	Alcoholic hydroxyl group – OH absent
3) Substance + alcohol + neutral FeCl₃	No Violet or bluish green colouration	Phenolic group Ar – OH group Is absent
4) Substance + CCl₄, dissolve + Bromine water drop wise	No Brown colour of solution Disappears	Unsaturation >C = C<) or (-- C ≡ C--) absent
5) Substance + NaOH+ Sodium nitroprusside	Red colouration	Ketonic group $C - \overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}} - C$ present

Reactions:

$$CH_3 - \overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}} - CH_3 + OH^- \rightarrow CH_3 - \overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}} - CH_2^- + H_2O$$

$$CH_3 - \overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}} - CH_2^- + [Fe(CN)_5 NO]^{2-} \rightarrow [Fe(CN)_5NO\ CH_3 - \overset{\displaystyle O}{\underset{\displaystyle \parallel}{C}} - CH_2]^{3--}$$

Nitroprusside ion   Red colouration

Precaution : Be careful while using organic compounds and sodium metal.

Result : 1) The functional group of the given compound is ……………
2) The structure formula of the functional group is ……

PART G : TESTS FOR FUNCTIONAL GROUPS
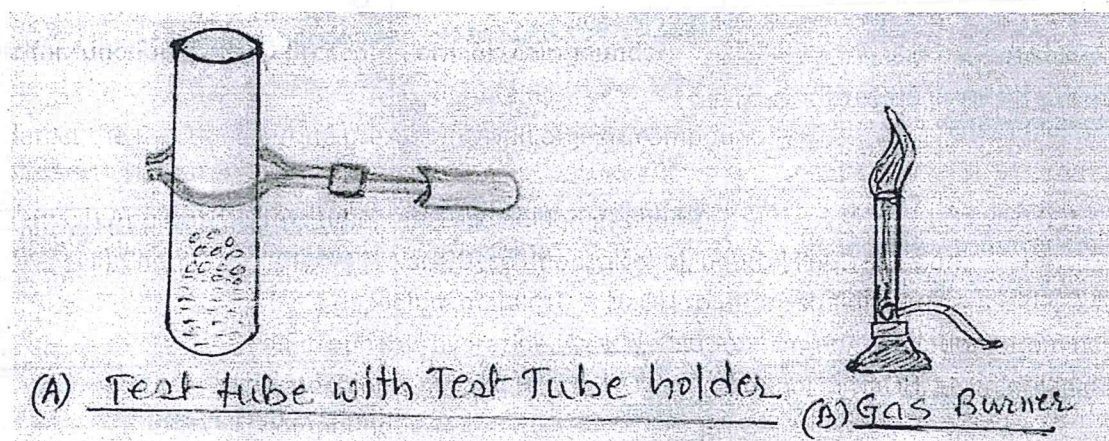
Experiment 19 (F)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitrophenyl hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β -naphthol in NaOH, given organic compound.

Diagram :-

(A) Test tube with Test Tube holder   (B) Gas Burner

Result : 1) The functional group of the given compound is ....
2) The structure formula of the functional group is ....

PART G : TESTS FOR FUNCTIONAL GROUPS
Experiment 19 (F)

Test for functional group present in organic compounds

Aim : To detect the functional group present in given organic compound.

Requirements: i) Apparatus : Test tube, test tube holder, gas burner.

ii) Chemicals: CCl_4, 10 % $NaHCO_3$ solution, alcohol, neutral $FeCl_3$ solution, piece of sodium metal, 10 % NaOH solution, sodium nitroprusside, 2,4-dinitropheny l hydrazine, $AgNO_3$, solution, ammonia solution, bromine water, concentrated HCl, $NaNO_2$, β-naphthol in NaOH, given organic compound.

Procedure : Carry out the following tests in serial order till a functional group is detected .
Use organic substance directly.

Test	Observation	Inference
1)Substance + saturated $NaHCO_3$	No Effervescence of CO_2	Carboxyl group absent $-C-OH$ $\overset{\|}{\underset{O}{}}$
2) Substance (in a dry test tube) + a small piece of sodium metal	No Effervescence of H_2	Alcoholic hydroxyl group – OH absent
3)Substance + alcohol + neutral $FeCl_3$	No Violet or bluish green colouration	Phenolic group Ar – OH group Is absent
4)Substance + CCl_4, dissolve + Bromine water drop wise	No Brown colour of solution Disappears	Unsaturation $>C = C<$) or ($-- C \equiv C--$) absent
5)Substance + NaOH+ Sodium nitroprusside	No Red colouration	Ketonic group $C - C - C$ Absent $\overset{\|}{\underset{O}{}}$
6) Substance + Tollen's Reagent, warm	Silver mirror formed on inner Side of the test tube	Aldehydic group present $H - C -$ $\overset{\|}{\underset{O}{}}$

[Preparation of Tollen's reagent : 2 mL $AgNO_3$ + 1 – 2 drops NaOH . Add dil. NH_4OH solution drop wise with shaking till the precipitate just dissolved.]

Reaction:

$$R - C\overset{}{\underset{\overset{\|}{O}}{}}-H + 2 [Ag (NH_3)_2] OH \xrightarrow{\text{Warm}} R - C\overset{}{\underset{\overset{\|}{O}}{}} - OH + 2 Ag + 4NH_3 + H_2 O$$
silver mirror

Precaution : Be careful while using organic compounds and sodium metal.

Result : 1) The functional group of the given compound is ……………

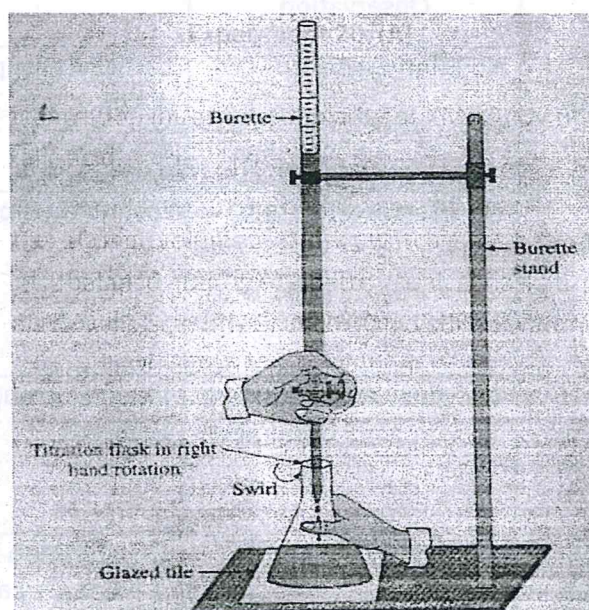2) The structure formula of the functional group is …………

PART H : VOLUMETRIC ANALYSIS
Experiment 20 (A)

Aim : To determine the concentration in terms of molarity of $KMnO_4$ by titrating it against a standard solution of 0.1 M oxalic acid.

Requirements : i) Apparatus : 100 mL volumetric flask , watch glass beaker , glass rod , balance . Burette, Pipette , conical flask, tripod stand , etc.

ii) Chemicals : Oxalic acid ($H_2 C_2 O_4.2H_2O$), distilled water. 0.1 M oxalic acid ,dil. $H_2 SO_4$, $KMnO_4$ solution.

Chemical reactions : $2KMnO_4 + 3H_2 SO_4 \rightarrow K_2 SO_4 + 2MnSO_4 + 8H_2 O + 10CO_2$

Diagram : -

Observation table :

Solution in burette : $KMnO_4$. (Unknown)

Solution by pipette : 0.1 M oxalic acid solution 10 mL oxalic acid)

Solution in the conical flask : 10 mL oxalic acid + 1 test tube dil . $H_2 SO_4$

Precaution : Heat the solution before the titration .

End point : Colourless to faint pink

Molar mass of $KMnO_4$ = 158 g mol^{-1}

Molarity of oxalic acid = 0.1 M

Pilot reading : .... To .... mL

PART H : VOLUMETRIC ANALYSIS

Experiment 20 (A)

Titration of KMnO₄ against standard solution of oxalic acid :

Aim : To determine the concentration in terms of molarity of $KMnO_4$ by titrating it against a standard solution of 0.1 M oxalic acid.

Preparation of a standard solution of 0.1 M oxalic acid ($H_2 C_2 O_4 . 2H_2O$) :

Molar mass of oxalic acid ($H_2 C_2 O_4 . 2H_2O$) = 126 g mol⁻¹

For 1000 mL of 1 M oxalic acid requires 126 g oxalic acid

100 mL of 0.1 M oxalic acid requires,

$$= \frac{100 \times 0.1 \times 126}{1000} = 1.26 \text{ g oxalic acid ,}$$

by weighing 1.26 gm o.A o.1m solution was prepared

Requirements : i)Apparatus : 100 mL volumetric flask , watch glass, beaker , glass rod balance . Burette, Pipette , conical flask, tripod stand, , etc.

ii) Chemicals : Oxalic acid ($H_2 C_2 O_4 . 2H_2O$), distilled water. 0.1 M oxalic acid , dil. $H_2 SO_4$ $KMnO_4$ solution.

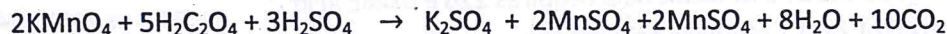**Chemical reactions :** $2KMnO_4 + 3H_2 SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2 O + 10CO_2$

Procedure :

1. Fill the burette with $KNnO_4$ solution .
2. Pipette out 10 mL of 0.1 M oxalic acid solution in a conical flask and add one test tube
3. of dil . H_2SO_4 . Then heat the solution to about 60⁰C to 70⁰ C on a wire gauze , since the reaction is very slow at room temperature.
4. Add $KMnO_4$ solution from burette, 1 mL at a time to the hot solution stirring continuously till the faint pink colour appears.
5. Record the range burette reading as a pilot reading.
6. Fill the burette with $KMnO_4$ up to zero level. Pipette out 10 mL 0.1 M oxalic acid in the conical flask and repeat the titration.
7. [Hint : Add 1 mL $KMnO_4$ at a time till 3 mL above the pilot reading and then for accuracy add drop wise till the solution acquires faint pink colour.]
8. This gives the volume of $KMnO_4$ required to titrate 10 mL oxalic acid.
9. Repeat the procedure thrice and obtain the constant burette reading (CBR).

Result : -

1) The strength of $KMnO_4$ solution = x = ... g L⁻¹
2) Molarity of $KMnO_4$ solution = y = ... M

60

Obs. No.	Volume of oxalic acid (0.1m) Solution by pipette (ml)	Volume of KMnO₄ solution by burette (ml)		Constant burette reading (CBR) (ml)
		Initial reading	Final reading	
1	10 ml	0.0 ml	.... ml	
2	10 ml	0.0 ml	.... ml	.... ml
3	10 ml	0.0 ml	.... ml	

Calcuations :

$$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 2MnSO_4 + 8H_2O + 10CO_2$$

2 mol   5 mol

$$10 \text{ mL } 0.1 \text{ M } H_2C_2O_4 = \frac{2 \times 158 \times 10 \times 0.1}{1000 \times 5} = 0.0632 \text{ g } KMnO_4$$

Now, **1) For strength : -**

10 mL 0.1 M $H_2C_2O_4$ ≡ (CBR) mL $KMnO_4$

(CBR) mL $KMnO_4$ ≡ 0.0632 g $KMnO_4$

100 mL $KMnO_4$ ≡ $\dfrac{0.0632 \times 1000}{(CBR)}$ = ...

$$x = ... \text{ g L}^{-1}$$

Strength of $KMnO_4$ solution = x = ... g L⁻¹

2.For Molarity : -

$$\text{Molarity of KMnO}_4 \text{ solution} = \frac{\text{Strength of KMnO}_4 \text{ solution}}{\text{Molar mass}}$$

$$y = \frac{x}{158} = ... \text{ M of } KMnO_4$$

Result : -

1) The strength of $KMnO_4$ solution = x = ... g L⁻¹
2) Molarity of $KMnO_4$ solution = y = ... M
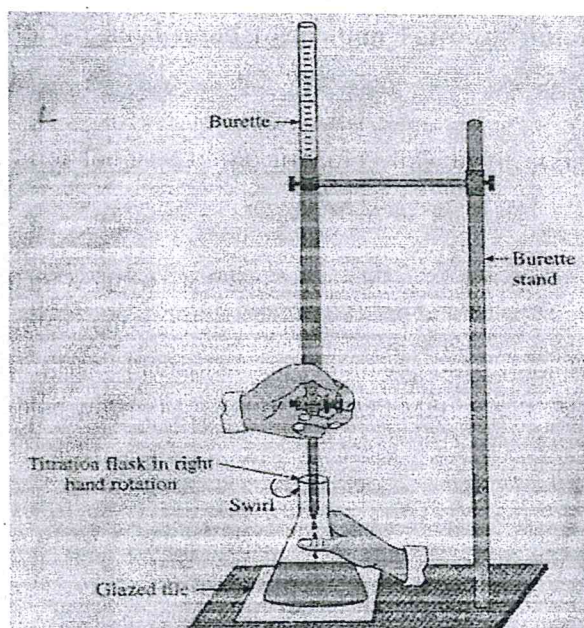
Experiment 20 (B)

(B) Titration of KMnO₄ against standard solution Ferrous ammonium sulphate (or Mohr's salt) :

Aim : To determine the concentration in terms of molarity of $KMnO_4$ by titrating it against standard solution of M / 20 Mohr's salt.

Requirement : i) Apparatus : 100 mL volumetric flask , watch glass, glass rod , balance. Burette, pipette, conical flask, beaker.

ii) Chemicals: Mohr's salt (Ferrous ammonium sulphate .) ,dil H_2SO_4 , distilled water, M / 20 (or 0.05 M) Mohr's salt solution , $KMnO_4$, dil . H_2SO_4.

Diagram : -

Observation table :

Solution in burette : $KMnO_4$.(Unknown)

Solution by pipette : 0.05 Mohr's salt solution.

Solution in the conical flask : 10 mL Mohr's salt solution + 1test tube dil . $H_2 SO_4$

End point : Colourless to faint pink.

Molar mass of $KMnO_4$ = 158 g mol⁻

Molarity of Mohr's salt solution = 0.05 M

Pilot reading : .... to .... mL

Experiment 20 (B)

(B) Titration of KMnO₄ against standard solution of Ferrous ammonium sulphate (or Mohr's salt) :

Aim : To determine the concentration in terms of molarity of $KMnO_4$ by titrating it against standard solution of M / 20 Mohr's salt.

(1) Preparation of a standard solution of Mohr's salt :

Molar mass of Mohr's salt ($FeSO_4 . (NH_4)_2 SO_4. 6H_2O$) = 392 g mol^{-1}

1000 ml M Mhor's salt solution requires 392 g salt

100 ml $\frac{1}{20}$ (or 0.05 M) Mohr's salt solution requires

$\frac{100 \times 0.05 \times 392}{1000}$ = 1.96 g Mohr's salt .

by weighing 1.96 gm of Mohr's salt 0.05M solution was prepared.

Requirement : i) Apparatus : 100 mL volumetric flask , watch glass , glass rod , balance. Burette, pipette, conical flask, beaker.

ii) Chemicals: Mohr's salt (Ferrous ammonium sulphate .) ,dil H_2SO_4 and, distilled water, M / 20 (or 0.05 M) Mohr's salt solution , $KMnO_4$, dil . H_2SO_4.

Procedure :

1) Fill the burette with KMnO₄ solution.
2) Pipette out 10 mL of 0.05 M Mohr's salt solution in the conical flask and add one test tube of dil . H_2SO_4 to it .
3) Add KMnO₄ solution from burette , 1 mL at a time to the solution in the conical flask till faint pink colour appears.

    Record the range of burette reading as a pilot reading.
4) Fill the burette with KMnO₄ up to zero level . Pipette out 10 mL 0.05 M Mohr's salt solution In the conical flask and repeat the titration .

    [Hint : Add 1 mL KMnO₄ at a time till 3 mL above the pilot reading and then for accuracy add drop wise till the solution acquires faint pink colour.]
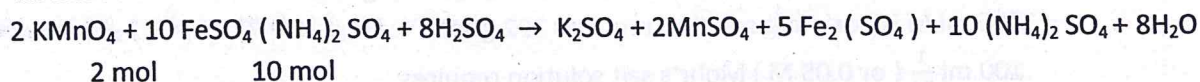
    This gives the volume of KMnO₄ required to titrate 10 mL Mohr's salt solution.
5) Repeat the procedure thrice and obtain the constant burette readings , (CBR)

Result : -

1) The strength of KMnO₄ solution = x = ... g L^{-1}
2) Molarity of KMnO₄ solution = y = ... M

Obs. No.	Volume of Mohr's salt Solution by pipette (ml)	Volume of KMnO₄ Solution by burette (ml)		Constant burette reading (CBR) (ml)
		Initial (ml)	Final	
1	10 ml	0.0 ml	.... ml	
2	10 ml	0.0 ml	.... ml	.... ml
3	10 ml	0.0 ml	.... ml	

Calculations:

$$2\ KMnO_4 + 10\ FeSO_4\ (NH_4)_2\ SO_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5\ Fe_2\ (SO_4) + 10\ (NH_4)_2\ SO_4 + 8H_2O$$

$\qquad$ 2 mol $\qquad\qquad$ 10 mol

10 ml 0.05 M Mohr's salt solution = $\dfrac{2 \times 158 \times 10 \times 0.05}{1000 \times 10}$ = 0.0158 g KMnO₄

Now , 1) **For strength :-**

10 mL 0.05 M Mohr's salt solution = (CBR) mL KMnO₄

(CBR) mL KMnO₄ solution = 0.0158 g KMnO₄

1000 mL KMnO₄ solution = $\dfrac{0.0158 \times 1000}{(CBR)}$ = ...

$\qquad$ x = ... g KMnO₄

Strength of KMnO₄ solution = x = ... g L^{-1}

2) **For molarity :**

Molarity of KMnO₄ solution = $\dfrac{\text{Strength of KMnO}_4\ \text{solution}}{\text{Molar mass}}$

$\qquad\qquad y = \dfrac{x}{158} = $ .... M of KMnO₄

Result : -

1) The strength of KMnO₄ solution = x = ... g L^{-1}

2) Molarity of KMnO₄ solution = y = ... M