RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029

Lab Manual

Chemistry (B.Sc.1st & 2nd Semester)

Department of Chemistry

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EXPERIMENT - 1

AIM:- Determine the number of molecules of water of crystallisation in ferrous ammonium sulphate $FeSO_4$ $(NH_4)_2SO_4.xH_2O$, 20 gm of which have been dissolved per litre Provided app. $\frac{N}{20}$ $KMnO_4$ solution.

APPARATUS REQUIRED:-

Burette, conical flask, dropper, glass rod

CHEMICAL REQUIRED:-

$FeSO_4$ solution, $KMnO_4$, mohr's salt

THEORY:-

Standardise $KMnO_4$ solution by titrating against $\frac{N}{20}$ $FeSO_4$ solution, then ferrous ammonium sulphate is titrated against the standard $KMnO_4$ solution the calculate the value of x

Chemical equation:

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$

$$[2FeSO_4.(NH_4)_2SO_4.6H_2O] + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + 13H_2O] \times 5$$

$$2KMnO_4 + 10FeSO_4.(NH_4)_2SO_4.6H_2O + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 10(NH_4)_2SO_4 + 68H_2O$$

First titration:- $FeSO_4$ against $KMnO_4$

Indicator:- $KMnO_4$ acts as a self indicator

End point:- Appearance of light pink colour

PROCEDURE:-

(i) Rins and fill the burette with $KMnO_4$ solution.
(ii) Pipette out 20 ml of $FeSO_4$ solution into a conical flask and then egg one test tube of dil. H_2SO_4.
(iii) Then add $KMnO_4$ solution dropwise with shaking.
(iv) At the end point light pink colour just appears.
(v) Repeat the titration to get a set of three concordant readings.

OBSERVATION:-

Volume of $\frac{N}{20}$ $FeSO_4$ taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $KMnO_4$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordante volume = V ml

2nd Titration :- Ferrous ammonium sulphate against $KMnO_4$

Indicator:- $KMnO_4$ acts as a self indicator

End point:- Appearance of light pink colour

PROCEDURE:-

(i) Rins and fill the burette with $KMnO_4$ solution.
(ii) Pipette out 20 ml of Ferrous ammonium sulphate solution into a conical flask and then egg one test tube of dil. H_2SO_4.
(iii) Then add $KMnO_4$ solution dropwise with shaking.
(iv) At the end point light pink colour just appears.
(v) Repeat the titration to get a set of three concordant readings.

OBSERVATION:-

S.No.	Initial reading	Final reading	Vol. Of $KMnO_4$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordant volume = X ml

Calculations:-

Ist titration:- Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

($FeSO_4$ solution) ($KMnO_4$ solution)

$$\frac{N}{20} \times 20 = N_2 \times V$$

N_2, i.e., normality of $KMnO_4$ solution $= \frac{N}{20} \times \frac{20}{V} = \frac{N}{V}$

2nd titration:- Again using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

(Ferrous ammonium sulphate solution) ($KMnO_4$ solution)

$$N_1 \times 20 = \frac{N}{V} \times X$$

∴ N_1, i.e., normality of ferrous ammonium sulphate $= \frac{N}{V} \times \frac{X}{20}$

∴ Strength of anhydrous ferrous ammonium sulphate = Normality × Eq.wt.

$= N_1 \times 284$

(∴ eq. wt. Of anhydrous salt is 284)

Now using the relation

$$\frac{Mol.wt.of\ FeSO_4\ (NH_4)2SO_4.xH_2O}{Mol.wt.of\ FeSO_4\ (NH_4)_2SO_4} = \frac{Strengt\ h\ of\ hydrous\ \ salt}{Strengt\ h\ of\ anhydrous\ \ salt}$$

$$\frac{284+18x}{284} = \frac{20}{N_1 \times 284}$$

From above relation, the value of x can be calculated.

PRECAUTIONS:-

(i) The apparatus should be cleaned and dried.

(ii) Always place the $KMnO_4$ solution in the burette and read the upper surface of its meniscus as the lower one is not clearly visible.

(iii) And about 20ml of dil. H_2SO_4 to a solution before titrating with $KMnO_4$ because of less amount is added then a brown ppt. Of hydrated MnO_2 is formed.

(iv) Never run large amount of $KMnO_4$ solution at a time otherwise a brown ppt. Of hydrated MnO_2 is formed

RESULT:-

Ferrous ammonium sulphate crystals have _ _ molecules of water of crystallisation.

EXPERIMENT – 2

AIM:- Given solution was prepared by dissolving 4g of ferrous oxalate in dil. H_2SO_4 and volume made to one litre. Determine volumetrically.

(iii) % purity of the sample.
(iv) % of oxalate ions in the sample.

Provided app. $\frac{N}{20}$ $KMnO_4$ solution.

APPARATUS REQUIRED:-
Burette, conical flask, dropper, glass rod

CHEMICAL REQUIRED:-
FeSO_4, ferrous oxalate, H_2SO_4, $KMnO_4$

THEORY:-
Standardises $KMnO_4$ solution by titrating it against $\frac{N}{20}$ $FeSO_4$ solution and then determined normality of ferrous oxalate by titrating it against $KMnO_4$ solution. Then % purity of the sample and % of oxalate ions can be calculated. Using normality equation

CHEMICAL EQUATIONS:-

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$2FeSO_4 + H_2SO_4 + O \rightarrow Fe(SO_4)_3 + H_2O$$

$$3FeC_2O_4 + 3H_2SO_4 + 3[O] \rightarrow Fe_2(SO_4)_3 + 4CO_2 + H_2O$$

Ist titration: $FeSO_4$ against $KMnO_4$

Indicator : $KMnO_4$ acts as a self – indicator

End point: Just appearance of permanent light pink colour.

PROCEDURE:-
(i) Rins and fill the burette with $KMnO_4$ solution.
(ii) Pipette out 20 ml of $FeSO_4$ solution into a conical flask and then egg one test tube of dil. H_2SO_4.
(iii) Then add $KMnO_4$ solution dropwise with shaking.
(iv) At the end point light pink colour just appears.
(v) Repeat the titration to get a set of three concordant readings.

OBSERVATION:-

Volume of $\frac{N}{20}$ $FeSO_4$ taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $KMnO_4$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordant volume = V ml

2nd titration: Ferrous oxalate against $KMnO_4$

Indicator : $KMnO_4$ acts as a self – indicator

End point: Just appearance of permanent light pink colour.

PROCEDURE:-

(i) Rins and fill the burette with $KMnO_4$ solution.
(ii) Pipette out 20 ml Ferrous oxalate solution into a conical flask and then egg one test tube dil. H_2SO_4
(iii) Heat the above solution on a wire gauze to 60-70°C.
(iv) Then add $KMnO_4$ solution dropwise with shaking.
(v) At the end point light pink colour just appears.
(vi) Repeat the titration to get a set of three concordant readings.

OBSERVATION:-

Volume of Ferrous oxalate taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $KMnO_4$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordant volume = X ml

CACLCULATIONS:-

1st titration:

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

($FeSO_4$ solution) ($KMnO_4$ solution)

$$\frac{N}{20} \times 20 = N_2 \times V$$

N_1,i.e., normality of $KMnO_4$ solution $= \frac{N}{20} \times \frac{20}{V} \times \frac{N}{V}$

2nd titration:

Again using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

(Ferrous oxalate solution) ($KMnO_4$ solution)

$$N_1 \times 20 = \frac{N}{V} \times X$$

N_1, i.e., normality of ferrous oxalate. $= \frac{N}{V} \times \frac{X}{20}$

∴ Strength of pure ferrous oxalate = Normality × Eq.wt.

$$= \frac{N}{V} \times \frac{X}{20} \times 60 = x \text{ g/litre (say)}$$

(∴ eq. Wt. Of ferrous oxalate = 60)

Weight of impure sample = 4g/litre

∴ % purity of ferrous oxalate sample = $\frac{X}{4} \times 100 = 25x$

Further amount of oxalate ions = Normality × eq. Wt.

$$= \frac{N}{V} \times \frac{X}{20} \times 44 = z \text{/litre (say)}$$

(∴ eq. Wt. Of oxalate ions, $C_2O_4^{2-} = \frac{88}{2} = 44$)

∴ % of oxalate ions in the smaple $= \frac{Y}{4} \times 100 = a$ (say)

RESULT:-

i. % purity of sample 25 y
ii. % of oxalate ions in the sample = a

EXPERIMENT – 3

AIM:- Determine the percentage purity of given sample of Mohr's salt, 20 gm of which are present per litre of solution. Provided solid $K_2Cr_2O_7$.

APPARATUS REQUIRED:-

Burette, beaker, conical flask, glass rod

CHEMICAL REQUIRED:-

Mohr's salt, $KgCr_2O_7$, N-phenyl-anthracitic acid

CHEMICAL EQUATIONS:-

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$FeSO_4\ (NH_4)_2SO_4.6H_2O \xrightarrow{aq.} FeSO_4 + (NH_4)_2SO_4 + 6H_2O$$

$$2FeSO_4 + H_2SO_4 + O \rightarrow Fe_2(SO_4)_3 + H_2O$$

Indicator: N-phenyl anthranilic acid.

End point: Green to violet red.

Titration of mohr's salt against $K_2Cr_2O_7$

PROCEDURE:-

i. Rins and fill the burette with $K_2Cr_2O_7$ solution.
ii. Pipette out 20ml of mohr's salt solution. Into titration flask and add about 100ml of $2NH_2SO_4$.
iii. Add 5-10 drops of N-phenyl anthranilic acid.
iv. Add $K_2Cr_2O_7$ solution dropwise till the colour changes from green to violet wet.
v. Repeat the titration to get a set of three cordant readings.

OBSERVATIONS:-

Weight of empty watch galss = wg.

Weight of watch glass +solid $K_2Cr_2O_7$ = (w+0.6125)g

∴ Weight of solid $K_2Cr_2O_7$ = 0.6125g

Volume of solution made = 250ml

∴ Normality of $K_2Cr_2O_7$ solution $= \frac{Strengt\ h}{Eq.wt.} = \frac{0.6125}{49} = \frac{1}{20}$

Volume of Mohr's salt solution taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $K_2Cr_2O_7$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordant volume = V ml

CALCULATIONS:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

(Mohr's salt) ($K_2Cr_2O_7$ solution)

$$N_1 \times 20 = \frac{N}{20} \times V$$

N_1, i.e., normality of Mohr's salt solution $= \frac{V}{400}$

∴ Strength of Mohr' salt solution = Normality × Eq. Wt.

$$= \frac{V}{400} \times 392 = x \text{ g/litre (say)}$$

∴ percentage purity of Mohr's salt $= \frac{x}{20} \times 100$

RESULT:-

% purity of Mohr's salt $= \frac{x}{20} \times 100$

PRECAUTIONS:-

i. Burette should be rinsed with $K_2Cr_2O_7$ solution before filling in the solution. Titration flask should be washed with distilled water after each titration.

ii. $K_2Cr_2O_7$ solution is always to be taken in the burette and its upper meniscus is to be considered while nothing the initial and final readings.

EXPERIMENT – 4

AIM:- To evaluate the value of x in $CuSO_4.xH_2O$, provided $\frac{N}{20}$ hypo solution.

APPARATUS REQUIRED:-

Burette, conical flask,. Beaker, glass rod

CHEMICAL REQUIRED:-

$No_2S_2O_3$, KI, starch solution

THEORY:-

Cupric ions can be determined iodometrically. The cupric salt solution is treated with an excess of pot. Iodide. The I_2 thus liberated is titrated with a standard solution of hypo using starch solution as indicator. The value of x can be calculated by suing the following relation.

Theoretical mol. Wt. Of $CuSO_4.xH_2O$ = Observed mol. Wt.

$$159.5 + 18x = \text{Observed mol. Wt.}$$

$$X = \frac{Observed\ \ mol.wt.-159.5}{18}$$

CHEMICAL EQUATIONS: -

$$2CuSO_4 + 4KI \rightarrow Cu_2I_2 + 2K_2SO_4 + I_2$$

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

Indicator: Freshly prepared starch solution.

End point: Blue to colourless with white ppt. (Hypo in burette)

PROCEDURE:-

A. Preparation of $CuSO_4.xH_2O$ solution

i. Weight accurately 3.35g of given $CuSO_4.xH_2O$ crystals.

ii. Dissolve them in 50ml of distilled water in a beaker.

iii. Transfer this into 250ml graduated flask and make the volume to the etched mark by adding more of distilled water.

iv. Stopper and shake the contents thoroughly.

B. Titration of $CuSO_4.xH_2O$ solution against hypo

i. Rinse and fill the burette with hypo solution.

ii. Pipette out 20ml of copper sulphate solution into the titration flask and add to it a few drops of dilute solution of sodium carbonate

until a slight turbidity is obtained. Add dil. Acetic acid dropwise until the turbidity disappears.

iii. Add about one gram of solid KI and dilute the solution with about 40ml of distilled water.

iv. Titrate the displaced I_2 with hypo solution run from the burette.

v. Add 2ml freshly prepared starch solution towards the end.

vi. At the end point, the blue colour sharply changes into a white ppt. Of Cu_2I_2.

vii. Repeat the titration to get a set of three concordant readings.

OBSERVATIONS:-

Weight of empty watch glass = w g

Weight of watch glass + copper sulphate = (w + 3.35)g

∴ weight of copper sulphate crystals = 3.350 g

Volume of solution made = 250ml

∴ Weight of hydrated copper sulphate/litre = $\frac{3.350}{250}$ × 1000 = 13.40 g

Volume of copper sulphate solution taken each time = 20ml

S.No.	Initial reading	Final reading	Vol. Of $K_2Cr_2O_7$ solution in ml
1.	.....	.....	.....
2.	.....	.....	.....
3.	.....	.....	.....
4.	.....	.....	.....

Concordant volume = V ml

CALCULATION:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

$(CuSO_4.xH_2O)$ (Hypo)

$$N_1 \times 20 = \frac{N}{20} \times V$$

N_1, i.e., normality of copper sulphate solution = $\frac{V}{400}$

$\therefore$ Eq. Wt. Of copper sulphate $= \frac{Strengt\ h}{Normality}$

$= \frac{13.40}{V} \times 400 = E$ (say)

As eq. Wt. Of $CuSO_4.xH_2O$ = Mol. Wt. Of $CuSO_4.xH_2O$

$\therefore$ Mol. Wt. Of $CuSO_4.xH_2O$ = E

Theoretical mol. Wt. = Observed mol. Wt.

Or

$159.5 + 18\ x = E$

$\therefore$ $x = \frac{E-159.5}{18}$

The value of x is to be changed into the nearest whole number since the water molecules can never be in fractions.

PRECAUTIONS:-

i. The indicator should be freshly prepared since on keeping, it is spoiled on account of bacterial attack.

ii. When I_2 is liberated in the titration flask, the indicator must be added towards the end when the liquid becomes lemon yellow. If the indicator is added in the beginning, it will result into a permanent deep blue colour.

RESULT:-

The value of x in $CuSO_4.xH_2O$ is five _ _ _ _

EXPERIMENT -5

AIM:- To Determine the percentage purity of the given sample of $MgSO_4.7H_2O$ and also determine the percentage of magnesium in it. Provided $\frac{N}{20}$ EDTA solution.

APPARATUS REQUIRED:-

Burette, watch glass, pipette, titrating flask, beaker

CHEMICAL REQUIRED:-

i. $\frac{N}{20}$ Solution of $MgSO_4.7H_2O$
ii. $\frac{N}{20}$ EDTA (Ethylene diamine tetra-acetic acid)
iii. Eriochrome black T indicator

THEORY:-

It is used to measure the % purity and % of Mg. for it take approximately $\frac{N}{20}$ solution of $MgSO_4.7H_2O$ is prepared by weighing 1.54 g and dissolving in 250 ml distilled water then it is titrated against $\frac{N}{20}$ solution of EDTA using Eriochrome black T indicator.

CHEMICAL REACTION:-

$$Mg^{2+} + Na_2 (H_2EDTA \rightarrow (Mg\ EDTA)_2 + 2Na^+ + 2H^+$$

PROCEDURE:-

1. **Preparation of approximately $\frac{N}{20}$ $MgSO_4.7H_2O$**
   - **i.** Firstly weigh out 1.54 g of $MgSO_4.7H2O$ accurately on a watch glass. Transfer it into 250 ml beaker and add about 50ml of distilled water.
   - **ii.** Dissolve the solid by stirring.
   - **iii.** Now transfer the solution into a 250 ml graduated flask.
   - **iv.** Wash out the beaker and make up the volume by dilution with distilled water to the etched mark.
2. **Titration of $MgSO_4.7H_2O$ solution against $\frac{N}{20}$ EDTA.**
   - **i.** Rinse and fill the burette with $\frac{N}{20}$ EDTA solution.
   - **ii.** Now pipette out 20 ml of prepared $MgSO_4.7H_2O$ solution into a titration flask.

iii. Add about 2 ml of buffer solution, 30 ml of distilled water and 2-3 drops of eriochrome black T indicator.

iv. Now run the EDTA solution from the burette into the flask dropwise with constant shaking.

v. Go on adding the EDTA solution till the colour changes from red to sky blue.

vi. Repeat the titration to get a set of 3 constant reading.

OBSERVATION:-

Weight of empty watch glass = w g

Weight of watch glass + $MgSO_4.7H_2O$ = (w+1.5375)g

∴ Weight of $MgSO_4.7H_2O$ = 1.5375 g

Volume of solution made = 250ml

Volume of $MgSO_4.7H_2O$ solution taken each time = 20ml

S.No.	Initial reading of burette	Final reading of burette	Vol. of EDTA solution used in ml.
1.	……..	……..	……..
2.	……..	……..	……..
3.	……..	……..	……..
4.	……..`	……..	……..

Concordant volume = Vml (say)

CALCULATIONS:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

($MgSO_4.7H_2O$ solution) (EDTA solution)

N_1, i.e., normality of $MgSO_4.7H_2O$ solution = $\frac{N_2V_2}{V_1}$

$$= \frac{N}{20} \times \frac{V}{20} \times \frac{V}{400}$$

∴ Strength of $MgSO_4.7H_2O = \frac{V}{400} \times 123 = x$ g/litre (say)

(where eq. wt. of $MgSO_4.7H_2O$ is 123)

Strength of sample (given) = 6.15 g/litre

∴ 6.15 gm of the sample contains pure $MgSO_4.7H_2O$ = x g

100 gm will contain $= \frac{a}{6.15} \times 100 = y$

∴ Percentage purity of $MgSO_4.7H_2O$ sample y

Now normality of Mg^{+2} ions = Normality of $MgSO_4.7H_2O$

∴ Strength of Mg = Normailty × Eq. wt.

$= \frac{V}{400} \times 24 = z$ g/litre

∴ 6.15 gm of $MgSO_4.7H_2O$ contain = z g of mg

100 gm of $MgSO_4.7H_2O$ will contain $= \frac{Z}{6.15} \times 100 = a$

∴ Percentage of Mg in the $MgSO_4.7H_2O$ sample = a

RESULT:-

% purity of $MgSO_4.7H_2O$ sample = y

% of Mg in sample = a

PRECATUTIONS:-

i. EDTA solution should be taken in the burette because in the titration flask, the proper pH of the solution has to be maintained.

ii. To avoid the high reactivity of the reagent with other ions, the pH of solution should he controlled by suing buffer solution.

iii. In order to avoid co-precipitation, adsorption etc. precipitation should not occur during the titration.

EXPERIMENT – 6

AIM:- To determine the strength of Zn^{2+} ion per litre in the given solution of $ZnSO_4.7H_2O$, provided $\frac{N}{20}$ EDTA solution.

APPARATUS REQUIRED:-

Burette, titration flask, dropper, etc.

CHEMICAL REQUIRED:-

i. EDTA
ii. Xylenol orange as indicator.

CHEMICAL EQUATIONS:-

$$Zn^{2+} + \text{Xylenol} \rightarrow [Zn - \text{xylenol}]^{2-} \text{ complex}$$

$$[Zn - \text{xylenol}]^{2-} \text{ complex} + \text{EDTA} \rightarrow [Zn - \text{EDTA}]^{2-} \text{ complex} + \text{Xylenol}$$

PROCEDURE:-

i. Rinse and fill the burette with EDTA solution.
ii. Now Pipette out 20ml of $ZnSO_4.7H_2O$ solution in the titration flask.
iii. Add about 30ml of distilled water, 3-4 drops of the indicator. Add hexamine powder till we get to red colour.
iv. Run EDTA solution from the burette into the flask till the colour of solution changes from red to lemon yellow.
v. Repeat the titration to get a set of three concordant readings.

S.No.	Initial reading of burette	Final reading of burette	Vol. of EDTA solution used in ml.
1.	……..	……..	……..
2.	……..	……..	……..
3.	……..	……..	……..
4.	……..`	……..	……..

Concordant volume = Vml (say)

CALCULATIONS:-

Using normality equation

$$N_1 \times V_1 = N_2 \times V_2$$

($ZnSO_4.7H_2O$) (EDTA solution.)

$$N_1 \times 20 = \frac{N}{20} \times V$$

N_1, i.e., normality of $ZnSO_4.7H_2O$ solution $= \frac{V}{400}$

Normality of $ZnSO_4.7H_2O$ = Normality of Zn^{2+} ions

∴ Strength of Zn^{2+} ions = Normality × Eq. wt.

$$= \frac{V}{400} \times 32.5$$

= x g/litre (say)

(Eq. wt. of Zn^{2+} = 32.5)

RESULT:-

Strength of Zn^{2+} ions/litre = a g/litre.

PRECAUTIONS:-

i. EDTA solution should be taken in the burette because in the titration flask, the proper pH of the solution has to be maintained.
ii. The complex formed must be highly stable otherwise we cannot get the sharp end point due to dissociation.
iii. In order to avoid co-precipitation, adsorption etc. precipitation should not occur during the titration.

EXPERIMENT – 7

AIM:- To separate or identify the constituents present in an inorganic mixture containing Pb^{2+}, CU^{2+} and Cd^{2+} by paper chromatography.

APPARATUS REQUIRED:-

Whatmann filter paper No. 1, chromatography jar, lead pencil, capillary tubes, scale.

CHEMICAL REQUIRED:-

i. N-butyl alcohol saturate with 3 N HCl.
ii. 1 % Na_2S solution.
iii. Inorganic mixture containing Pb^{2+}, Cu^{2+} and Cd^{2+} ions.

THEORY:-

Chromatography is the technique which is used for separation of constituents of a mixture. The technique of chromatography is based on adsorption of the components of a mixture on a suitable adsorbent. It is used for coloured and colourless both type of mixture.

PROCEDURE:-

i. First of all cut a strip of about 20 × 5 cm from whatmann filter paper no. 1.
ii. Now draw a horizontal line with pencil about 2 cm from the bottom.
iii. Apply a spot of mixture solution containing Pb^{2+}, Cu^{2+} and Cd^{2+} ions on the pencil line by means of a capillary tube. Dry the spot with the help of air dryer.
iv. Now lower the filter paper strip into chromatography jar in such a way that it does not touch the sides of the jar and hangs vertically straight.
v. Place the developing liquid i.e. mixture of n-butyl alcohol saturated with 3 N HCl in the jar and cover it. The level of the solution should be 2-3 cm only.
vi. Allow the developing liquid to rise till it has travelled a distance of about 15cm. then dry the strip with the help of air dryer.
vii. After that spray 1% Na_2S solution on the filter paper strip when three spots (two black and other yellow) will appear on the strip.
viii. Measure the distance travelled by spots as well as by the solvent.

OBSERVATION AND CALCULATIONS:-

When 1% Na_2S solution is sprayed then the colours produced on the various spots are:

Pb^{2+} black ……..rose pink

[In case dithiozone is used then the colours are rose pink, purple brown and purple]

Cu^{2+} black ……purple brown

Cd^{2+} yellow ……purple

Distance travelled by black spot (due to Pb^{2+}) = d_1 cm

Distance travelled by black spot (due to Cu^{2+}) = d_2 cm

Distance travelled by yellow spot (due to Cu^{2+}) = d_3 cm

Distance travelled by solvent (due to Cu^{2+}) = d_4 cm

R_f value of black spot (Pb^{2+}) = $\frac{d_1}{d_4}$

R_f value of black spot of Cu^{2+} ion = $\frac{d_2}{d_4}$

and R_f value of yellow spot of Cu^{2+} ion = $\frac{d_3}{d_4}$

RESULT:-

R_f value of Pb^2 = $\frac{d_1}{d_4}$ = 0.27

R_f value of Cu^{2+} ion = $\frac{d_2}{d_4}$ = 0.20

and R_f value of Cd^{2+} ion = $\frac{d_3}{d_4}$ = 077

PRECAUTIONE:-

i. Always keep the glass jar covered and undisturbed during the experiment.
ii. The paper strip should be erect and not curled.
iii. The spots of the solution must not dip in the developing solvent.
iv. Use a fine capillary tube for applying a spot of solution.

EXPERIMENT – 8

AIM:- Identify the inorganic anions CI^-, Br^- and I^- by paper chromatography.

APPARATUS REQUIRED:-

Whatmann filter paper no.1, capillary tube, chromatography jar, lead pencil, scale.

CHEMICAL REQUIRED:-

i. Inorganic mixture containing CI^-, Br^- and I^- ions.
ii. Developing solvent (10ml of n-butyl alcohol + 5ml pyridine + 10ml 1.5 M NH_4OH).
iii. Visualising reagent (ammonical $AgNO_3$ solution)

THEORY:-

Chromatography is the technique which is used for separation of constituents of a mixture. The technique of chromatography is based on adsorption of the components of a mixture on a suitable adsorbent. It is used for coloured and colourless both type of mixture.

PROCEDURE:-

i. First of all cut a strip of about 20 × 5 cm from whatmann filter paper no. 1.
ii. Now draw a horizontal line with pencil about 2 cm from the bottom.
iii. Apply a spot of mixture solution containing CI^-, Br^- and I^- ions on the pencil line by means of a capillary tube. Dry the spot with the help of air dryer.
iv. Now lower the filter paper strip into chromatography jar in such a way that it does not touch the sides of the jar and hangs vertically straight.
v. Place the developing liquid i.e. (10ml of n-butyl alcohol + 5ml pyridine + 10ml 1.5 M NH_4OH)in the jar and cover it. The level of the solution should be 2-3 cm only.
vi. Allow the developing liquid to rise till it has travelled a distance of about 15cm. then dry the strip with the help of air dryer.
vii. After that spray ammonical $AgNO_3$ solution on the filter paper strip when three spots (two black and other yellow) will appear on the strip.
viii. Measure the distance travelled by spots as well as by the solvent.

OBSERVATION AND CALCULATIONS:-

Distance travelled by CI^- spot = $d_1$cm

Distance travelled by Br^- spot = $d_2$cm

Distance travelled by I^- spot = $d_3$cm

Distance travelled by solvent front = d_4 cm

∴ R_f value of CI^- = $\frac{d_1}{d_4}$

R_f value of Br^- = $\frac{d_2}{d_4}$ = 0.36

R_f value of I^- = $\frac{d_3}{d_4}$ = 0.47

RESULT:-

R_f value of CI^- (chloride) ion = 0.24

R_f value of Br^- (bromide) ion = 0.36

R_f value of I^- (iodide) ion = 0.47

PRECAUTIONS:-

i. Always keep the glass jar covered and undisturbed during the experiment.
ii. The paper strip should be erect and not curled.
iii. The spots of the solution must not dip in the developing solvent.
iv. Use a fine capillary tube for applying a spot of solution.

EXPERIMENT – 9

AIM:- To determine the specific reaction rate of the hydrolysis of ethyl acetate (or methyl acetate) catalyzed by hydrogen ions at room temperature.

APPARATUS REQUIRED:-

Beaker, burette, pipette, conical flask, stop watch.

CHEMICAL REQUIRED:-

i. Ethylacetate
ii. $\frac{N}{20}$ NaOH
iii. $\frac{N}{2}$ HCl

THEORY:-

The hydrolysis of ethyl acetate in dilute aqueous solution gives acetic acid and ethyl alcohol. It is an example of pseudounimolecular reaction.

$$CH_3COOC_2H_5 + H_2O \xrightarrow{H^+} CH_3COOH + C_2H_5OH$$

The molecularity of this reaction is 2 because it requires simultaneous collision of 2 molecules (one of ester and another of water) but water is present in such a large excess that its concentration remains practically unchanged during the course of reaction. The result is that the rate of reaction depends only on the concentration of ethyl acetate and hence the order of the reaction is one.

Rate $= \frac{d[ester]}{dt} = k\ [Ester]$

The rate constant for a first order reaction is given by

$K = \frac{2.303}{t} \log \frac{a}{a-x}$

We study the progress of this reaction by determining the concentration of acetic acid in solution at different intervals of time by titration with a standard solution of alkali.

If V_0, V_t and $V_\propto$ are the volumes of alkali used after zero, t and infinite time. Then

$$K = \frac{2.303}{t} \log \frac{V_\propto - V_0}{(V_\propto - V_0) - (V_t - V_0)}$$

$$K = \frac{2.303}{t} \log \frac{V_\propto - V_0}{V_\propto - V_t}$$

REQUIREMENTS:-

Beakers, conical flask, burette, pipette, stop watch, ethylacetate, $\frac{N}{2}$ HCl, $\frac{N}{20}$ NaOH, ice, etc.

PROCEDURE:-

i. Take 100 ml of $\frac{N}{2}$ HCl in a clean beaker and about 10ml of given ethyl acetate (or methyl acetate in a clean test tube. When both have acquired the room temperature, pipette out 5ml of ethyl acetate and transfer it to the flask containing acid.

ii. After shaking well immediately withdraw 5ml of the reaction mixture into the conical flask containing some crushed ice to arrest the reaction, after it immediately start the stop watch. Titrate it against $\frac{N}{20}$ NaOH using phenolphthalein as indicator. The volume of NaOH used corresponds to V_0.

iii. After 10 minutes, again pipette out 5ml of the reaction mixture into a conical flask containing some ice and titrate it against NaOH. Similarly repeat the experiment after 20, 30, 40 minutes.

iv. Finally, heat the reaction mixture in a water bath at about 70°C for half an hour. Allow the flask to cool to room temperature.

v. Pipette out 5 ml from this reaction mixture and titrate against $\frac{N}{20}$ NaOH. The volume of NaOH used corresponds to V_∞.

OBSERVATIONS AND CALCULATION:-

S.No.	**Time in minutes T**	**Vol. of NaOH used in ml, V_t**	**$V_\infty - V_t$**	**$K = \frac{2.303}{t} \log \frac{V_\infty - V_0}{\infty - V_t}$**
1.	0	V_0 =	$V_\infty - V_0$	
2.	10	……..	$V_\infty - V_{10}$	$K = \frac{2.303}{10} \frac{V_\infty - V0}{V_\infty - V_{10}}$
3.	20	V_{10}	$V_\infty - V_{20}$	$K = \frac{2.303}{20} \frac{V_\infty - V0}{V_\infty - V_{20}}$
4.	30	=……	$V_\infty - V_{30}$	$K = \frac{2.303}{30} \frac{V_\infty - V0}{V_\infty - V_{30}}$
5.	40	V_{20} =	$V_\infty - V_{40}$	
6.	∞	…….	$V_\infty - V_{40}$	
		V_{30} =		
		…….		
		V_{40} =		
		……..		
		V_∞ =		

		……..		$\frac{V_\infty - V0}{V_\infty - V_{40}}$

RESULT:-

As the value of k, as calculated above, comes out to the almost same in each case, therefore, the reaction is of first order.

Alternatively, plot a graph of log $\frac{V_\infty - V_0}{V_\infty - V_t}$ against t. A straight line indicates that the reaction is of 1st order. The value of k can be calculated from the slope of the curve which is equal to $\frac{k}{2.303}$.

EXPERIMENT:- 10

AIM:- To prepare a colloidal solution of arsenious sulphide.

APPARATUS REQUIRED:-

Conical flask, test tubes, filter paper, Kipp's apparatus for H_2S.

CHEMICAL REQUIRED:-

i. Aresenious oxide (As_2O_3).
ii. Distilled water.

THEORY:-

Aresenious sulphide is a lyophobic sol. It can be obtained by passing H_2S gas through arsenic oxide solution.

$$As_2O_3 + 3H_2O \leftrightharpoons 2H_3\,As\,O_3$$

$$2H_3AsO_3 + 3H_2S \rightarrow As_2S_3 + 6H_2O$$

PROCEDURE:-

i. Take a 250ml conical flask and add 1gm arsenious oxide and 200 ml distilled water into the flask.
ii. Now heat the contents of the flask to boiling for about 15 minutes.
iii. Cool and filtered the solution.
iv. Now pass H_2S gas through the above filterate till it develops a yellow colour.
v. Continue to pass H_2S gas till it does not intensify the colour of the solution.
vi. After it bubble H_2 or CO_2 gas into the yellow solution to remove excess of H_2S gas.
vii. Alternatively the sol may be boiled to remove excess of H_2S.
viii. Allow the sol to achieve the room temperature this bright yellow solution is known as arsenious sulphide sol.

PRECAUTIONS:-

i. Use pure H_2S gas.
ii. The whole apparatus should be cleaned before use.
iii. Handle aresenious oxide carefully, since it is poisonous.

EXPERIMENT:-11

Aim: Determine the surface tension of a given liquid at room temp using stalagmometer by drop number method.

REQUIREMENTS:-

Stalgmometer, specific gravity bottle, a small rubber tube with a screw pinch cork, distilled water, experimental liquid.

THEORY:-

In the drop number method, the number of drops formed by equal volumes of two liquid is counted. If m_1 and m2 is the mass of one drop of each of the liquid having densities d1 and d2 respectively. If n1 and n2 is the number of drops formed by volume v of the two liquids, then their surface tensions are related as

$$\gamma 1/\gamma 2 = (d1/d2)*(n2/n1)$$

One of the liquid is water its surface tension and density are known.Then te surface tension of the given liquid can be calculated.

PROCEDURE:-

1. Clean the stalgmometer with chromic acid mix, wash with water and dry it.

2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the stalgmometer.

3. Immerse the lower end of the stalgmometer in distilled water and suck the water 1-2cm above mark A. adjust the pinch cork so that 10-15 drops fall per minute

4.Clamp the stalgmometer allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B

5. Repeat the exercise to take three to four readings

6. Rinse the stalgmometer with alcohol and dry it

7. Suck the given liquid in the stalgmometer and count the drops as in case of water

8. Take a clean dry weighing bottle weighs it with water as well as with liquid.

9. Note the temp of water taken in a beaker.

OBSERVATIONS: -

Room temp =t 0°C

Density of water=dw

Surface tension of water=Ɣ dynes/cm

S. no.	**No. of drops with water nw**	**No. of drops with liquid nl**
1.		
2.		
3.		
4.		
mean	Nw =	Nl =

Weight of empty specific gravity bottle=w1gram

Weight of specific gravity bottle+water=w2 gram

Weight of empty sp.gravity bottle+liquid=w3gram

Weight of water= (w2-w1)gram

Weight of liquid= (w3-w1)gram

CALCULATIONS:-

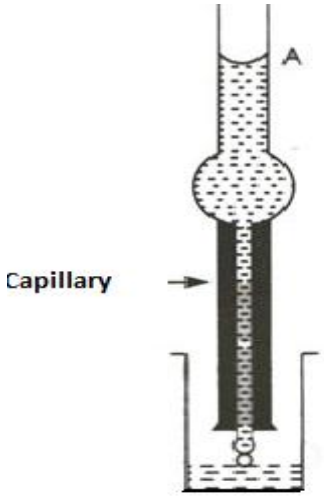
Density of the liquid

Dl= (w3-w1)/(w2 –w1) *dw

Surface tension of liquid= Ɣl/Ɣw = (dl/dw)*(nw/nl)* ¥w

Result

The surface tension of liquid is ……dynes/cm.



Stalagmometer

EXPERIMENT -12

AIM:- To determine the viscosity of the given liquid.

APPARATUS REQUIRED:-

Ostwald's viscometer, specific gravity bottle, Rubber tube.

CHEMICAL REQUIRED:-

Distilled water, liquid whose viscosity is to be determined.

THEORY:-

Viscosity of the given liquid is determined by using Ostwald's viscometer. A known volume of the given liquid is allowed to flow through the capillary of the viscometer and time of flow (t_1) from mark h_1 to h_2 is noted. The experiment is then respected with an exactly same volume of water and the time of flow (t_2(from mark h_1 to h_2 is noted. If η_1 and η_2 are the viscosities of the two liquid respectively, then

$$\frac{\eta_1}{\eta_2} = \frac{t_1 d_1}{t_2 d_2}$$

or

$$\eta_1 = \frac{t_1 d_1}{t_2 d_2} \times \eta_2$$

PROCEDURE:-

A. To determine the density of the given liquid.

i. Wash and clean the specific gravity bottle. Rinse it with ethyl alcohol and dry it with the help of drier.

ii. Weigh the specific gravity bottle accurately.

iii. Fill the specific gravity bottle with water and measure its weight. Then remove the water and dry it again. Now fill the specific gravity bottle with the given liquid and again measure its weight.

B. To determine the viscosity of the given liquid.

i. Wash and clean the viscometer Rinse it with ethyl alcohol and dry it with the help of drier.

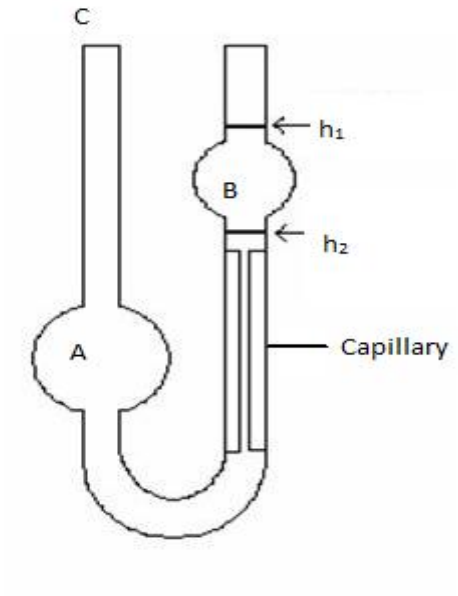
ii. Attach a piece of rubber tube to the end C and clamp the viscometer vertically on a stand.

iii. Add 15ml of distilled water through the arm of bulb a. The quantity of water should be such that when sucked from C, it stands above the bulb B and also some water remains in bulb A.

iv. Suck up water through the rubber tubing so that it rises above mark h_1. Press the rubber tubing with hand.

v. Now release the pressure on rubber tubing and allow the water to flow back. Start the stop watch as soon as the water reaches mark h_1 and stop it when the water just crosses the mark h_2. Repeat the experiment thrice.

vi. Remove water from the viscometer. Dry it and then Repeat steps (iii) to (V) with the given liquid. Repeat the experiment with given liquid thrice.



Specific gravity bottle

Ostwald's viscometer

OBSERVATION:-

Room temperature = t°C

Weight of empty specific gravity bottle = w_1 g

Weight of specific gravity bottle = w_2 g

Weight of specific gravity bottle + given liquid = w_3 g

∴ Weight of water = $(w_2 - w_1)$ g

Weight of liquid = $(w_3 - w_1)$ g

Density of water at t°C = d_w g/l

Viscosity of water at t°C = η_w poise

S.No.	Time taken by water to flow from h_1 to h_2 in second	Time taken by liquid to flow from h_1 to h_2 in second
1.	……..	……..
2.	……..	……..
3.	……..	……..
4.	……..`	……..
Mean time	t_w =	t_l =

CALCULATION:-

Density of liquid $= \frac{Wight\ of\ liquid}{weig\ ht\ of\ water}$

$d_l = \frac{(w_3 - w_1)}{(w_2 - w_1)}$

Viscosity of the liquid can be calculated by using the following relation:

$$\frac{\eta_l}{\eta_w} = \frac{t_l}{t_w} \times \frac{d_l}{d_w}$$

$\eta_l = \frac{d_l \times t_l}{t_w \times d_w} \times \eta_w$ Poise

RESULT:-

The viscosity of given liquid in η_l poise

PRECAUTION:-

i. The specific gravity bottle and viscometer should be thoroughly cleaned.
ii. Exactly same volume of the water as well as liquid should be used.
iii. The viscometer should not be disturbed during noting the time of flow of water as well as liquid.

EXPERIMENT – 13

AIM:- To determine the specific refractivity of a given liquid.

APPARATUS REQUIRED:-

Pyknometer or specific gravity bottle, abde's refractometer, source of light, dropper, weight box.

CHEMICAL REQUIRED:-

Given liquid whose specific refractivity is to be determined.

PROCEDURE:-

1. **Determination of density of given liquid**
   i. Clean the pyknometer with chromic acid (solid $K_2Cr_2O_7$ + conc. $H_2SO_{4)}$ carefully and then wash it thoroughly several times with water. Rinse it with ethyl alcohol and dry it with the help of a drier.
   ii. Suspend the pyknometer from the end of the balance beam by means of a copper hook and weigh it accurately.
   iii. Now attach a clean rubber tube to the end 'a' of pyknometer and immerse the end 'b' in distilled water and suck the distilled water through the rubber tube gently till water fills the bulb and stands to the mark on end a. In case water stands below the mark then suck more water and in case the water stands above the mark then remove excess water with the help of a filter paper strip. Ensure that no air bubble is present in pyknometer. Dry it from outside by wiping with a filter paper and weigh it.
   iv. Then remove the water and dry it again. Repeat the experiment with the given liquid.
2. **To determine the refractivity of the given liquid.**
   i. Open the prism box, clean the prism surfaces with ethyl alcohol (with soft cotton) and allow it to dry.
   ii. Introduce 3-4 drops of the given liquid with the help of a dropper between the prisms and press them tightly together.
   iii. Allow the light from the lamp to fall on the mirror. Set the mirror to reflect maximum light to the prism. Black spots in the field of view are due to insufficient liquid, then introduce 1 or 2drops of given liquid more.
   iv. Rotate the prism box by moving the lever until the boundary between the shaded and bright parts appears sharp.

v. If the light shade disc has a band of colours, make it sharp by rotating the knob of compensator.

vi. Adjust the prism box lever with screw so that light shade disc passes through the centre of the cross wires.

vii. Read the refractive index of the given liquid directly on the scale through the eye-piece. Take three readings from bright to dark field and another three readings from dark to bright field. Take average of these readings.

OBSERVATIONS:-

Weight of empty pyknometer = W_1 g

Weight of pyknometer + water = w_2 g

Weight of pyknometer + given liquid = w_3 g

$\therefore$ Weight of water = $(w_2 - w_1)$ g

Weight of liquid = $(w_3 - w_1)$ g

$\therefore$ Density of liquid = $\frac{weig\,ht\ of\ liquid}{weig\,ht\ of\ water} = \frac{w_3 - w_1}{w_2 - w_1}$ g

Assuming density of water as one we can calculate the specific refractivity of given liquid

$\therefore$ Specific refractivity, r = $\left(\frac{{n_r}^2 - 1}{{n_r}^2 + 2}\right)\frac{1}{d}$

Where n_r is refractive index and d is density of given liquid.

RESULT:-

Therefore specific refractivity of given liquid is 'r'.

Table: Refractive indeces of Some liquids

Table 1. Density of water at different temperatures

Liquid	Refractive index
Acetic acid	1.3718
Acetone	1.3616
Benzene	1.5044
CCl_4	1.4631
Chloroform	1.4486
Ethyl lcohol	1.3620
Ethyl acetate	1.3726
Methyl	1.3312
alcohol	1.4999
Toluene	1.3333
Water	

Temperature in °C	Density in g/ml
0	0.99987
1	0.99993
2	0.99997
3	0.99999
4	1.00000
5	0.99999
6	0.99997
7	0.99993
8	0.99988
9	0.99981
10	0.99973
11	0.99963
12	0.99952
13	0.99940
14	0.99927
15	0.99913
16	0.99897
17	0.99884
18	0.99862
19	0.99843
20	0.99823
21	0.99802
22	0.99780
23	0.99757
24	0.99733
25	0.99708
26	0.99681
27	0.99654
28	0.99626
29	0.99598
30	0.99568
31	0.99537
32	0.99506
33	0.99473
34	0.99440

35	0.99406
36	0.99372
37	0399336
38	0.99300
39	0.99262
40	0.99225
50	0.98807

EXPERIMENT:-14

AIM:- To determine the melting point of organic compound.

REQUIREMENTS:-

i. A 100 ml beaker or a Thiele's tube, a fine capillary tube, an iron stand with clamp, porous plate, spatula, burner etc.

ii. The organic compound whose melting point is to be determined and conc. H_2SO_4 or Paraffin wax.

PROCEDURE:-

i. First of all powder the crystalline substance.

ii. Take a capillary tube and seal it's one end by heating it.

iii. Fill the capillary tube with the substance whose m.pt has to be determined. To fill the tube, make a heap of the powdered substance on the porous plate. Push one end of the capillary tube into the heap. Some of the substance will enter the capillary tube.

iv. Now tap the sealed end of the capillary tube on the porous plate gently. Fill the capillary tube upto 2-3 mm.

v. Attach the capillary tube to a thermometer using a thread.

vi. Take liquid paraffin in a beaker or in a thiele's tube and place it over a piece of wire gauze placed over a tripod stand.

vii. Clamp the thermometer carrying the test tube to an iron stand and immerse them in the bath of liquid paraffin. The surface tension of the bath liquid is sufficient to hold the capillary tube in position.

viii. Heat the beaker slowly while constantly stirring the contents using a stirrer to maintain a uniform temperature throughout.

ix. When the temperature is within 15^o of the melting point of the pure substance, the flame is reduced. Then the temperature rises slowly.

x. Note the temperature (t_1) when the substance starts melting.

xi. Again note the temperature (t_2) when the substance has completely melted.

xii. The average of the two readings gives the correct melting point of the substance.

OBSERVATION:-

M.Pt. of the given compound in 1^{st} case = t_1°C

M.Pt. of the given compound in 2nd case = t_2°C

∴ M.Pt. of the unknown given compound is $\left(\frac{t_1 + t_2}{2}\right)°C$

PRACAUTIONS:-

i. Tap the capillary tube gently while filling the powdered compound, to avoid breaking of the tube.

ii. Don't take excess of the acid in the beaker.

iii. The capillary tube should not be attached to the thermometer using a rubber band as it gets spoiled.

iv. Heating of the acid should be gentle with constant stirring to maintain uniform temperature.

v. The bulb of the thermometer and the capillary should not touch the bottom of the acid bath.

EXPERIMENT-15

AIM:- To determine boiling point of organic compound.

APPARATUS REQUIRED:-

A thiele's tube or beaker, thermometer, a fine capillary tube, an ignition tube, an iron stand with clamp, burner etc.

CHEMICAL REQUIRED:-

The organic liquid and conc. H_2SO_4.

PROCEDURE:-

i. First of all fill two-thirds of the small test tube with the given liquid whose boiling point has to be determined.
ii. Fix this test tube to the thermometer with a rubber band in such a way that the bottom of the tube is at the middle of the thermometer bulb. The rubber band should be fixed near the mouth of the tube so that it remains outside the acid bath.
iii. Fill half of the beaker with Con. sulphuric acid and place it over a wire gauze placed over a tripod stand.
iv. Clamp the thermometer carrying the test tube to an iron stand through a cork. Lower the thermometer along with the tube into the acid bath.
v. Adjust the thermometer so its bulb is well under the acid and the open end of the tube with the rubber band is sufficiently outside the acid bath.
vi. Take the capillary tube and seal at it about 1 cm from one end by heating it in flame and giving it a slight twist.
vii. Place the capillary tube in the test tube containing the given liquid so that the sealed part of it stands in the liquid.
viii. Start heating the acid bath slowly and stir the bath gently. Keep an eye on the liquid and the test tube and also on the thread of the mercury in the thermometer.
ix. At first a bubble or two will be seen escaping at the end of the capillary tube dipped in the liquid, but soon a rapid and continuous stream of air bubbles escapes from it. At this stage the vapour pressure of the liquid just exceeds the atmospheric pressure.

x. Note the temperature (t_1) when continuous stream of bubbles starts coming out.

xi. Remove from the flame and note the temperature (t_2) when the evolution of bubbles from the end of the capillary tube just stops.

xii. The mean of these two temperatures gives the boiling point of the liquid.

xiii. Allow the temperature to fall by 10^oC and repeat the heating and again note the boiling point.

OBSERVATION:-

The temperature when a rapid and continuous stream of bubbles comes out = t_1°C.

The temperature at which the evolution of bubbles just stops = t_2°C

$$\text{Mean} = \frac{t_1^\circ + t_2^\circ}{2} = t°C.$$

PRECAUTIONS:-

1. If on placing the sealed capillary tube in the test tube, the liquid is seen rising in the capillary tube, it indicates that the capillary tube is not properly sealed. Reject this capillary tube and use a new one.
2. The seal point of the capillary tube should be well within the liquid.
3. The acid bath must be heated very slowly and the acid is stirred to ensure uniform heating.

RESULT:-

The boiling point of the given organic liquid = t°C

EXPERIMENT - 16

Aim : To Prepare pure sample of Iodoform

Iodoform (CHI_3) is the iodine analogue of chloroform. It is a pale yellow crystalline solid (m.p. 119°C), having a characteristic odour. It is used as a mild antiseptic and disinfectant. It is also used in the preparation of many medicinal ointments used as pain-relievers.

lodoform can be prepared by treating any organic compound containing $CH_3CH(OH)$ - group (e.g., ethanol, 2-propanol, 2-butanol) or $CH3CO^-$ group (e.g., propanone, 2-butanone) with iodine in presence of sodium hydroxide. In the laboratory, it is usually prepared from either ethanol or propanone. The chemical reactions involved are:

(a) With ethanol

$2NaOH + I_2 \rightarrow NaOI + NaI + H_2O$

$CH_3CH_2OH + NaOI \rightarrow CH_3CHO + NaI + H_2O$

$CH_3CHO + 3NaOI \rightarrow I_3CCHO + 3NaOH$

$I_3CCHO + NaOH \rightarrow CHI_3 + HCOONa$

(b) With Acetone

$CH_3COCH_3 + 3NaOI \rightarrow CH_3COCI_3 + 3NaOH$

$CH_3COCI_3 + NaOH \rightarrow CHI_3 + CH_3COONa$

PROCEDURE :-

(i) Dissolve 5 g of iodine in 5 ml acetone or ethanol in a 100 ml conical flask or round bottomed flask.

(ii) Add 5% NaOH solution in small portions with constant shaking the flask. Cool the flask from time to time under tap water so that temperature does not rise above 40°c. The addition of NaOH solution is further continued till the brown colour of iodine just disappears.

(iii) Allow the flask to stand at room temperature for 5-10 minutes.

(iv) Filter the iodoform, wash with little cold water and then dry on a filter paper.

(vi) Recrystallize the crude iodoform by addition of small amount of rectified spirit in a 100 ml conical flask and heat it on a water bath.

(vii) Add more rectified spirit slowly till the iodoform dissolves.

(viii) Filter the solution quickly through a fluted filter paper into a beaker.

(ix) Cool the solution in ice. The iodoform will crystallize rapidly.

(x) Filter the crystals on a Buchner funnel, dry the crystals.

RESULT:-

(i) Yield of crystals = ...g
(ii) Colour of crystals = Sparkling yellow
(iii) Melting point = 119°c

EXPERIMETN:- 17

AIM:- To prepare a sample of m-Dinitrobenzene from nitrobenzene.

APPARATUS REQUIRED:-

Round bottom flask, Air condenser, Water bath, glass rod, boiling chips.

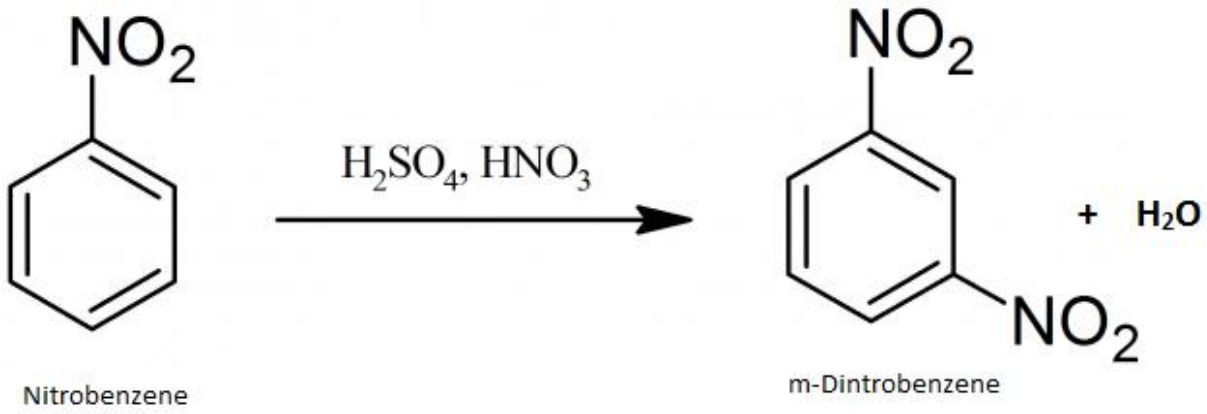
CHEMICAL REQUIRED:-

Nitrobenzene 5ml

Conc. HNO_3 6-7ml

Conc. H_2SO_4 10ml

CHEMICAL REACTION:-



PROCEDURE:-

i. Take 10ml of conc. H_2SO_4 and 6-7 ml of conc. HNO_3 in a 100ml round bottom flask.
ii. Add few boiling chips in the flask. Add nitrobenzene slowly with shaking and cooling the flask thoroughly.
iii. When whole of the nitrobenzene has been added shake the flaks vigorously fit it with air condenser and then place it on a boiling water bath.
iv. Clamp both the neck of the flask and condenser.
v. Remove and shake the flask time to time.
vi. After half an hour remove a drop of reaction mixture by means of glass rod and pour it into cold water. If it solidifies to a pale yellow solid, stop heating, remove the condenser and pour the reaction mixture from the

flask into about 200 ml of cold water with continuous stirring when a yellow solid is formed.

vii. Filter the precipitates through a fluted filter paper. Wash it with cold water and dry it completely.

viii. Purification: add the precipitates in 25 ml of rectified spirit and heat the flask on a boiling water bath until the solid has completely dissolved. Filter while hot through a filter paper and dry them on a porous plate.

PRECAUTIONS:-

i. Add nitrobenzene in small installments and shake the flask thoroughly after each addition.

ii. Cool the flask in water if it becomes hot after each addition.

RESULT:-

i. Yield of crystals = …… g

ii. Colour of the crystals = …….

iii. Melting point = 90°C

EXPERIMENT -18

AIM:- To prepare a pure sample of Dibenzal acetone.

APPARATUS REQUIRED:-

Conical flask, cork, beaker, glass rod.

CHEMICAL REQUIRED:-

Benzaldehyde = 7.5ml

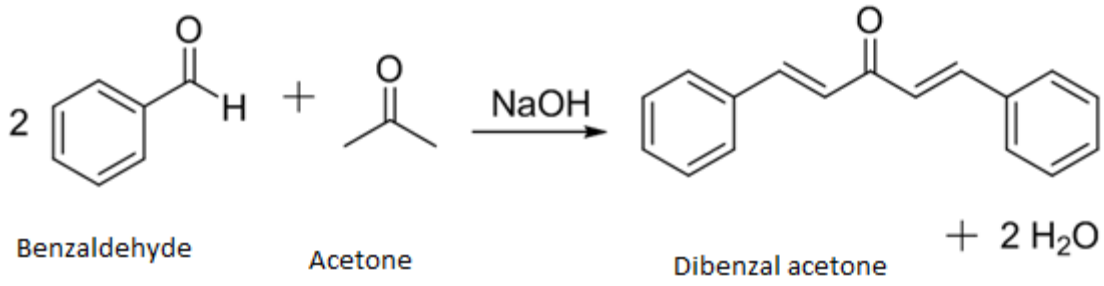
Acetone = 3.0ml

10% aq. NaOH = 15ml

Rectified spirit = 50 ml

CHEMICAL REACTION:-

Dibenzal acetone can be obtained by Claisen Schmidt reaction by condesnsing two moles of benzaldehyde with one mole of acetone in presence of alkali.



PROCEDURE:-

i. Take 7.5ml benzaldehyde, 3ml acetone and 25ml rectified spirit in a conical flask fitted with a cork. Shake it.

ii. Add 15 ml of 10% aq. NaOH into the conical flask drop by drop with continuous shaking of the solution.

iii. Cork the flask and shake it vigorously for about 10 min. releasing pressure from time to time keeping the loose fitting of cork.

iv. Allow it to stand for 30 min. at room temperature and then cool in ice bath for about 5min.

v. Filter the ppt. and wash with water to remove excess of alkali.

vi. Recrystallised the ppt. by dissolving these in minimum amount of hot rectified spirit and allow it to cool slowly filter the crystal and dry them between the folds of filter paper.

PRECAUTIONS:-

i. Wash the ppt. thoroughly with distilled water to remove excess of alkali.

ii. Use minimum amount of hot rectified spirit to dissolve the crude product for recrystallisation.

RESULT:-

Yield of crystals = …….g

Colour of crystals = Pale yellow

Melting point = 112°C

EXPERIMENT:- 19

AIM:- To prepare a sample of 2,4-Dinitrophenyl derivative of Acetophenone.

APPARATUS REQUIRED:-

Conical flask, beaker, glass rod etc.

CHEMICAL REQUIRED:-

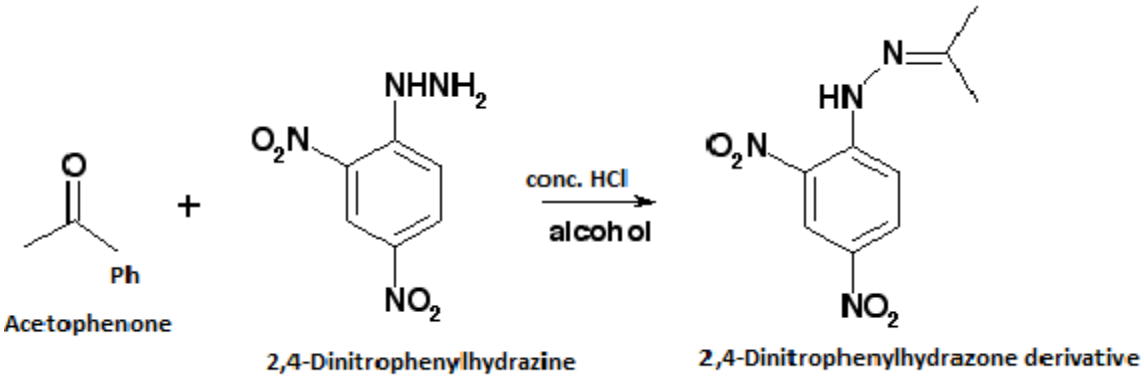
2, 4-DNP hydrazine - 1g

Acetophenon - 0.5g

Ethanol - 20ml

Conc. HCl - 2 ml

CHEMICAL REACTION:-



PROCEDURE:-

i. Add 1g 2, for –DNP in 20ml ethanol in a conical flask.
ii. Add 2ml conc. HCl and warm gently.
iii. Filter and add 0.5g acetophenone in the solution.
iv. Boil the solution and then cool it to room temperature.
v. Filter the crystals of 2, 4 DNP derivative and recrystallise them from ethanol.

RESULT:-

Colour of crystals - Orange

Yield - …..

M.Pt. - 237 -239°C

EXPERIMETN -20

AIM:- To Prepare a sample of p-Bromoacetanilide from acetanilide.

APPARATUS REQUIRED:-

Conical flask, beaker, glass rod, burette etc.

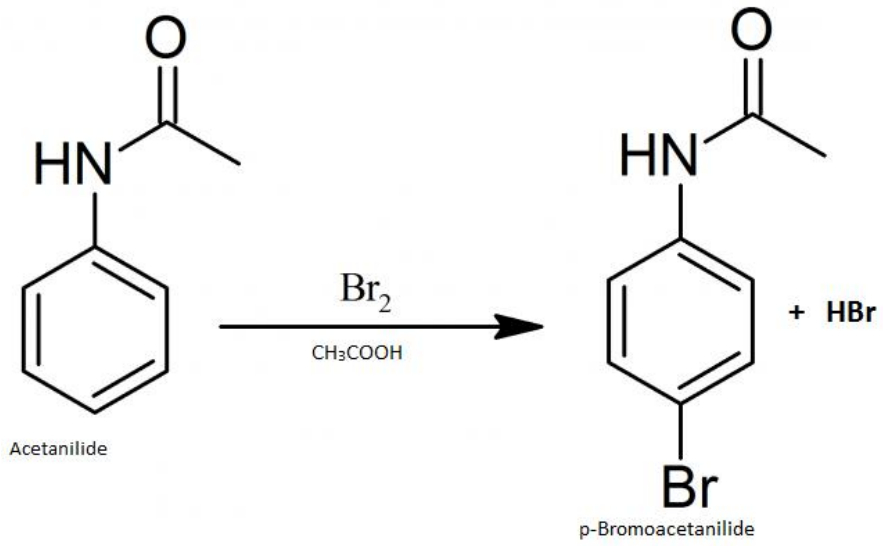
CHEMICAL REQUIRED:-

Acetanilide 4g

Glacial acetic acid 20ml

Bromine 1.8ml

CHEMICAL REACTION:-



PROCEDURE:-

i. Take 4 g of acetanilide into a 100 mL conical flask Add 10 mL of glacial acetic acid.Stirring with a glass rod may be necessary to dissolve the acetanilide.

ii. Now add 1.8 ml of bromine into 10 ml of acetic acid add bromine-acetic acid solution to acetanilide solution with stirring then leave the mixture 15 min.

iii. Transfer the mixture into beaker contain 100 ml of water with stirring. Collect the product by vacuum filtration using Büchner funnel.

iv. Purify the product by crystallization method using ethanol. Collect the white crystals by vacuum filtration, dried and weigh and calculate the percent yield.

PRECAUTIONS:-

i. Wear gloves and goggles during performing the experiment.

ii. Use extreme caution. Bromine burns can be quite severe.

RESULT:-

Yield- ……

Melting point - 167°C

EXPERIMENT:- 21

AIM:- To purify a given sample of phthalic acid by sublimation.

APPARATUS REQUIRED:-

China dish, funnel, tripod stand, wire gauze, cotton.

CHEMICAL REQUIRED:-

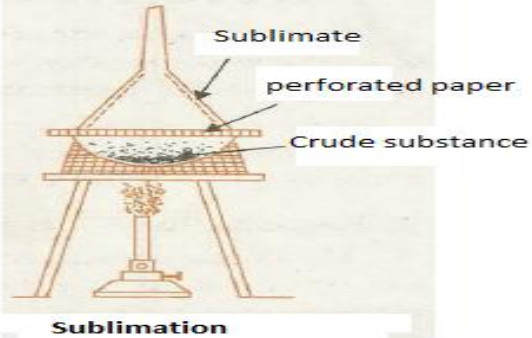
Impure phthalic acid - 5g

THEORY:-

Substances, which vapourise on heating, are purified by the method of sublimation. The substance, which has to be purified, is taken in a china dish, covered by a funnel. The china dish is heated on wire gauze. The substance volatilizes and the vapour condenses on the cooler portions of the funnel.

PROCEDURE:-

i. Take about 5g of impure phthalic acid in a dry and clean china dish and place it on a wire gauze kept on the tripod stand.
ii. Cover the china dish with a perforated paper and place and inverted funnel on it. Close the stem of the funnel with cotton.
iii. Heat the china dish on a low flame. Phthalic acid sublimes and condense on the cooler portions of the funnel.
iv. Remove the burner when whole of phthalic acid sublimes.
v. Cool and remove the funnel. Scratch pure phthalic acid from the inner walls of the funnel with a spatula on a watch glass.



Sublimation

EXPERIMENT:- 22

AIM:- To purify a given sample of camphor by sublimation.

APPARATUS REQUIRED:-

China dish, funnel, tripod stand, wire gauze, cotton.

CHEMICAL REQUIRED:-

Impure camphor - 5g

THEORY:-

Substances, which vapourises on heating, are purified by the method of sublimation. The substance, which has to be purified, is taken in a china dish, covered by a funnel. The china dish is heated on wire gauze. The substance volatilizes and the vapour condenses on the cooler portions of the funnel.

PROCEDURE:-

i. Take about 5g of impure camphor in a dry and clean china dish and place it on a wire gauze kept on the tripod stand.
ii. Cover the china dish with a perforated paper and place and inverted funnel on it. Close the stem of the funnel with cotton.
iii. Heat the china dish on a low flame. Impure camphor sublimes and condense on the cooler portions the funnel.
iv. Remove the burner when whole of camphor sublimes.
v. Cool and remove the funnel. Scratch pure camphor from the inner walls of the funnel with a spatula on a watch glass.