PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH
BILASPUR

LABORATORY MANUAL

Bachelor of Science
Chemistry
(B.Sc. Ist Year)

Department of Chemistry

PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH, BILASPUR

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LABORATORY MANUAL

Chemistry (BSc First Year)

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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

2^{nd} 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 $= \frac{N}{20} \times \frac{20}{V} = \frac{N}{V}$

2^{nd} 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

$$\therefore \quad \text{Normality of } K_2Cr_2O_{7\ \text{solution}} = \frac{Strengt\ h}{Eq.wt.} = \frac{0.6125}{49} = \frac{1}{20}$$

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.

$$\text{Theoretical mol. Wt. Of } CuSO_4.xH_2O = \text{Observed mol. Wt.}$$

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

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

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 = $\frac{3.350}{250} \times 1000 = 13.40$ 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 = $\frac{V}{400}$

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

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

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

∴ Mol. Wt. Of $CuSO_4.xH_2O$ = E

Theoretical mol. Wt. = Observed mol. Wt.

Or

$$159.5 + 18\,x = E$$

∴ $$x = \frac{E-159.5}{18}$$

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)

$$N_1\text{, i.e., normality of } MgSO_{4.}7H_2O \text{ solution} = \frac{N_2V_2}{V_1}$$

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

∴ 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:-

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

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

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

$$\begin{bmatrix}\text{In case dithiozone is used then the colours}\\ \text{are rose pink, purple brown and purple}\end{bmatrix}$$

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.

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

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 $V_\propto$.

OBSERVATIONS AND CALCULATION:-

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

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

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 $\frac{V_\propto - V_0}{V_\propto - V_t}$ 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:-

1. Aresenious oxide (As_2O_3).
2. Distilled water.

THEORY:-

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

$$As_2O_3 + 3H_2O \leftrightharpoons 2H_3AsO_3$$

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

PROCEDURE:-

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

PRECAUTIONS:-

1. Use pure H_2S gas.
2. The whole apparatus should be cleaned before use.
3. 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:-

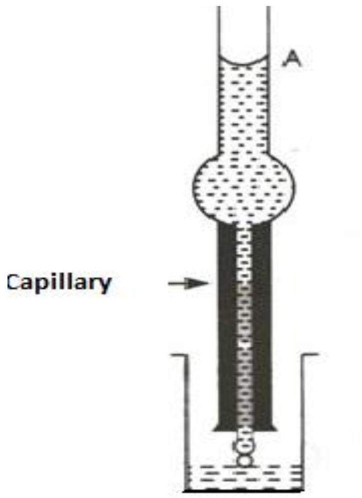
Density of the liquid

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.

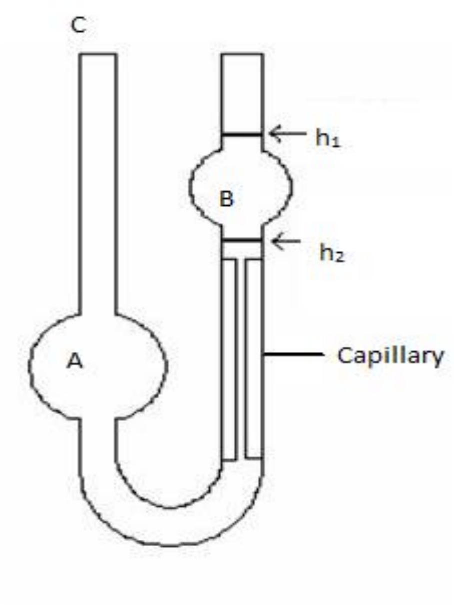
B. To determine the viscosity of the given liquid.

- 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 $= \frac{Wight\ of\ liquid}{weight\ of\ water}$

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

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

∴ Weight of water = $(w_2 - w_1)$ g

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

∴ Density of liquid = $\frac{\text{weight of liquid}}{\text{weight of water}} = \frac{w_3 - w_1}{w_2 - w_1}$ g

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

∴ 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 1st 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°C 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

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

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.

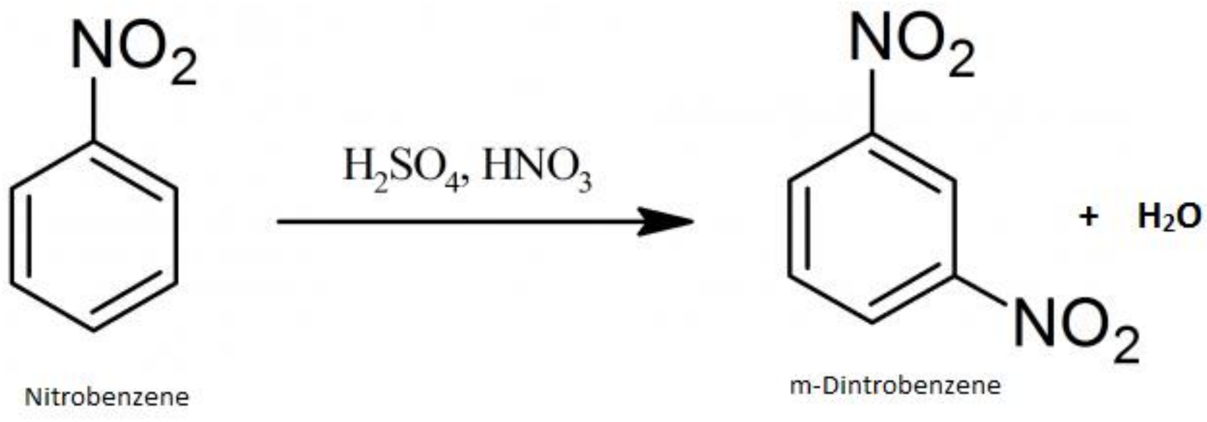
APPARATUS REQUIRED:-

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:-

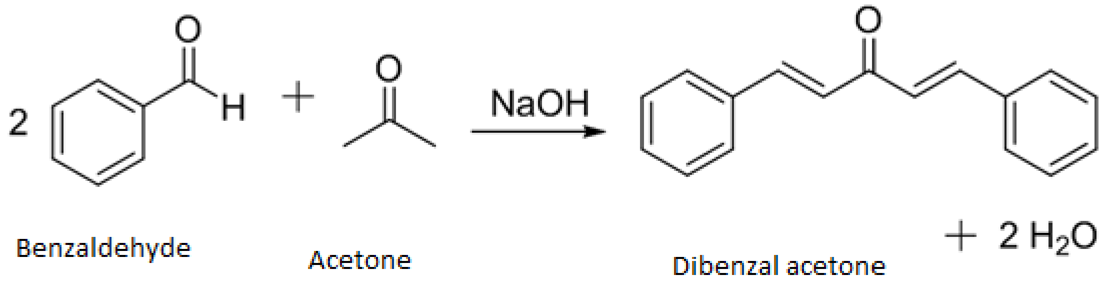
Benzaldehyde = 7.5ml

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:-

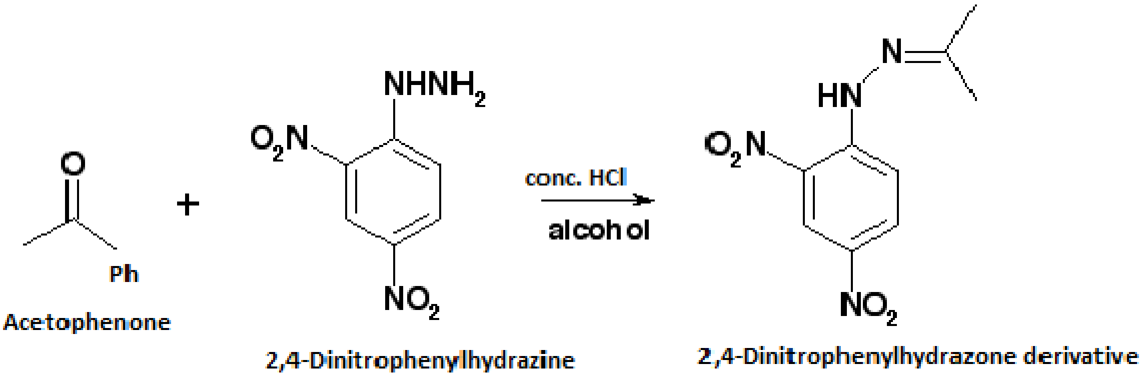
2, 4-DNP hydrazine - 1g

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

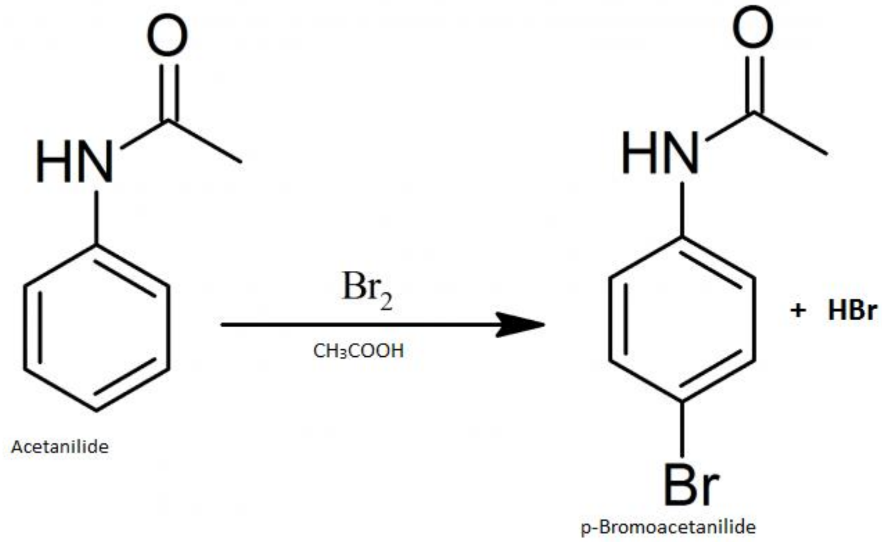
AIM:- To Prepare a sample of p-Bromoacetanilide from acetanilide.

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:-

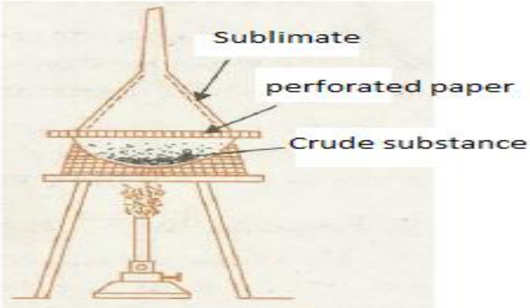
Impure phthalic acid - 5g

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.

VERIFIED

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PSSOU, CG Bilaspur

PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH
BILASPUR

LABORATORY MANUAL

Bachelor of Science
Chemistry
(B.Sc. IInd Year)

Department of Chemistry

PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH, BILASPUR

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LABORATORY MANUAL

Chemistry (BSc Second Year)

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EXPERIMENT 1

AIM : - Find out gravimetrically the percentage of Cu in given solution of $CuSo_4.5H_2O$, 20g of which has been dissolved per litre.

APPARALUS REQUIRED:-
Beaker, watch glass, pipette, glassrod.

CHEMICAL REQUIRED:-

1. Ammonium thiocyanate solution.
2. Saturated solution of NH_4HSO_3.
3. Solution of $CuSO_4.5H_2O$.

REACTIONS:-

$$2CuSO_4 + H_2O + NH_4HSO_3 \rightarrow Cu_2SO_4 + NH_4HSO_3 + H_2SO_4$$

$$Cu_2SO_4 + 2NH_4SCN \rightarrow 2CuSCN + (NH_4)_2SO_4$$

OBJECTIVE:-
In this experiment firstly, cupric salt is reduced to cuprous salt either by using sulphurous acid or ammonium bisulphate solution. Then cuprous ions are precipitated by ammonium thiocyanate (NH_4SCN). After then precipitates formed are filtered, washed dried and weighed using sintered glass crucible. From the weight of precipitates formed percentage of copper is determined.

PROCEDURE:-

1. Take 20 ml of given solution of copper sulphate in a clean 400 ml beaker.
2. Add few drops of HCl, followed by addition of 25 ml of 10% NH_4HSO_3 Solution.
3. Dilute the above solution by adding 150 ml of distilled water. Boil the solution.
4. Now put the beaker on an asbestos sheet, and then add drop wise 10% ammonium thiocyanate solution with constant stirring till the supernatant solution becomes colourless. (To check for complete precipitation add few drops more of ammonium thiocyanate).
5. Cover the beaker and allow the contents to stand for 2-3 hours preferably overnight.

6. Filter the precipitates using G-4 crucible, wash the precipitates using 1% cold dilute solution of NH_4HSO_3 till the filterate is free from SCN^- ions.
7. Finally wash the precipitates with 20% alcohol to remove NH_4SCN.
8. Heat the crucible in oven at 110^0 - 120^0C to constant weight.

OBSERVATIONS:-

Volume of given solution = 20ml

Strength of given Copper sulphate solution=20 g/litre

Weight of sintered glass crucible Before experiment i.e., empty=W_1g

Weight of sintered glass crucible

and $Cu_2(SCN)_2$ = W_2g

Weight of Cu_2 $(SCN)_2$ formed = $w_{2\text{-}W}1$ =Wg

CALCULATIONS:-

I. From 20 ml of given solution weight $Cu_2(SCN)_2$ formed = Wg

From 1 ml of given solution weight = $\frac{W}{20}$

From 1000 ml given solution weight = $\frac{W}{20}\times 1000 = 50$ Wgm

II. $Cu_2(SCN)_2$ = 2 cu

243 gm $Cu_2(SCN)_2$ is formed from copper =127 gm

1 gm $Cu_2(SCN)_2$ is formed from copper = $\frac{127}{243}$

50w gm of $Cu_2(SCN)_2$ is formed from copper =

$\frac{127}{243} \times 50\, w = a\ gm{-}litre\ (say)$

III. In 20.0n gms of copper sulphate, actual amount of Cu present = a gms

In 1 gms of copper sulphate,

Actual amount of Cu present = $\frac{a}{20}$

In 100 gms of copper sulphate,

Actual amount of Cu present + $\frac{a}{20} \times 100 + 5a$

RESULT:-

Percentage of Cu = 5a

PRECAUTIONS:-

1. Precipitate i.e. NH_4SCN should not added in excess to avoid solubility of $Cu_2(SCN_2$ as complex ion
2. Washing of the precipitates is to be done with dilute solution of NH_4HSO_3 to avoid oxidation of Cu(I) to Cu (II)

EXPERIMENT:-2

AIM :- Find out gravimetrically the percentage purity of $NiSO_4.\ 7H_2O$, 20.0g of which has been dissolved per litre.

CHEMICAL REQUIRED:-

1. One % dimethyl glyoxime solution in alcohol
2. Aqueous ammonia solution
3. Solution of nickel salt

APPARATUS REQUIRED:-

Pipette, glass rod, watch glass, beaker, sintered glass crucible

PROCEDURE:-

1. Take a 400 ml beaker add into it 20 ml of given nickel salt solution with the help of pipette.
2. Add 5 ml of HCl and dilute the mixture by adding nearly 200 ml distilled water. Heat the solution to 70 – 80℃
3. Place the beaker on an asbestos sheet and add slowly 30 ml of 1% alcoholic DMG solution with constant stirring.
4. After it adds immediately ammonia solution with constant stirring till it gives ammonical smell.
5. Now keep the beaker undisturbed for 45 minutes approx. Then digest the precipitates on a water bath for 30 minutes.
6. Cool the solution and check for complete precipitation by adding two drops of DMG.
7. Now filter the precipitates using G-3 crucible using a suction pump.
8. Wash the precipitates with small lots cold water several times.
9. Dry the crucible containing precipitates in an electric oven to constant weight.

OBSERVATION:-

Vol. of given solution taken for precipitation = 20 ml

Strength of given nickel sulphate solution = 20.0 gms/litre
Weight of sintered glass crucible before experiment i.e., empty = W_1 gms
Weight of sintered glass crucible and nickel dimethyl glyoxime = W_2 gms
Weight nickel dimethyl glyoxime formed = $W_2 - W_1 = W$ gms

CALCULATIONS:-

I. From 20 ml of given solution weight of nickel dimethyl glyoxime formed = W gms

From 1 ml of given solution weight of nickel dimethyl glyoxime formed = $\frac{W}{20}$

From 1000 ml of given solution weight of nickel dimethyl glyoxime formed = $\frac{W}{20} \times 1000 = 50\, W\, gms$

II. $Ni\,(C_4H_7O_2N_2)_2 \; = \; NiSO_4.\,7H_2O$

288.7 gms of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O$ = 280.7 gms

1 gm of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O = \frac{280.7}{288.7}$

50 w gms of nickel dimethyl glyoxime is obtained from $NiSO_4 \cdot 7H_2O$

$= \frac{280.7}{288.7} \times 50$ W = a g/litre (say)

III. In 20.0 gms of impure sample of $NiSO_4 .\, 7H_2O$, actual amount of $NiSO_4 \cdot 7H_2O$ present = a g

In 1 gm of impure sample of $NiSO_4.\, 7H_2O$ actual amount of $NiSO_4 \cdot 7H_2O$ present = $\frac{a}{20}$

In 100gms of impure sample of $NiSO_4 .\, 7H_2O$ present = $\frac{a}{20} \times 100 = 5a$

RESULT:-

Percentage of purity of $NiSO_4\, .7H_2O$

Sample = 5a%

PRECAUTIONS:-

1. Excess addition of precipitant is avoided, so that precipitant may not get separated along with the precipitates.
2. To avoid decomposition of precipitates the drying should not carried out above 120°C.

Experiment:3

Aim:-To verify Beer-Lambert law for KMnO4 and determine the concentration of the given KMnO4 solution.

Chemical Required:- solid KMn04.

Apparatus Required:- Spectrophotometer or Elico colorimeter, measuring flasks (100ml and 1000ml), weight box, fractional weights, graph papers.

OBJECTIVE:-

In it we used Beer Lamberts law, this law was dependent on absorbance phenomena.For it number of standard solutions of different concentrations are prepared. Their absorbance is determined. Then a plot of A vs c is drawn. It is a straight line passing through the origin. This proves the validity of Beer-Lambert law. Then the absorbance of the unknown solution is determined under the same experimental conditions. The concentration corresponding to this absorbance is read from the calibration graph.

PROCEDURE:-

(i) Prepare a stock solution of 10-^{3}M KMn04 by dissolving 0.0316g solid KMn04 in one liter distilled water.

.

(ii) Took four 100ml flat-bottomed measuring flasks and name them as A, B, C and D respectively. (iii)Now pipette out 20, 40, 60 and 80ml of stock solution of KMn04 into flask A, B, C and D respectively. Make the solution up to the given mark in Conical flask by dilution with distilled water in every 100ml flask.

20ml stock solution+	40ml stock sol.+	60ml stock sol+	80ml stock sol+
80ml distilled	60ml water	40ml water	20ml water

A **B** **C** **D**

(iv) Fill one optical cell with the stock solution and 2nd with distilled water. Insert them in colorimeter. Insert different filters one by one and find the filter that gives maximum absorbance. This filter is not to be changed throughout the experiment.

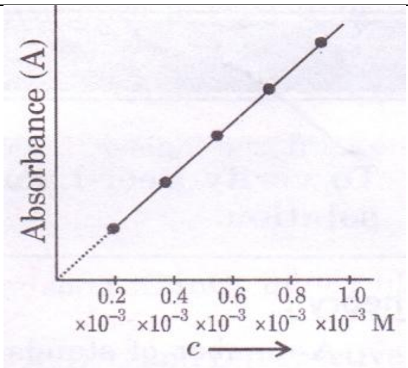
(v) Remove the stock solution and fill that optical cell with the solution (minimum 4ml) from flask A. Note the absorbance. Repeat the experiment with solution from flask B, C and D and note the absorbance in each case. Plot the calibration curve between *A* vs c.

(vi) Now fill the unknown solution and note the absorbance.

OBSERVATION AND CALCULATION:-

Solution	**Concentration**	**Absorbance**
Stock solution	10-^{3} M	....
Flask A	0.2 x 10-^{3} M	....
Flask B	10-^{3} M	....
Flask C	0.6 x 10-^{3} M	....
Flask D	10-^{3} M	....

From the calibration curve, read the concentration of the unknown solution corresponding to the absorbance. Further a straight line verified the Beer-Lambert's law.



Calibration curve

RESULT:

The concentration of given KMnO4 solution is..................... ....

EXPERIMENT NO. 4

AIM :- Prepare a pure sample of cuprous schloride, Cu_2Cl_2

APPARATUS REQUIRED :- Tripod stand, burner, funnel, round bottom flask, measuring cyclinder, etc.

CHEMICAL REQUIRED:-

1. Copper turnings = 10 gm
2. Sodium Chloride (NaCl) = 5 gm
3. Sodium sulphite = 5 gm
4. Copper sulphate ($CuSO_4.5H_2O$) = 10gm
5. Conc. HCl = 30ml

CHEMICAL REACTION:-

$$CuSO_4 + 2NaCl \rightarrow CuCl_2 + Na_2SO_4$$

$$CuCl_2 + Cu \rightarrow Cu_2Cl_2\downarrow$$

$$Cu_2Cl_2 + HCl \rightleftharpoons H^+[Cu_2Cl_3]$$

OBJECTIVE:- Cuprous chloride (Cu_2Cl_2) can be obtained by action of cupric chloride with excess copper in acidic solutions in presence of NaCl. Mixture is heated till the solution becomes colorless. The Colourless solutions when diluted results in formation of white powder i.e Cuprous chloride.

PROCEDURE:-

1. Measure 10 gm powdered Copper Sulphate, 10 gm Copper turnings, 5 gm Sodium Chloride and 30 ml of conc. HCl in a 250 ml round bottom flask.
2. Now heat the above mixture with occasional shaking. Continue the heating till the blue colour of solution disappears and a straw colour is obtained. It is the end point of heating.
3. Now stop the heating and pour the contents into a beaker containing 300 ml waterd.
4. Filter the white colour ppt. obtained and wash with very dilute solution of sodium sulphite.
5. Finally wash the ppt. with alcohol and ether.
6. Dry the ppt. in oven or by use of filter paper.

RESULT:-

1. Yield = 4 -6 gm
2. Colour = White

PRECUATIONS:-

1. Cuprous Chloride prepared is kept in a properly corked dry test tube to avoid oxidation of copper due to which it changes to green colour.
2. HCl should be added carefully.

EXPERIMENT 5

AIM:- To prepare pure crystals of Prussian blue from Iron fillings.

APPARATUS REQUIRED:- Measuring cyclinder, beaker, dropper, water bath, wire gauze, etc.

CHEMICAL REQUIRED:-

1. Potassium ferrocyanide ($K_4[Fe(CN)_6]$ = 25 gm
2. Iron fillings = 5 gm
3. Conc. HCl = 25 ml
4. Conc. HNO_3 = 5 ml

OBJECTIVE:- Prussian complex can be prepared by adding potassium ferrocyanide to ferric salt. A dark blue coloured compound is obtained i.e. Prussian complex.

REACTIONS:-

$$2\,HNO_3\,(\text{Conc.}) \rightarrow H_2O + 2NO_2 + [O]$$

$$2\,Fe + 6\,HCl + 3\,[O] \rightarrow 2\,FeCl_3 + 3H_2O$$

$$K_4\,[Fe(CN)_6] + FeCl_3 \rightarrow K\,Fe[Fe\,(CN)_6] + 3\,KCl$$

PROCEDURE:-

1. Weigh out 5 gm of Iron fillings in a clean 250 ml beaker. Pour into it 20 ml conc. HCl and 5 ml conc. HNO_3.
2. Allow the above mixture to stand for sometime till the reaction starts.
3. Now heat the contents of the beaker till the fumes are removed.
4. Now add 25 gm of Potassium ferrocyanide with constant stirring till blue colour ppt. formed.
5. Filter the ppt. and wash with dil. HCl.
6. Dry the ppt. in oven or by using filter paper.

OBSERVATIONS:-

1. Colour – blue
2. Yield = 18 to 20 gm

PRECAUTIONS:-

1. Pottasium ferrocyanide in powder form should be added to hot solution with constant stirring.

Experiment:6

Aim:-To prepare pure crystals of tetra ammine copper (II) sulphate.

Objective:-________________________

Tetra ammine copper sulphate is a complex of Cu(II) with co-ordination no. 4. When $NH_4$0H i: gradually added to an aqueous solution ofCuS0_4, CU(OH)2 is first precipitated and the precipitate ther dissolves, yielding a solution of a bright blue colour which is due to formation of tetra ammine copper (II) ion, [Cu(NH_3)4]

$$CuSO_4 + 2NH_4OH \rightarrow Cu\ (OH)_2 + (NH_2)_2\ SO_4$$

$$\underset{\text{(ppt.)}}{Cu(OH)_2} \rightleftharpoons \underset{\text{(dissolved)}}{Cu(OH)_2} \rightleftharpoons Cu^{2+} + 2OH^-$$

$$Cu^{2+} + 4NH_3 \rightleftharpoons [Cu(NH_3)_4]^{2+}$$

The removal of practically the whole cupric ion from the solution shifts the equilibrium of equation (2) towards the right, until the cupric hydroxide has completely passed into solution. The deep blue solution containing tetraamminecopper (II) ion is known as **Schweitzer's reagent.**
It has an ammonical odour, and crystals ofthe complex are obtained by adding ethyl alcohol to the above solution.

CHEMICAL REQUIREMENTS:-

Copper sulphate	5gm
1 : 1 Ammonia	20ml
Ethyl Alcohol	20-25ml
Conc. H_2S0_4	1-2ml

APPARTUS REQUIRED:- Beaker,Burner,Measuring Cylinder.

PROCEDURE:-

(i) Take 5gm powdered copper sulphate in a 250ml clean beaker and dissolve it in a minimum amount of water. Add 1-2ml cone. H_2S0_4 to make the solution clear.

(ii) Now pour 1 : 1 $NH_4$0H very slowly into the beaker with constant stirring till a ppt. of Cu(OH)2 first formed is redissolved yielding a deep blue solution due to formation of [Cu(NH_3)4j2+ and smell of ammonia is present due to slight excess of $NH_4$0H.

(iii) Now to the blue solution, add 20-25ml of ethanol dropwise with constant stirring. Add about 2ml of $NH_4$0H and heat the beaker in a water bath at 60-70°C for about 15-20 minutes.

(iv) Stop heating, cover the beaker with clock glass and allow it to stand undisturbed for 2-3 hours (preferably overnight)

(v) Long needle shaped blue crystals of complex are formed. Filter, wash with little alcohol and dry the crystals gently by pressing in between the folds of the filter paper or by placing in a desiccator. .

OBSERVATIONS:-

(i) Yield= about 5gm

(ii) Colour = Blue

(iii) Shape = Needle-shaped crystals

EXPERIMENT: 7

AIM:- To prepare pure crystals of chrome alum.

APPARATUS REQUIRED:- Measuring cyclinder, ice bath, round bottom flask etc.

CHEMICAL REQUIRED:-

1. Potassium dichromate ($K_2Cr_2O_7$) = 5 gm
2. Ethyl alcohol (C_2H_5OH) = 5 - 10 ml
3. Conc. H_2SO_4 = 5 ml
4. Water = 20 – 25 ml

REACTIONS :-

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

$$C_2H_5OH + [O] \rightarrow CH_3CHO + H_2O$$

$$K_2SO_4 + Cr_2(SO_4)_3 + 24H_2O \rightarrow K_2SO_4Cr_2\,(SO_4)_3\,.24H_2O$$

OBJECTIVE:-

When acidified $K_2Cr_2O_7$ solution is reduced by ethyl alcohol and the reduced solution is allowed to stand dark purple coloured crystals of chrome-alum separates out.

PROCEDURE:-

1. Dissolve 5 gm of $K_2Cr_2O_7$ in about 20 ml of water in round bottom flask and add 5 ml of conc. H_2SO_4 to it.
2. Shake well till whole of solid dissolves.
3. Cool the contents at room temperature in placing ice bath.
4. Now add dropwise ethyl alcohol with constant stirring. take care that the temperature does not exceed 50^0 C. If required add 2 – 3 pieces of ice.
5. Cool the solution in ice bath and allow the contents to stand for about 10 hours.
6. Filter and wash the crystals with cold water and dry it using filter paper. Dark purple colour crystals formed that are chrome alum crystals.

RESULT:-

1. Colour - dark purple
2. Yield = 10-12 gm

PRECAUTIONS:-

1. Add conc. H_2SO_4 carefully.
2. Take care that temperature should not exceed 50^0C.

EXPERIMENT: 8

AIM:- To prepare pure crystals of potassium trioxalatochromate (iii)

APPARATUS REQUIRED:- Stirrer, beaker, measuring cyclinder, ice bath, glass rod etc.

CHEMICAL REQUIRED:-

1. Oxalic acid = 12 – 15 gm
2. Ethyl alcohol = 10 – 15 ml
3. Potassium dichromate = 5 gm
4. Potassium oxalate monohydrate = 6 gm

OBJECTIVE:-

Potassium trioxalatochromate can be obtained by mixing potassium dichromate with oxalic acid and potassium oxalate. The mixture on cooling yields the crystal of the complex.

PROCEDURE:-

1. Add 5 gm of powdered $K_2Cr_2O_7$ in a minimum amount of boiling distilled water in 100 ml beaker.
2. Dissolve 15 gm of powdered oxalic acid in about 25 ml distilled water in a 250 ml beaker separately.
3. Cool the dichromate solution and pour its slowly in small amount to oxalic acid solution.
4. After the completion of reaction the solution is heated to half of the volume of the mixture.
5. Pour 6 gm of potassium oxalate slowly in small amount with constant stirring till the potassium oxalate completely dissolves.
6. Now add 10 – 15 ml ethyl alcohol to the above mixture and place it in ice. Bluish green crystals of the complex separates out.
7. Filter the ppt. and dry them using oven or by filter paper.

RESULT:-

1. Colour – Bluish green
2. Yield = 12 – 13 gm

PRECAUTIONS:-

1. Add oxalic acid carefully so that no violent reaction takes place.
2. Mixture should be place in ice bath so that temperature does not exceed.

EXPERIMENT:-9

AIM:- Determine enthalpy of dissolution of calcium chloride solid in water at room temperature.

APPARATUS REQUIRED:- Polythene bottle, thermometer, beaker, stirrer.

CHEMICAL REQUIRED:-
Powdered calcium chloride = 8 gm

OBJECTIVE:-
The objective of the experiment is to calculate the amount of enthalpy change when substance is dissolved in large amount of solvent.

$$\text{Enthalpy of solution} = \frac{q}{w} \times \text{mJ}$$

Q = heat change
w = weight of the substance taken
m = molecular weight

PROCEDURE:-

1. Determination of water equivalent
   a. Take 100 ml of water in a polythene bottle and note the temperature after 5 – 10 minutes.
   b. Take 250 ml beaker containing water heat it to a temperature higher then room temperature.
   c. Now measure 100 ml of hot water. Measure the exact temperature of hot water then quickly add it to the polythene bottle containing 100 ml of water at room temperature. Stir and note the final temperature.
2. Determination of enthalpy of solution
   a. Take 200 ml of water in a polythene bottle for which water equivalent has already been determined. Note the temperature after 5 – 10 minutes i.e. when it becomes constant.
   b. Now add 8 gm of powdered $CaCl_2$ to the polythene bottle containing 200 ml water. Stir and note the temperature.

OBSERVATIONS:-

(a) For water equivalent:-

Initial temperature of cold water = t_1℃

Temperature of hot water = t_2℃

Final temperature after mixing = t_3℃

Vol. of water taken polythene bottle = 100 ml = 100 g

Vol. of hot water added = 100 ml = 100 g

Assuming density of water = 1g/ml

Applying the principle of Heat lost = Heat gained

Heat lost by hot water = $100 \times (t_2 - t_3)$ cal.

Heat gained by cold water = $(100 + w)(t_3 - t_1)$ cal.

$$100 + w = \frac{100 \times (t_2 - t_3)}{(t_3 - t_1)}$$

$$W = \left[\frac{100 \times (t_2 - t_3)}{t_3 - t_1}\right] - 100 \text{ cal.}$$

(b)Temperature of water taken = t_1℃

Volume of water taken = 200 ml = 200 g

Final temperature after mixing = t_4℃

Heat absorbed by 8.0 g of solid

(Calcium chloride) = $(200 + 8 + w)(t_1 - t_4)$

Heat absorbed by rule – 1 of solid i.e., M

$$= \frac{(200+8+w)t_1 - t_4}{8} \times \text{M Cal}$$

M = Molecular weight = a cal (say)

RESULT:-

Enthalpy of solution = a cal/mol

= 4.184 × a J/mol

PRECAUTIONS:-

1. Fresh calcium chloride should be taken otherwise it will absorb moisture from atmosphere.

2. Temperature should be noted with the help of a thermometer graduated at 0.1℃

EXPERIMENT: 10

AIM:- Determine experimentally the partition coefficient of I_2 in CCI_4 and water.

APPARATUS REQUIRED:-

Four stoppered glass bottles, burette, pipette, conical flask.

CHEMICAL REQUIRED:-

1. 2% I_2 solution in CCI_4
2. N/20, N/100, $Na_2S_2O_3$ solution
3. starch solution
4. 10% KI solution
5. Distilled water

OBJECTIVE:-

When a mixture of two immiscible liquids a solute is added, it dissolves and distributes itself in a fixed ratio in both the liquids provided the form of solute does not change during distribution.

Suppose C_1 and C_2 are the concentration of solute (I_2) in two immiscible liquids i.e., CCI_4 and H_2O. Then

Where k is partition or distribution coefficient. The values of k is different for different pairs of liquids.

PROCEDURE:-

1. Take for clean and dry glass stoppered bottles. Label them as 1,2,3,and 4. Bottles must fit tightly.
2. Take 25 ml, 20 ml, 15 ml, and 10 ml saturated solution I_2 in CCl_4 in to bottle no. 1,2,3 and 4.
3. Now fill pure CCl_4 in another burette and take 0,5,10 and 15 ml CCl_4 into Bottle no. 1,2,3 and 4 resp. So that the total volume in each bottle is 25 ml.
4. Now add 150 ml distilled water in each bottle so that the total volume in each bottle is 175 ml.
5. Stopper each bottle and shake the contents of each bottle for atleast half an hour.

6. Now keep the bottles as such till the two layers separate clearly.
7. The lower CCl_4 layer will be intense violet and the upper aqueous layer will be yellowish.
8. Separate the two layers of each bottle with the help of a separating funnet in separate beakers.
9. Now Pipette out 20ml of aqueous layer from bottle no. 1 into the conical flask containing 10 ml 10% KI solutions and 1ml starch solution. Titrate the solution against N/100 sodium thiosulphate solution ($Na_2S_2O_3.5H_2O$) using starch indicator to know the volume of titrant used. Repeat the titration with the aqueous layer of bottle no. 2,3 and 4 and record the observations.
10. Now pipette out 5ml of CCl_4 layer from bottle no. 1 into the conical flask. Add 1g solid KI, containing 10 ml of 10% KI solution and add 20ml distilled water and shake. (Don't add starch in the beginning.) Titrate it against N/20 $Na_2S_2O_3.5H_2O$ solution. Add 1ml freshly prepared starch solution when the colour of the solution in the flask is light yellow. Continue adding hypo solution till the blue colour disappear. Repeat the experiment with bottle no. 2, 3 and 4.

Bottle No.	Volume of Aqueous layer	Burette reading		Vol. of Hypo used in ml
		Initial	Final	
1.	5ml 5ml 5ml			V_1 (say)
2.	5ml 5ml 5ml			V_2 (say)
3.	5ml 5ml 5ml			V_3 (say)
4.	5ml 5ml 5ml			V_4 (say)

Bottle No.	Volume of CCI_4 layer	Burette reading		Vol. of Hypoa Used in ml
		Initial	Final	

1.	5ml 5ml 5ml			V_5 (say)
2.	5ml 5ml 5ml			V_6 (say)
3.	5ml 5ml 5ml			V_7 (say)
4.	5ml 5ml 5ml			V_8 (say)

CALCULATIONS:-

For bottle no.1

(i) Fueor aqous layer

20ml of aqueous layer × N_1 = V_1 × N/100

N_1' i.e., normality of I_2 in water layer = $V_1/20 \times 1/100$

Strength or concentration of iodine = Normality × Eq. wt.

$=V_1/20 \times 1/100 \times 127$ =x g/ litre (say)

(ii) For CCI_4 layer

$$5 \times N_3 = V_5 \times N/20$$

N_3, i.e., normality of iodine in CCI_4 layer = $V_5 \times 1/20 \times 1/5$

Strength or concentration of I_2 in CCI_4 layer = $V_5 \times 100 \times 127$=y g/ litre (say)

Similarly, calculate the concentration of I_2 in aqueous layer as well as in CCI_4 layer for bottle no. 2, 3 and 4 respectively.

Bottle No.	Conc. In H_2o layer	Conc. In CCI_4 layer	K = $CCCI_{4/}CH_2O$

1.	X g/litre	Y g/litre	$K = \frac{Y}{X}$
2.	……...........	……...........	K = ……...........
3.	……...........	……...........	K = ……...........
4.	……...........	……...........	K = ……...........

RESULT:-

The value of distribution coefficient K will come out to be nearly same in every case or we can take the average value of K.

PRECAUTIONS:-

1. Only use glass stoppers as rubber stoppers can be attacked by organic solvents.
2. Use powdered form of iodine.
3. Much care should be taken to separate the 2 layers.
4. All the bottles should be kept at a constant temperature after shaking the contents of each bottle thoroughly.

OBSERVATION:-

Room temperature= t^0C

Table:-

CALCULATIONS:-

For Bottle No.-(i)

(i) Aquecous layer

Vol. taken for each titration = 5ml
Normality of I_2 in aq. Layer = N_1
Normality of $Na_2s_2O_3$ = N/100
Volume used (from table) = V_1

Applying normality equation

(I_2 in aq. Layer) $N_1V_1 = N_2V_2(Na_2S_2O_3)$

$N_1{}^x5 = 1/100\ {}^x\ v_1 = v_1/500$

Strenght of I_2 = Normality ^{x} Eq. wt.

$= v'_1/500\ {}^x\ 127$ g/l = a g/litre

(ii) CCl_4 layer

Volume taken for each titration = 5 ml

Normality of I_2 in CCl_4 layer = N_2

Normality of $Na_2S_2O_3$ = N/20

Volume used = v''_1

Applying normality equation

(I_2 in CCl_4 layer) $N_3V_3 = N_4V_4\ (Na_2S_2O_3)$

$N_3{}^x\ 5 = 1/20\ {}^x\ v''_1$

$N_3 = v''_1/100\ {}^x\ 127$ gl = b g/l

Similarly, for Bottle No. ii, iii and iv.

(i) Table:- (i): Titration of aqueous layer using N/100 $Na_2S_2\backslash O_3$

Bottle No.	Volume Taken (ml)	Burette reading		Vol.of $Na_2S_2O_3$ Used (ml)
		Initial	Final	
I	5.0 5.0 5.0			

EXPERIMENT NO. 11

AIM :-

To determine the CST of phenol – water system.

APPRATUS USED :-

A boiling tube, a stirrer, a thermometer graduated to 0.1 degree, 400ml beaker, iron stand, tripod stand.

CHEMICALS REQUIRED :-

Phenol and distilled water.

OBJECTIVE :-

It is based on the fact that when water and phenol are mixed together at room temperature they form heterogeneous mixture having white turbidity. Now when the system is heated a temperature comes where turbidity disappears on cooling turbidity appears again. Noting the temperature at which turbidity disappears and at which it appears the mean of the two temperatures give the temperature of mixing of phenol water system. Now the solutions of phenol od different compositions are prepared and the temperature of miscibility of the two is determined in each case. Then a graph of composition of mixture vs. miscibility temperature is plotted. The temperature corresponding to the maximum is the CST of the system.

PROCEDURE :-

(I)Take 60gm of phenol in a previously weighed boiling tube.

(ii)Add 2ml of distilled water with the help of a graduated pipette into the phenol taken In a boiling tube. Thus the % of phenol by weight is 75%.

(iii)Fill 2/3rd of the 400ml beaker with water and keep it on the wire gauge placed on the tripod stand.

(iv)Clamp the boiling tube into the beaker as shown. Fit the cork with two holes, one for the stirrer and other for thermometer.

(v)Heat the beaker slowly and stir the phenol water mixture. Note the temperature at which turbidity just disappears. Stop heating. Now allow thw mixture to cool and note the temperature when thw turbidity just appears.

(vi)Now again add 2ml of distilled water with the help of a graduated pipette. Thus the % of phenol by weight is 60%. Repeat step (v).

Repeat the process after adding 2ml of distilled water each time taking at least seven or eight readings.

OBSERVATIONS :-

Weight of empty boiling tube = $w_1$gm

Weight of tube + phenol = $(w_1 + 6)$gm

Weight of phenol = 6.0gm

Density of water = 1gm/ml (assuming)

RESULT :-

(i)CTS of phenol-water system = …………..^{o}C

(ii)Composition of the system

Phenol = …………..%

Water = ……………%

PRECAUTIONS :-

(i)Handle phenol very carefully as it causes severe skin burns.

(ii)Take care that in each case the level of phenol-water system in the tube must be atleast one cm below the level of water in the beaker.

(iii)For gradual and more uniform heating, surrounded the boiling tube with outer jacket (a more bigger tube).

(iv)Stirring inside the solution and outside in water must be done constantly.

(v)The bulb of the thermometer must remain dipping in phenol water system.

EXPERIMENT:- 12

AIM:- Determine solubility of benzoic acid at different temperatures and calculate ΔH of dissolution.

APPRATUS REQUIRED:-
Test tubes, Test tube holder, beaker, stand, burner, thermometer etc.

CHEMICAL REQUIRED:-

1. Benzoic acid
2. Water

OBJECTIVE:-
It was perform to check the solubility of benzoic acid. A graph is plotted between solubility an temperature and from graph solubility an any temperature can be determined by using Vant's Hoff's equation.

$$\text{Log } S_2 - \log S_1 = \text{Type equation here.}$$

PROCEDURE:-

1. Take 4 boiling test tubes and label them as 1,2,3, and 4.
2. Now weigh 0.1 gm, 0.15 gm, 0.20 gm and 0.25 gm of benzoic acid and transfer to the labeled tube.
3. Add 20 ml of water to each tube and place them in beaker containg water.
4. Now stir the contents of tube 1 and note the temperature at which benzoic acid dissolves completely.
5. Similarly note down the temperature of all tubes.

OBSERVATIONS:-

Test tube No	Temperature at Which benzoic acid dissolves	Amount Taken(g)	Solubility Mole/litre
I	t_1	0.10	$\frac{0.1}{122} \times 50$
II	t_2	0.15	$\frac{0.15}{122} \times 50$

III	t_3	0.20	$\frac{0.20}{122} \times 50$
IV	t_4	0.25	$\frac{0.25}{122} \times 50$

CALCULATION:-

Plot a graph between solubility and temperature from the graph determine the solubility of benzoic acid using vant’s Hoff’s equation.

PRECAUTIONS:-

1. Handle the acid carefully.
2. Temperature should not exceed.

EXPERIMENT:-13

AIM :-

To determine the enthalpy of neutralization of a weak acid (say acetic acid) versus strong base (say NaOH) and determine the enthalpy of ionization of the weak acid.

APPRATUS USED :-

Polyethene bottles thermometer, stirrer $, beakers\ etc$

CHEMICALS REQUIRED :-

$\frac{N}{2}$ acetic acid, $\frac{N}{2}\ NaOH$, distilled water.

OBJECTIVE :-

It is used to determine the value of enthalpy of neutralization which is defined as enthalpy change accompanying when one gram equivalent of the acid (or base) by base (or an acid) in a dilute solution for strong acids and for strong bases its value comes out to be nearly -58 kJ. It is nearly same for both strong acids and strong bases because in this case all acids and bases are almost completely ionised.

PROCEDURE :-

Determination of water equivalent of polyethene bottle.

(i)Take 100ml of distilled water in polyethene bottle fitted with a stirrer and a thermometer. The thermometer should be 1cm above the bottom of the bottle. When thermal equilibrium is achieved then note down the temperature say it is t_1^oC.

(ii)Heat 150ml of distilled water in a 250ml beaker at a temperature which is about 10-20^oC higher than the room temperature.

(iii)Take 100ml of hot water, note down its temperature (say it is t_2^oC) and immediately transfer it into the polyethene bottle already containing 100ml of cold water. Stir well and note down the temperature of water(after mixing hot and cold water) say it is t_3^oC.

(iv)Throw away water from the polyethene bottle and cool the room temperature.

(v)Take 100ml of 0.5 N acetic acid in a polyethene bottle fitted with a rubber cork with two holes. Insert a thermometer into one hole and a stirrer into the second hole.

(vi)Place 100ml of 0.5 N NaOH in another similar polyethene bottle.

(vii)The temperature of each solution is noted separately. Let these are $t_4{}^{o}C$ and $t_5{}^{o}C$ respectively.

(viii)transfer 100ml 0.5 N NaOH into the acid as quickly as possible. The mixture is well stirred.

(ix)The temperature is noted after every minute till a constant temperature is attained. Let it be $t_3{}^{o}C$.

OBSERVATIONS :-

(i) Determination of water equivalent

$$100\,(t_2 - t_3) = (100 + w)\,(t_3 - t_1)$$

$$\text{Water equivalent, } w = \left[\frac{100(t2 - t3)}{(t3 - t1)}\right] - 100$$

(ii)Determination of enthalpy of neutralization

Initial temperature of acid = $t_4{}^{O}C$

Initial temperature of base = $t_5{}^{o}C$

Final temperature after neutralization = $\left(t6 - \frac{t4+t5}{2}\right) - 100$

Water equivalent of polyethene bottle = w g

PRECAUTIONS :-

(I)Temperature should be noted using thermometer graduated to 0.1°C.

(II)The mixture of acid and base should be well stirred.

EXPERIMENT:-14

AIM :- Detection of organic compounds.

I. PRELIMINARY TESTS FOR ORGANIC COMPOUNDS

PHYSICAL CHARACTERSTICS

(a) Colour – from the colour of organic compounds following observation can be drawn

Experiment	Observation	Inference
(a) Note the colour of compound	I. Yellowish	Nitro compounds
	II. Pale yellow liquid	Nitrobenzene
	III. Deep orange yellow	Nitro aniline, nitrophenols.

(b) Odour:- From the odour of compound following observation can be drawn

Experiment	Observation	Inference
(a) Note the odour of the compound	(a) Fruity smell	Esters
	(b) Vinegar like smell	Acetic acid
	(c) Pungent smell	Benzoyl chloride
	(d) Wine like smell	Alcohol
	(e) Fishy smell	Amines

(c) **Ignition Test:- On burning organic compounds give following information**

Experiment	Observation	Inference
(a) Take a small amount of compound on a nickel spatula and heat.	a) Burns with a smoky flame.	Aromatic compounds
	b) Burns with non smoky flame.	Non aromatic compounds
	c) Burnt with sugar smell.	Carbohydrates
	d) Ammonical smell.	Urea
	e) Irritating smell with coughing.	Benzoic acid, succinic acid, salicyclic acid
	f) rotten eggs smell	Sulphur present

(d) Solubility Test:- The organic compound either dissolved in water or NaOH or dil. HCl gives following information

Experiment	**Observation**	**Inference**
a) Take a small amount of compound in a clean test tube and add 3 – 4 ml water. Shake well.	a) Sparingly soluble or insoluble b) Soluble	Hydrocarbons, esters, ethers etc. Lower alcohols, aldehydes, ketones, acids etc.
b) Test the compound with litmus.	c) Turns blue litmus to red d) Turns red to blue	Acids, phenols Lower amines
c) Test the solubility in dil. HCl	e) Soluble and reprecipitated on adding alkali	Base
d) Test the solubility in NaOH	f) Soluble but reprecipitated on adding acid	Phenol

PRELIMINARY CHEMICAL TESTS

1. **Sodalime test:-** On mixing organic compound with sodalime following observation are seen

Experiment	Observation	Inference
a) Take about 0.5 gm of solid compound with 2 gm of sodalime (NaOH + CaO) in a test tube and heat it	a) Ammonical smell b) Burnt sugar smell c) Smell of phenol d) Smell of bitter almonds	Urea, acetamide Carbohydrates Phenolic acid Benzaldehyde derivatives

2. **Ferric chloride test:-** with ferric chloride following observation are seen

Experiment	**Observation**	**Inference**
a) Dissolve 0.5 gm of compound in 2 – 3 ml of water and add 4- 5 drops of neutral $FeCl_3$	(a) Violet (b) Blue (c) Blue violet	Phenols, salicyclic acid P-cresol Resorcinol, m-

solution	(d) White ppt. changing to violet	cresol α-napthol

3. Conc. H_2SO_4 test:-

Experiment	**Observation**	**Inference**
a) Heat 0.5 gm of compound with 2 – 3 ml of conc. H_2SO_4	a) Dissolves with violent reaction.	Alcohols
	b) Soluble in cold water.	Ethers
	c) Dissolves without charring and CO is given out.	Formic acid or oxalic acid
	d) Charring with no gas is evolved.	Aldehydes, ketones and aromatic hydroxy acid Urea, oxalate
	e) Dissolves without charring and CO_2 is given out.	Aromatic
	f) Dissolves slowly but not precipitated on dilution.	Carbohydrates
	g) Dissolves on heating with charring CO_2	

4. **Sodium bicarbonate test:-**

Experiment	Observation	Inference
a) Mix the compound in water and add solid $NaHCO_3$	a) Evolution of CO_2 with effervescence.	Acid
	b) Dissolves with no evolution of CO_2 gas	Phenols

5. **Action of $KMnO_4$ solution:-**

Experiment	Observation	Inference
a) Add a very dil.	a) Decolorised	Unsaturated

Solution of $KMnO_4$ and dil. H_2SO_4 to the solution of compound	immediately b) Decolorised slowly	compounds Aldehydes

DETECTION OF EXTRA ELEMENTS

1. **For detection of extra elements we have to prepare Lassaigne's extract**
   a. **Preparation of lassaigne's extract:-**

      Cut small pieces of dry sodium metal into ignition test tube now heat it till the pieces changes to silver globule. Now add a pinch of organic compound into this test tube again heat it till the tube becomes red hot. Now take a china dish containing 10 ml water now break the hot tube into this china dish. Now the solution is heated and filtered. The filtrate is known as lassaign's extract. (L.E.) or sodium extract (S.E.)

Experiment	**Observation**	**Inference**
a) Test for nitrogen:- To about 2ml of the S.E., added a few drops of NaOH solution, followed by the addition of about 2 ml $FeSO_4$ solution. Boiled the solution and add dill. H_2SO_4 and shake.	No deep blue or blush green colour	Nitrogen absent
b) Test for Sulphur:- Add two drops of sodium nitroprusside solution to about 1ml of Lassaigne's extract.	No violet colour	Sulphur absent
c) Test for Halogens:-		
I. Belistein's Test:- Heated a copper wire in the non-lumionous flame, till it imparts no colour. Dipped the copper wire into the organic compound and heated again in the flame.	A green colour	Halogens present
II. $AgNO_3$ Test:- Added a fw drops of conc.	a) No white ppt.	Cl absent

HNO_3 to about 5ml of Lassaigne's extract. Boiled off all gases. The solution is cooled and treated with $AgNO_3$ solution.	b) No light yellow ppt. c) Yellow ppt. insoluble in NH_4OH.	Br absent I confirmed

IDENTEIFICATION OF FUNCTIONAL GROUP

1. Test for – COOH group:-

Experiment	Observation	Inference
a) Sodium bicarbonate test:- to the given compound add a saturated solution of sodium bicarbonate	a) Dissolve with brisk evolution of CO_2 gas.	-COOH group confirmed
b) Ester test:- To a small amount of organic compound add 1 – 2 ml alcohol and 2 -3 drops of conc. H_2SO_4	b) Fruity smell	-COOH group confirmed

2. Test for phenolic group:-

Experiment	Observation	Inference
a) Litmus test:- add few drops of blue litmus solution to aqueous compound	a) Blue litmus turned red	-COOH or Phenolic group present
b) Ceric ammonium nitrate test:- To aqueous solution of organic compound add few drops of ceric ammonium nitrate.	b) Brown ppt.	Phenolic group confirmed

3. Test for Ketonic group $> C = O$:-

Experiment	Observation	Inference
a) Sodium nitroprusside	a) Presence of red	Presence of

test:- add a pinch of organic compound in alkaline sodium nitroprusside solution. b) Schiff's reagent test:- add small amount of organic compound to 1 – 2 ml of schiffs reagent and shake.	colour b) Pink colour	$> C = O$ group Presence of $> C = O$ group Or Aldehyde present

4. **Test for Carbohydrates:-**

Experiment	**Observation**	**Inference**
a) Molisch test:- add few drops of alcoholic α-naphthol aolution to about 1 ml of aqueous solution of organic compound b) Conc. H_2SO_4 test:- add about 1 ml of con. H_2SO_4 to the given compound and warm.	a) Deep violet ring at the junction b) Charring	Carbohydrates present Carbohydrates present

5. **Test for esters:-**

Experiment	**Observation**	**Inference**
a) Hydrolysis Test:- To about 1 ml of substance, a drop of phenol phthalein and few drops of dil NaOH are added. Heat the contents on a water bath.	a) Pink colour disappears	Ester group present

6. Test for amide (-CONH$_2$) group:-

Experiment	**Observation**	**Inference**
a) HNO_2 test:- add a pinch of organic	a) Effervescence	-CONH$_2$ group present

compound to ice cold solution of $NaNO_2$ and dil. CH_3COOH b) NaOH test:- Heat a small amount of compound with NaOH solution	b) NH_3 (g) Evolved	-$CONH_2$ group present

7. Test for – NH_2 group:-

Experiment	Observation	Inference
a) HNO_2 test:- To ice cold solution of compound in dil. HCl. Add few drops of saturated $NaNO_2$ solution.	a) Brisk effervescence	Aliphatic -NH_2 group present
b) Dye test:- To about 0.5 g of compound add 2-3 ml of water containing 1 ml of conc. HCl. Then cool the contents in ice, add 2 ml of 10% $NaNO_2$ solution dropwise with constant stirring then add ice cold solution of alkaline β-naphthol	b) Orange-red dye	Aromatic D(-NH_2) group present

8. Test for anilides:-

Experiment	Observation	Inference
a) NaOH Test:- Heat a small amount of compound with 1-2 ml concentrated solution of NaOH	a) Peculiar smell	-NHCOR (anilide) group present

9. Test for –No_2 group:-

Experiment	Observation	Inference

a) Dye Test:- Heat the given compound with 1 ml of conc. HCl and few pieces of granulated tin and heat the mixture for about 5 minutes in a boiling water bath. Filters and cool the filterate in ice bath, then add 1, 2 – ml of $NaNO_2$ solution followed by 1, 2 – ml of ice cold alkaline β-naphthol solution.	a) Orange red	$-NO_2$ group

10. Test for halogen group:-

Experiment	Observation	Inference
a) Boil about 0.2 or 2-3 ml of compound with 2-3 ml of KOH or NaOh (alc.) for 5 minutes. Cool and add dil. HNO_3 and $AgNO_3$.	a) Ppts. Formed	Halogen group is present

11. Test for –OH (alcoholic) group:-

Experiment	Observation	Inference
a) Ester test:- add 1g of CH_3COONa to about 1-2 ml of compound and then 2-3 drops of conc. H_2SO_4 b) Ceric ammonium nitrate test:- To few drops (10-15), add 2 ml of ceric ammonium nitrate	a) Fruity smell b) Red or pink colour	Alcoholic group present Alcoholic group confirmed

solution		

12. Test for – CHO group:-

Experiment	Observation	Inference
a) Fehiling’s solution test:- add a small amount of organic compound to 2-3 ml of fehling’s solution. Heat the contents on a water bath.	a) Red ppts.	CHO group present
b) Tollen’s reagent test:- Warm 4-5 ml of Tollen’s reagent with small amount of organic compound on a water bath.	b) Silver mirror is formed	-CHO group present
c) Sodium bisulphite test:- To about 1 ml of organic compound, add 1-2 ml of saturated sodium bisulphite solution.	c) White ppts.	-CHO group $> C = O$ group present

PREPARATION OF SOLID DERIVATIVES

1. **DERIVATIVES OF PHENOLS:-**
    a. Picrates: - Mix equal amounts of saturated solution of compound and picric acid in benzene (say 3 ml each). Shake the contents vigorously. Filter the precipitates formed and recrystallise from benzene (if required).
2. **DERIVATIVES OF CARBOXYLIC ACIDS:-**
    a. S-Benzylisothiruronium salts :- Dissolve 0.5 g of given compound in 5-6 ml of water by heating. Add a drop of phenolphthalein indicator and then add NaOH till it becomes pink. Add 1-2 drops of HCl. Dissolve approximately 2.0 g of S-Benzyl isothiuronium chloride in 5-6 ml of water. Mix both the solution stir and cool the precipitates formed are filtered and crystallized from hot water.
3. **DERIVATIVES OF CARBOHYDRATES:-**
    a. Osazones :- Shake about 1 g of compound, 2 g of phenyl hydrazine hydrochloride and 3g of sodium in a boiling water bath.
    Yellow mass separates out, recrystallise from alcohol.
    Glucose, fructose and sucrose form osazone in approximately 2, 5 and 30 minutes respectively.
4. **DERIVATIVES OF ALDEHYDES AND KETONES:-**
    a. 2 ,4- Dinitrophenyl hydrazone: - Add approximately 5 ml of 2, 4-Dinitro hydrazine to 1g or 1ml of the compound in a dry test tube. Then add 1-2 drops of conc. H_2SO_4 Shake the contents and heat for few minutes. Cool, filter and recrystallise from alcohol or benzene.

VERIFIED

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PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH
BILASPUR

LABORATORY MANUAL

Bachelor of Science
Chemistry
(B.Sc. IIIrd Year)

Department of Chemistry

PANDIT SUNDARLAL SHARMA (OPEN) UNIVERSITY CHHATTISGARH, BILASPUR

LABORATORY MANUAL

Chemistry (BSc Third Year)

CHEMISTRY

B.Sc. IIIrd Year

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2. **Laboratory Techniques**
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     To separate a mixture of coloured organic compounds (fluorescein and methylene blue) by column chromatography.
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     To seprate a mixture of O- and p- nitrophenols by steam distillation.
3. **Synthesis of the following organic compounds:**
   - a) To prepare O-chlororobenzoic acid from anthranilic acid.
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Section – A (INORGANIC)

SCHEME OF MIXTURE ANALYSIS

The scheme of mixture analysis involves the following three main steps:-

1. Preliminary Tests.
2. Wet Tests for Anions or Acid radicals.
3. Wet Test for Cations or Basic radicals.

Wet Tests :- For wet tests, the solution of given mixture is required.

I. Water extract (WE) :- Dissolve small amount of the given mixture in distilled water. If the mixture is almost soluble then it is filtered and the filtrate is taken as water extract.

II. Sodium carbonate extract (SE) :- If the given mixture is insoluble in water then mix about 1g of Na_2CO_3 in the above solution, boild and filtered. The filtrate is taken as sodium carbonate.

1. **PRELMINARY TESTS:-**

Some of common preliminary test are :-

I. **Colour and smell :-** Note down the colour and smell of the given mixture.

(a) Colour

Dark green - Cr Salt

Light green - Ferrous salt

Green - Ni Salt

Blue green - Cu Salt

Dark brown - Ferric salt

Yellow - Ferric salt

Light pink - Mn salt

Pink violet - Co salt

White - Cu^{2+},Fe^{2+},Fe^{3+},Cr^{3+},Co^{2+},Ni^{2+},Mn^{2+} etc absent

(b) Smell

Vinegar smell - CH_3COO^-

Ammonical Pungent smell - NH^+_4 salt

Rotten egg smell - S^{2-}

II. Dry Heating Test :- Heat a small amount of mixture in a dry test tube to get following inference.

Observation	Inferences
Colourless, odourless gas which turns lime water, milky –Co_2	CO_3^{2-}
Colourless gas with rotten egg smell - H_2S gas	S^{2-}
Colourless gass which turns dichromate paper green –SO_2 gas	SO_3^{2-} and $S_2O_3^{2-}$
Colourless gas with Vinegar smell.	CH_3COO^-
Colourless gas with ammonical smell –NH_3	NH_4^+ salt
Brown gas which turns $FeSO_4$ solution black –NO_2	NO_2^- or NO_3^-
Reddish brown gas which turns starch paper yellow – Br_2	Br^-
Greenish yellow gas which bleaches moist litmus paper – Cl_2	Cl^-
Violet gass which turns starch paper blue – I_2	I^-
Yellow colour when hot and white colour when cold	Zn salt
Brown colour when hot and yellow colour when cold	Pb salt
Cracking noise	$Pb(NO_3)_2$

III. Charcoal Cavity Test :- Add a pinch of given mixture with twice its amount of anhydrous Na_2CO_3 and place in charcoal cavity add water heat in a reducing flame to get following in inferences.

Observation	Inferences
Red scales	Cu salt
Yellow residue on heating and white on cooling	Zn salt
Brown residue when hot and yellow when cold	Pb salt
White residue	Ba, Al, Ca, Mg salt
Black residue	No inference

IV. **Cobalt Nitrate Test:-** To the white residue is obtained in charcoal cavity then added a drop of cobalt nitrate solution and heat in an oxidizing flame to get the following inferences.

Observation	Inference
Green residue	Zn salt
Blue residue	Al salt
Pink residue	Ba salt

V. **Borax Bead Test:-** The test is applicable only for the coloured salt heat a crystal of borax ($Na_2B_4O_7.10H_2O$) on a clean Pt. –wire loop till a transparent glassy mass is obtained. Touch this glassy mass with coloured mixture and again heated in an oxidizing flame.

Observation	Inference
Pink bead	Mn salt
Yellow when hot and cold	Fe salt
Deep blue bead	Co salt
Reddish brown when cold	Ni salt
Green when hot, blue when cold	Cu salt
Dark green bead	Cr salt

VI. **Flame Test:-** Mix a pinch of mixture with conc. HCl and dipped the loop of Pt-wire in it and put the loop at the base of a non luminous flame of the burner and observe the colour of the flame to get the following inferences.

Observation	Inference
Brick red flame	Ca salt
Grassy green flame	Ba salt
Crimson red flame	Sr salt

VII. **Dilute H_2SO_4 Test:-** Mix few ml of H_2SO_4 to a pinch of given mixture and note the reaction.

Observation	Inference
Brown gas which turns $FeSO_4$ solution black – No_2 gas	No_2
With brisk effervescence colourless, odourless gas which turns lime water milky	CO_3^{2-}
Rotten egg smell gas with no colour	S^{2-}
Colourless gas which turns dichromate paper green	SO_3^{2-} or $S_2O_3^{2-}$
No action with dil. H_2SO_4	CO_3^{2-}, S^{2-} , SO_3^{2-} , S_2O^{2-} and NO_2^- are absent

VIII. **$KMnO_4$ Test:-** From the solution, boil off all the gases and mix 2 drops of $KMnO_4$ solution and note the observation.

Observation	Inference
Pink colour is discharged with a evolution of a gas	OX^{2-}, Cl^-, Br^- or I^-
Pink colour is discharged without evolution of any gas	NO_2^-
Pink colour is not discharged	NO_2^-,Cl^-,Br^-,I^- and OX^{2-} are absent

IX. **Conc. H_2SO_4 Test:-** With about 5ml conc. H_2SO_4, heat a pinch of given mixture and not the change.

Observation	Inference
Brown gas which becomes dense by mixing copper turning	NO_3^- present
Pungent smelling, colourless gas which gives dense white fumes with ammonia – HCl	Cl^- may be absent
Brown gas, which is not affected by mixing copper turning and turns starch paper yellow – Br_2 gas	Br^- present
Violet gas which turns starch paper blue – I_2 gas	I^- present
Vinegar smell gas –CH_3COOH	CH_3COO^- present
No reaction with conc. H_2SO_4	CO_3^{2-}, S^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, Cl^-, Br^-I^-, NO_3^-, CH_3COO^- are absent.

2. **Wet Tests for Anions or Acid radicals:-** These are the wet tests as the mixture is treated in the form of its water extract (WE) for sodium carbonate extract (SE) with the reagents.

Test for CO_3^{2-}

Experiment	Observation	Inference
I. Add about 5ml distilled water to a small amount of mixture, shake and filtered.	A. Residue B. Filtrate	For insoluble CO_3^{2-} in residue soluble CO_3^{2-} in filterate
II. To one part of filtrate mix few ml of dil. HCl.	Brisk effervescence with the evolution of colourless gas.	Soluble CO_3^{2-} present.
III. Pass the gas evolved through the lime water.	Turns milky	Soluble CO_3^{2-} Confirmed
IV. Mix few drops of $MgSO_4$ solution to the portion of filtrate.	White ppt. formed	Soluble CO_3^{2-} Confirmed.
V. For insoluble CO_3^{2-} To the residue add few drops of dilute HCl	Brisk effervescence with the evolution of colourless, odourless gas.	Insoluble CO_3^{2-} Confirmed

Test for Sulphide ion, (S^{2-}) :-

Experiment	Observation	Inference
1. To S.E. add to drops of sodium nitroprusside solution.	Purpul Colour	S^{2-} confirmed
2. To S.E. add 2-3 drops of acetic acid and lead acetate solution	Black ppt.	S^{2-} confirmed

Test for Sulphite ion (SO_3^{2-}) :-

Experiment	Observation	Inference
1. To the 2-3 drops of SE, add few drops of dil. H_2SO_4 and few drops of Potassium dichromate solution.	Green colour obtained	Sulphite ion confirmed
2. To SE, add 2-3 drops of $BaCl_2$ solution.	White ppt. which on treatment with dil. H_2SO_4 to give SO_2 gas	SO_3^{2-} confirmed

Test for Thiosulphate ion ($S_2O_3^{2-}$) :-

Experiment	Observation	Inference
1. To SE, add few drops of freshly prepared $FeCl_3$ solution.	Violet or purple colour which fades on standing	$S_2O_3^{2-}$ confirmed
2. Add few drops of $AgNO_3$ solution to SE.	White ppt. changing to yellow, orange, brown and finally black	$S_2O_3^{2-}$ confirmed

Test for nitrite ion, (NO_2^-) :-

Experiment	Observation	Inference
1. To the water extract add 2-3 drops of ferrous sulphate solution.	Black colour	NO_2^- confirmed
2. To water extract add 2-3 drops of diphenylamine.	Deep blue colour	NO_2^- confirmed
3. Add dil. H_2SO_4 to a pinch of mixture. Boil off gas evolved and mix 2 drops of $KMnO_4$ solution.	Pink colour is discharged	NO_2^- confirmed

Confirmatory tests or wet tests or acid radicals which do not react with dilute H_2SO_4 like Cl^-, Br^-, I^-, NO_3^-, CH_3COO^-, oxalate ion

Test for Nitrate ion (NO_3^-):-

Experiment	Observation	Inference
1. Add few drops of conc. H_2SO_4 to a pinch of mixture, boil and then add few copper turnings.	Dark brown fumes of NO_2 gas evolved	NO_3^- confirmed
2. Ring test:- To the WE add few drops of freshly prepared $FeSO_4$ solution. Shake and add few drops of conc. H_2SO_4 along the side of test tube.	At the junction a dark brown ring is formed of two layers	NO_3^- confirmed

Test for Chloride ion (Cl^-):-

Experiment	Observation	Inference
1. To WE add $AgNO_3$ solution.	White ppt. soluble in NH_4OH.	Cl^- present
2. Chromyl Chloride test:- Heated a pinch of mixture with solid	Red vapours of Chromyl Chloride are formed	Cl^- present
$K_2Cr_2O_7$ and few ml of conc. H_2SO_4 pass the red vapours through NaOH solution.	Yellow colouration	Cl^- present
To the yellow colour solution add dil. Acetic acid and lead acetate solution.	Yellow ppt. soluble in NaOH solution	Cl^- present

Test for Bromide ion (Br^-) :-

Experiment	Observation	Inference
1. CS_2 or CCl_4 Test:- To the WE add 4-5 drops of CS_2 or CCl_4 and few ml of	Orange coloure in CS_2 or CCl_4 layer	Br^-confirmed

freshly prepared chlorine water and shake thoroughly.		
2. Add few drops of $AgNO_3$ solution to the WE.	Light yellow ppt. partially soluble in NH_4OH	Br^- confirmed

Test for iodide (I^-) :-

Experiment	Observation	Inference
1. CS_2 or CCl_4 Test:- To the WE or SE after boiling off CO_2 by heating with dilute HNO_3, add few drops of CS_2 or CCl_4 and then add freshly prepared chlorine water with constant shaking.	Purple violet colour in CCl_4 layer	I^- Confirmed
2. To the WE or SE after boiling off CO_2, add $AgNO_3$ solution.	Yellow ppt. insoluble in NH_4OH	I^- Confirmed

Wet Test for Acetate (CH_3COO^-) :-

Experiment	Observation	Inference
1. Ester test:- Heat a pinch of mixture with small conc. H_2SO_4 and few drops of ethyl alcohol.	A fruity smell of ethyl acetate	CH_3COO^- confirmed
2. $FeCl_3$ Test:- To the WE add 2-3 drops of neutral $FeCl_3$ solution.	Blood red colour	CH_3COO^- confirmed

Wet Test for oxalate ion :-

Experiment	Observation	Inference
1. Heat a pinch of mixture	A mixture of CO and CO_2	Oxalate ion may be

with conc. H_2SO_4	evolved	present
2. To a part of SE, add dil. acetic acid. Boil of all gases and then cool. Add few ml of $CaCl_2$ solution.	White ppt.	Oxalate ion confirmed
3. Filter the solution and wash the ppt. with distilled water and extract the ppt. with about 1 ml of dil. H_2SO_4 added about two drops of $KMnO_4$ solution.	Pink colour of $KMnO_4$ discharge with evolution of CO_2	Oxalate ion confirmed

Wet Test for acidic radicals which do not react both with dil. H_2SO_4 like SO_4^{2-}, PO_4^{3-}, BO_3^{3-}

Test for sulphate ion SO_4^{2-} :-

Experiment	**Observation**	**Inference**
1. $BaCl_2$ Test:- To few ml of SE, add dil. HCl, boil off all gases and then cool. Then add 3-4 drops of $BaCl_2$ solution.	White ppt. of $BaSO_4$	SO_4^{2-} confirmed
2. Match – stick Test:- Filter the solution and wash the ppt. with distilled water mix the ppt. with twice the amount of Na_2CO_3. Apply a part of the mixture on wooden part of match stick. Heat the and in reducing flame till charred mass. Through this mass in sodium nitroprusside	Purple streaks	SO_4^{2-} confirmed

solution taken in china dish. 3. Lead Acetate Test:- Boil S.E. will dil. Acetic acid in a test tube and then add lead Acetate Solution.	White ppt.	SO_4^{2-} confirmed

Test for Borate ion (BO_3^{3-}):-

Experiment	Observation	Inference
1. In a few drops of ethyl alcohol add few drops of conc. H_2SO_4 to a pinch of mixture taken in china dish. Heat the mixture and ignite the vapours so evolved.	A green edged flame	BO_3^{3-} confirmed
2. Turmeric paper Test:- Dissolve few mg of the mixture in few drops of dil. HCl. Dip turmeric paper in the above solution and wrap it around the neck of semi – micro tube containing water. Boil the water to dry the turmeric paper.	Turmeric paper turns greenish brown	BO_3^{3-} confirmed

Test for Phosphate (PO_4^{3-}) :-

Experiment	Observation	Inference
1. Megnesia mixture:- To a part of SE add dil. HCl, boil of CO_2 gas and cool. Add NH_4OH solution till alkaline and then add	White ppt.	PO_4^{3-} confirmed

few drops of magnesia mixture (equal amounts of $MgSO_4$, NH_4Cl and NH_4OH solution). 2. Ammonium Molybdate Test:- Add few drops of conc. HNO_3 to a part of mixture, boil and then add a pinch of solid ammonium molybdate solution, boil again.	Yellow ppt.	PO_4^{3-} confirmed

3. Wet Test for Cations or Basic radicals:- The classification of cations in the six groups is based upon the fact that the radicals belonging to a particular group are precipitated by some specific group reagent. A group reagent is that which have following properties:-
    1) For the complete precipitation of the cations of particular group, it should be effective.
    2) The resulting precipitate must easily dissolve in acid to get the cation in the form of solution.
    3) For a specific group cation, it should be specific.

The scheme for separating cations or basic radicals into six analytical groups is shown as follows:-

	Group I	IIA	IIB	III	IV	V	VI
Cations	Ag++′ HG_2^{2+} Pb^{2+}	Hg^{2+},Pb^{2+} Bi^{3+}, Cu^{2+} Cd^{2+}	As^{3+} Sb^{3+} Sn^{2+}	Fe^{3+}, Al^{3+}, Cr^{3+}	Co^{2+},Ni^{2+} Mn^{2+}, Zn^{2+}	Ba^{2+},Sr^{2+} Ca^{2+}	Mg^{2+} Na^+,K^+ NH^+_4
Group Reagent	Dil.HCl	H_2S gas in presence of dil. HCl	H_2S gas in presence of dil. HCl	NH_4OH in presence of NH_4CL	H_2S gas in presence of dil. HCl	$(NH_4)_2Co_3$ in presence of NH_4Cl & NH_4OH	No group reagent

Original solution (OS) :- original solution is clear solution of the mixture which is prepared

(i) By using distilled water (DW) and dil. HCl

Or

(ii) **By using distilled water and conc. HCl**

Wet Test for group –I Cations :- To the OS, add to drops of dil. HCl. If precipitate appears, then add more HCl to make precipitation complete. Centrifuge and wash the ppt. with distilled water and reserve the filtrate for the analysis of Group II cations.

White ppt. - for Group I cation

Filtrate or supernate - for group II cation

With few ml of distilled water boil the white ppt. and filter.

-ppt. for Hg_2^{2+} and Ag^+

Filtrate for Pb^{2+} as $PbCl_2$

ppt. (for Hg_2^{2+} and Ag^+)	**Filterate (for Pb^{2+})**
With hot water wash the ppt., centrifuge and reject the filtrate. Add few drops of dil. Ammonia solution to the ppt. and centrifuge. Centrifuge :- I- Black residue (for Hg_2^{2+}) :- add stannous chloride to the solution of mercurous salt. White ppt. turns gray – Hg_2^{2+} confirmed II- Supernate or filtrate (for Ag^+) :- To filtrate add few drops of dil. HNO_3 -white ppt. – Ag^+ confirmed	Filtrate is classified into two parts 1) To one part add few drops potassium chromate solution (yellow ppt.) 2) To second part add few drops KI solution (yellow ppt.) -Pb^{2+} confirmed

Group II :- If Group I cation is present, then take the filterate of Group I and pass H_2S gas. To OS add dil. HCl and pass H_2S gas.

ppt. – for Group II cations

Filtrate - for Group III

Ppt - contain Pb^{2+},Hg^{2+},Bi^{3+}, Cu^{2+}, Cd^{2+}, As^{3+} Sb^{3+} , Sn^{2+} in the form of their sulphide.

Analysis of cation of IIA or II B in the acidic OS after passing H_2S gas, the ppt. obtained centrifuge and wash the ppt. with distilled water. For group III reserve the filtrate.

<table>
<tr><th>Coloured ppt. – for group II</th><th colspan="2">ppt. for group II</th></tr>
<tr><td rowspan="2">Mix few ml of yellow ammonium sulphide to the above ppt. shake and warm the content and centrifuge. In a beaker, decant off the liquid portion to the remaining ppt. add 2 ml of yellow ammonium sulphide, shake, warm and centrifuge.
Analysis of group II-B cation:-
To the above filtrate add acid dil. HCl to make the solution. Warm the solution and centrifuge to the ppt. mix 2 ml of distilled water and 5 ml conc. HCl and warm. Centrifuge and wash the ppt. with dil. HCl
Yellow ppt – for As^{3+}
Filterate – for Sb^{3+}, Sn^{4+}</td><td colspan="2">Ppt – for group IIA
Filterate - for group IIB
Analysis of group II A cations
With few ml of dil. HNO_3 heat the ppt. and centrifuge</td></tr>
<tr><td>Residue:-
-Black residue for Hg^{2+}
With the help of the water wash the residue boil the ppt. with conc. HCl and pinch of potassium chlorate. Boil of cases and then mix $SnCl_2$ solution. White ppt. turns grey
- Hg^{2+} confirmed</td><td>Filterate :-
-For Pb^{2+}, Bi^{3+}, Cu^{2+} and Cd^{2+}.
Mix few drops of conc. H_2SO_4 and transfer the contains to china dish. Evaporate till few drops remain, cool and add 2ml H_2O and centrifuge</td></tr>
</table>

Coloured ppt.- for Group II	**Filterate – Group III**
Test for As^{3+} :-	Ppt for Pb^{2+}

Wash the ppt. with hot water boil with few ml conc. HNO_3 and then mix few drops of ammonium molybdate. Yellow ppt. – As^{3+} confirmed Test for Sb^{3+} and Sn^{3+} The filterate is divided into two parts :- 1) Mix few mg of oxalic acid to one part and pass H_2S gas. -orange ppt. - Sb^{3+} confirmed 2) Warm the second part with a piece of Al metal. Centrifuge if any ppt. reject them. To filterate add 5ml $HgCl_2$. - White ppt. – Sn^{4+} confirmed	Filterate for Bi, Cu, Cd- Wash the ppt. with H_2O reject is washing mix few drops of conc. Ammonium acetate and heat with shaking ppt. dissolve mix few drops potassium chromate solution and few drops of acetic acid. Yellow ppt. – Pb^{2+} confirmed Tests for Bi, Cu, Cd:- Add conc. Ammonia drop wise (in excess) Centrifuge :- (i) Ppt for Bi^{3+} (ii) Filterate for Cu^{2+}, Cd^{2+} In the ppt., add few drops of sodium stannite solution It turns black – Bi^{3+} confirmed For $Cu^{2+}$and Cd^{2+} :- Divide the filterate in two parts. 1) Mix dil. HCl and few drops of potassium ferrocyanide solution to one part of the filterate Reddish colour – Cu^{2+} confirmed 2) Mix KCN solution to the second part of the filterate till blue colour disappears Pass H_2S gas – yellow ppt. - Cd^{2+} confirmed

Analysis of group III cations (Fe^{3+}, Cr^{3+},Al^{3+})

From filterate of group – II, boil off H_2S gas mix few drops of conc. HNO_3, boil and cool the content. Now mix solid NH_4Cl again, boil and cool. Then add NH_4OH solution in excess.

Centrifuge

- Ppt for group III
- Filterate for group IV

Mix 2 ml distilled water and few mg sodium peroxide to the ppt. Boil, cool and centrifuge.

Brown ppt. for Fe^{3+}	**Filterate for Cr^{3+} and Al^{3+}**
Dissolve the ppt. dil. HCl. Divide the solution in two parts:- 1) Mix KCNS solution to first part of the solution. Blood red colour - Fe^{3+} confirmed 2) Mix potassium Ferrocyanide solution to the second part of the solution. Deep blue colour ppt. - Fe^{3+} confirmed	Divide the filterate into two parts 1) Add few drops of lead acetate solution and dil. HCl to one part of the filterate - Yellow ppt - Cr^{3+} confirmed 2) Mix few mg NH_4Cl to the second part of the filterate. - White gelatinous ppt. Dissolve the ppt in dil. HCl and then add few drops of blue litmus solution and mix NH_4OS dropwise - Blue ppt. - Al^{3+} confirmed

Analysis of Group IV cations (Co^{2+},Ni^{2+}, Mn^{2+},Zn^{2+})

To the filterate of group III, mix NH_4OH solution in excess and pass H_2S gas.

Centrifuge

-ppt for group IV
- filterate for group V

Mix the ppt. with HCl, Shake and centrifuge

<table>
<tr><th rowspan="2">Black ppt. for Co^{2+} and Ni^{2+}</th><th colspan="2">Filterate for Mn^{2+} and Zn^{2+}</th></tr>
<tr></tr>
<tr><td rowspan="2">Take the ppt. to china dish, mix conc. HCl and a crystal of $KClO_3$. Evaporate the solution till dryness and observe the colour of the residue.
- Blue or green colour – for</td><td colspan="2">Boil off H_2S gas, cool and add few ml NaOH solution and then add few drops of H_2O_2. Heat the content & centrifuge.</td></tr>
<tr><td>Dark brown ppt. (for Mn^{2+}):-</td><td>Filterate (for Zn^{2+}):-</td></tr>
</table>

<table>
<tr>
<td colspan="2">Co^{2+}
- Yellow colour - for Ni^{2+}
To the residue mix few ml of distilled water.
Divide the solution into two parts.
For Co^{2+} :- For Ni^{2+}:-</td>
<td rowspan="2">Ppt. divide into two parts :-
1. Mix few ml conc. HNO_3 and pinch of PbO_2 to one part of ppt. Boil, cool and dil. with distilled water.
- Pink colour
- Mn^{2+}confirmed
2. Borax bead test:- Apply borax bead test to the second portion of ppt.
- Pink bead
- Mn^{2+}confirmed</td>
<td rowspan="2">Filterate divide into two parts:-
1. Mix few drops of dil. HCl and potassium ferrocyanide to one part of the filterate
- Bluish white ppt.
- Zn^{2+} confirmed
2. To the second part of the solution of filterate pass H_2S gas
- Dirty white ppt.
- Zn^{2+}confirmed</td>
</tr>
<tr>
<td>Mix few crystal of ammonium sulphocyanide and amyl alcohol with shaking
- Blue colour in alcohol layer
- Co^{2+} confirmed</td>
<td>Mix few drops of dimethyl gloxime and NH_4OH to second part of the solution
- Bright red colour
- Ni^{2+} confirmed</td>
</tr>
</table>

Analysis of Group V Cation (Ba^{2+},Sr^{2+},Ca^{2+}):-

From filterate of group IV, boil off H_2S gas mix solid ammonium nitrate. Boil the content, cool and then mix NH_4Cl and few drops of NH_4OH. Now mix Ammonium carbonate solution and scratch the sides of test tube for few minutes.

Centrifuge:-

- white ppt. for group V
- filterate for group VI

The above white ppt. dissolved in small amount of acetic acid and boil off CO_2 gas, cool and mix few drops of Pot. Chromate and then centrifuge.

<table>
<tr>
<td rowspan="2">Residue:-
- Yellow ppt.
- Ba^{2+}confirmed
Wash the ppt. with water and reject the washings.
Flame test :- Apply flame test with the ppt.
- Grassy green flame
- Ba^{2+}confirmed</td>
<td colspan="2">Filterate :- For Sr^{2+} and Ca^{2+}
To the filterate mix ammonia dropwise and then mix an excess of ammonium sulphate. Boil, cool and centrifuge. If no white ppt. Sr^{2+} is absent.</td>
</tr>
<tr>
<td>White ppt. :-
Sr^{2+}confirmed
Flame test :- Apply flame test with the ppt.
- Crimson red flame
- Sr^{2+} confirmed</td>
<td>Filterate for Ca^{2+}ion:-
To above filterate mix ammonium oxalate solution and wait for 2-3 minutes.
White ppt. – Ca^{2+}confirmed
Flame test :- Apply flame test with the ppt.
- Brick red flame
- Ca^{2+}confirmed</td>
</tr>
</table>

Analysis of Group VI – (Mg^{2+}, NH_4^+):-

From group V heat the filterate to dryness, cool and mix few drops of conc. HNO_3. Again heat to dryness and dissolve the residue in few ml of distilled water.

Test for Mg^{2+}	Test for NH_4^+
Mix few drops of Magneson reagent (an alkaline solution of p-nitrobenzeneazo – resorcinol – a dye) to the above solution. Sky blue ppt. – Mg^{2+}confirmed	1. Mix strong solution of caustic soda (NaOH) to a pinch of mixture. - Pungent smell, colourless gas which turns turmeric paper brown. - NH_4^+ confirmed 2. Mix NaOH solution to the pinch of mixture, heat and add Nessler's reagent (K_2HgI_4)

	Brown ppt- NH_4^+ confirmed

Section – B (Physical)

EXPERIMENT – 1

AIM:- To determine the strength and normality of given acid solution (approx. $\frac{N}{10}$ HCl) by titrating it against standard 0.5 NaOH solution conductometrically.

THEORY:-

Substances which allow electricity to pass through them are known as conductors. Conductors are divided into two classes:-

i. Metallic or electronic conductors
ii. Electrolytic conductors

The conductance of the solution of an electrolyte depends upon following factors:-

- i. Nature of the electrolyte
- ii. Concentration of the solution
- iii. Speed of the ion

In conductometric titrations, the conductance of electrolytic solution are measured with the help of conductometer.

When we titrate the strong acid with strong base, in the starting the conductance is high and then decreases till end point end after end point the conductance is increases.

PRINCIPLE:-

The basic principal of such titrations is that when the addition of solution of one electrolyte to another electrolyte solution, ions of latter solution are replaced by those of the former and hence the conductance changes. In this titration, the mixing of NaOH to HCl will cause the conductivity change due to replacement of highly mobile H^+ ions by lower mobile Na^+ ions. A particular type of curve is obtained which intersect at a point no age equilvalence point or end point. During the titration of HCl solution against NaOH solution, the following reaction takes place:-

CHEMICAL REACTION $H^+Cl^- + Na^+OH^- \rightarrow Na^+Cl^- + H_2O(l)$

When the solution of HCl is to be titrated against NaOH, the acid solution is taken in the beaker and NaOH solution is taken in the burette. The conductance of the acid solution is noted initially and after each successive mixing of small amount of NaOH solution, the change in conductance is recorded and plotting the graph. In the beginning the conductance of the acid is very high because it contains highly mobile H^+ ions. As NaOH solution is added to HCl solution, highly mobile H^+ ions are replaced by slow mobile Na^+ ions and conductance of the solution keep on decreasing till the end point is reached. After the end point is reached, the addition of NaOH brings the fast moving OH^- ions and the conductance again start increasing.

CHEMICAL USED:-

1. Standard NaOH

2. Approx. $\frac{N}{10}$ HCl solution

APPARATUS USED:-

1. Conductometer
2. Conductivity cell
3. Beaker
4. Burette
5. Pipette

PROCEDURE:-

1. Take a definite volume (20 ml) of given 0.1 N HCl in a beaker. Adding distilled water so that the electrodes of conductivity cell completely dip in the solution.
2. Wash the conductivity cell with distilled water, dip in HCl solution and connect it to conductometer.
3. Noted the conductance.
4. Rinse and fill the burette with 0.5 NaOH solution.
5. Now mix 0.5 ml of NaOH solution from the burette into beaker containing HCl solution.
6. After each addition, stir the contents thoroughly and note down the conductance.
7. Repeat the procedure.

OBSERVATION:-

Volume of given acid solution taken = 20 ml

Normality of standard NaOH = $\frac{N}{2}$ (0.5N)

Sr. No.	Volume of NaOH Added (ml)	Observed conductance (ohm^{-1})
1.	0.0	
2.	0.5	
3.	1.0	
4.	1.5	
5.	2.0	
6.	2.5	
7.	3.0	

8.	3.5	
9.	4.0	
10.	4.5	
11.	5.0	
12.	5.5	
13.	6.0	
14.	……	
15.	……	

CALCULATION:-

Suppose volume of NaOH required for neutralization = V_2 ml

Plot volume of NaOH (in ml) along x-axis, and the observed conductance along y-axis.

By using normality equation ,

$$\underset{(HCl)}{N_1V_1} = \underset{(NaOH)}{N_2V_2}$$

$$N_1 \times 20 = 0.5 \times V_2$$

∴ N_1 is normality of given HCl = $\frac{0.5 \times V_2}{20} = y$

∴ Strength of HCl = Normality × eq. wt. of HCl

$$N_1 \times 36.5 = y \times 36.5$$

$$= W \text{ g/litre}$$

PRECAUTIONS:-

1. The apparatus should be rinsed and cleaned properly before use.
2. After each mixing of the NaOH, the solution should be thoroughly stirred.
3. Rinse the electrodes of the conductivity sell thoroughly(with the given acid) before starting the experiment
4. A strong solution of NaOH should be taken, the solution of NaOH should be about five times stronger then HCl solution.

EXPERIMENT – 2

AIM:- To determine the strength of given weak acid (CH_3COOH) solution by titrating it against standard base (NaOH) solution conductometrically.

THEORY:-

In conductometric titrations the conductivity of a solution depends upon the number of ions and their conductance. The solution of electrolyte are used to find out the conductance with the help of conductometer. During the titrations the nature and sometimes concentration of ionic reactants is changed. This is because the conductance of the solution is changed.

PRINCIPLE:-

The principle of such titration is the substitution of ions of one conductance by ions of another conductance. Therefore, by the addition of definite volume of the solution of one electrolyte into the fixed volume of the solution of another electrolyte the conductance change is determined.

In case of weak acid (like CH_3COOH) against strong base (NaOH), the curve shape is found different.

CHEMICAL REACTION:-

$$CH_3COOH\ (aq) + Na^+ + OH^- \xrightarrow{-H_2O} CH_3COO^- + Na^+ + H_2O\ (l)$$

At the end point, an abrupt change occurs in the slope of the conductance titrant volume graph. Before and after the end point the curve segments are linear.

The conductance of the acid initially is very low because of low ionization of weak acetic acid. With the addition of more and more NaOH, the conductance keeps on increasing, as the number of ions in solution increasing. But the increase is slow due to low mobility of CH_3COO^- ions. After the complete neutralization of CH_3COOH, further addition of NaOH results in increase in the conductance of the solution due to increase in number of high mobile Na^+ and OH^- ions.

The point of intersection tells the exact volume of NaOH used for complete neutralization.

CHEMICAL USED:-

1. Standard NaOH solution
2. CH_3COOH solution

APPARAUTS USED:-

1. Beaker
2. Pipette
3. Burette
4. Conductometer
5. Conductivity cell

PROCEDURE:-

1. Take twenty ml of given CH_3COOH solution in a beaker. Dilute the solution so that conductivity cell dips in the solution.
2. Wash the conductivity cell with distilled water and connect it to conductometer.
3. Dip the cell in acetic acid and find out the conductance.
4. From the burette, mix 0.5 ml an of NaOH solution into CH_3COOH solution with shaking and note down the observed conductance.
5. Repeat the above procedure on recording the observed conductance.
6. Plot the graph between the observed conductance and volume of NaOH mixed and find out the volume of NaOH required complete neutralization.

OBSERVATION:-

Volume of given acetic and solution taken = 20 ml

Normality of NaOH solution =0.5 ml

S.no.	Volume of NaOH Added (ml)	Observed conductance (Ohm^{-1})
1.	0.0	
2.	0.5	
3.	1.0	
4.	1.5	
5.	2.0	
6.	2.5	
7.	3.0	

8.	3.5	
9.	4.0	
10.	4.5	
11.	……	
12.	……	

CALCULATION:-

Suppose volume of NaOH required for neutralization = V_2 ml

The values of observed conductance are plotted along y-axis and the values of volume of NaOH added along x-axis.

By using normality equation,

$$N_1V_1 = N_2V_2$$

(acetic acid) (NaOH)

$$N_1 \times 20 = 0.5 \times V_2$$

$$N_1 = \frac{0.5 \times V_2}{20}$$

N_1 i.e. normality of acetic acid,

∴ Strength of acetic acid = Normality × eq. wt.

= $N_1 \times 60$ (eq. wt. of acetic acid = 60)

= W g/litre

PRECAUTIONS:-

1. The apparatus used should be rinsed and clean properly.
2. After each mixing of the NaOH, the solution should be thoroughly stirred.
3. A strong solution of NaOH should be taken so that during titration there is not much change in the volume.
4. Before starting the experiment rinse the electrodes of the conductivity cell thoroughly with the given acid.
5. The temperature should be kept constant during the experiment.

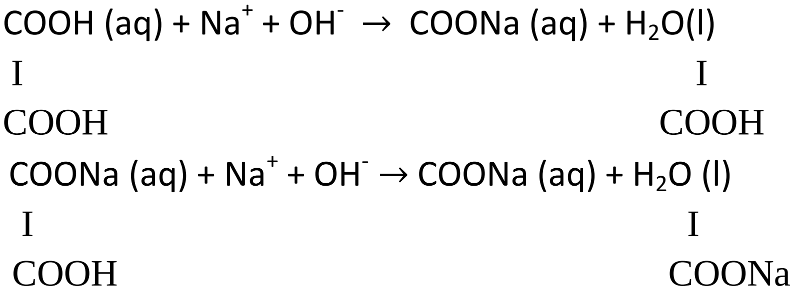
EXPERIMENT – 3

AIM:- To determine the normality and strength of given dibasic acid (oxalic acid) solution by titrating against standard NaOH solution conductometrically.

THEORY:-

In case of dibasic acid like oxalic acid vs strong base, the shape of the curve is found different. In oxalic acid, the two dissociation exists one after the other as the first dissociation is similar to that of strong acid and second one is similar to that of a weak acid like acetic acid. The titration curve shows two well marked break points.

CHEMICAL REACTION:-



Step-1 $\begin{array}{c}\text{COOH}\\ | \\ \text{COOH}\end{array}\text{(aq)} + Na^+ + OH^- \rightarrow \begin{array}{c}\text{COONa}\\ | \\ \text{COOH}\end{array}\text{(aq)} + H_2O(l)$

Step – 2 $\begin{array}{c}\text{COONa}\\ | \\ \text{COOH}\end{array}\text{(aq)} + Na^+ + OH^- \rightarrow \begin{array}{c}\text{COONa}\\ | \\ \text{COONa}\end{array}\text{(aq)} + H_2O\ (l)$

CHEMICAL USED:-

1. Oxalic acid solution
2. Standard NaOH solution

APPARATUS USED:-

1. Beaker
2. Pipette
3. Buretee
4. Conductometer
5. Conductivity cell

PROCEDURE:-

1. Take 20 ml of oxalic acid in a beaker. Dilute the solution so that conductivity cell dips in the solution.
2. Wash the conductivity cell with distilled water and connect it to the conductometer.
3. Dip the cell in the oxalic acid and find out the conductance.
4. From the burette, mix standard NaOH solution into oxalic acid solution with shaking and note down the observed conductance.
5. Repeat the procedure on recording the observed conductance.

OBSERVATION AND CALCULATION:-

Volume of oxalic acid taken = 20 ml

Normality of NaOH = $\frac{N}{10}$

Suppose the volume of NaOH required for neutralization = V ml

By using normality equation

$$N_1V_1 = N_2V_2$$

(oxalic acid) (NaOH)

$$N_1 \times 20 = .1 \times V$$

Strength (g/litre) = $\frac{V}{200} \times 45 = x\ g/litre$ $N_1 = \frac{V}{200}$

eq. wt. of oxalic acid = 45

PRECAUTIONS:-

1. The temperature should be kept constant during the experiment.
2. The apparatus used should be clean.
3. Rinse the electrodes of the conductivity cell thoroughly with the given acid.

EXPERIMENT-4

AIM:- To determine the solubility and solubility product of a given sparingly soluble salt conductometerically.

THEORY:-

Sparingly soluble salts are those which are very less soluble in solvent. When such salt is put into water, the saturated solution is obtained.

At a particular temperature the maximum amount of substance which forms the saturated solution in a fixed amount of solvent is called its solubility.

For $PbSO_4$,

$$PbSO_4 \rightleftharpoons Pb^{2+} + SO_4^{2-}$$

$$K_{sp} = [Pb^2+] [SO_4^{2-}] \qquad K_{sp} = K \times [PbSO_4]$$

The saturated solution of $PbSO_4$ is prepared. The specific conductance is determined by multiplying the observed conductance with cell constant. At infinite dilution the equivalent conductance is determined by Kohlrausch's law.

$$\lambda_0 = \lambda_0 (Pb^{2+}) + \lambda_0 ({SO_4}^{2-})$$

$$= 73 + 79.8$$

$$= 152.8 \text{ at } 298 \text{ K}$$

By using the following relation, the solubility 'S' of the sparingly soluble salt are obtained

$$S = \frac{1000 \times k}{\lambda_0} \; g \; equival/litre$$

$$= \frac{1000 \times k \times equivalent \quad weig\,ht}{\lambda_0} \; g/litre$$

$$K_{sp} = [Pb^{++}] [SO_4^{--}]$$

$$K_{sp} = S \times S$$

$$K_{sp} = S^2 \text{ in case of } PbSO_4$$

$$S = \sqrt{K_{sp}}$$

CHEMICAL USED:-

1. KCl
2. $PbSO_4$
3. Conductivity water

APPARATUS USED:-

1. Conductometer
2. Conductivity bridge
3. Conductivity cell
4. Pipette
5. Burette
6. Beaker
7. Connecting wires

PROCEDURE:-

It occurs in two steps:-

Step-1:- Determination of cell constant of cell

Step-2:- Determination of solubility and solubility product of the given sparingly soluble salt

Step-1:- Determination of cell constant of cell

I. Prepare $\frac{N}{10}$ KCl solution by dissolving 7.450g KCl in distilled water or conductivity water to prepare 100 ml solution.
II. In a beaker transfer 50 ml of this solution and dip the conductivity cell.
III. Connect the electrodes of cell to conductometer marked “cell”.
IV. The observed conductance should be recorded.
V. Repeat the procedure by taking KCl solution with normality $\frac{N}{50}, \frac{N}{100}$ etc.
VI. From ratio of specific conductance to observed conductance find out the cell constant.

Step-2:- Determination of solubility:-

In 100 ml distilled water boil 2 gm $PbSO_4$ for 5-10 minutes. To determine the conductance cool, filter and use the filterate by dipping conductivity cell. Repeat the experiment by taking fresh sample of saturated solution of $PbSO_4$.

OBSERVATION AND CALCULATION:-

I. Determination of cell constant

Room temperature =…………….t°C

Sr. No.	***Concentration (KCl)***	***Observed Conductance***	***Cell Constant***	***Mean***

1. *2.* *3.*	$\frac{N}{10}$ $\frac{N}{50}$ $\frac{N}{100}$	*….ohm-1* *….ohm-1* *….ohm-1*		

Mean value = ………………ohm^{-1}

$$\text{Cell constant} = \frac{Specific\ \ conductance}{observed\ \ conductance}\ (\text{cm}^{-1})$$

II. Determination of solubility and solubility product

Cell constant = …………….cm^{-1}

S.No.	Observed conductance	Specific conductance k = cell constant × observed conductance
1. 2. 3.	......ohm^{-1} ......ohm^{-1} ......ohm^{-1}	………… ………… Constant value of K =….

By using the relation

$$S = \frac{1000\ \times k}{\lambda_0}\ g\ equivalent/litre$$

$$\frac{1000\ \times k\ \times equivalent\ weight}{\lambda_0}\ g/litre$$

$$\text{Equivalent weight of PbSO}_4 = \frac{Molecular\ \ weight}{2} = \frac{207+32+64}{2} = \frac{303}{2} = 151.5$$

$$\Lambda_0 \text{ (PbSO}_4) = \Lambda_{0(Pb^{2+})} + \Lambda_{0(so_4{}^{2-})}$$

$$= 73 + 79.8$$

$$= 152.8 \text{ ohms at } 298K$$

By putting the values of specific conductance, equivalent wt., equivalent conductance, we can find out the value of S (solubility of sparingly soluble salt)

$$K_{sp} = [Pb^{++}]\,[SO_4{}^{--}] \text{ i.e. } S \times S^2$$

$$K_{sp} = S^2 \text{ in case of } PbSO_4.$$

PRECAUTIONS:-

I. To save the device from the damage due to voltage fluctuations, a voltage stabilizer should be used.
II. Wash the electrodes with the solution which is to be taken in the cell.
III. All solution should be prepared in conductivity water.
IV. Platinized electrodes should be used to improve the sharpness of sound.
V. No change in distance or shapes of electrodes should be made, once the cell constant is determined.
VI. Conductivity measurements are influenced by change of temperature, so these measurement should be made at controlled temperature.

EXPERIMENT – 5

AIM:- To determine the molecular weight of a non-volatile solute by Rast method.

THEORY:-

The rast method is based upon the measurement of depression of freezing point. The molten camphor is used as a solvent. Camphor has a very high molal depression constant, about 39.7^0. Even a very dilute solution of the solute in camphor has the freezing point many degrees below that of pure camphor and for the measurement of the depression of freezing point an ordinary (0.1^0) thermometer is adequate.

This method is used only to those substances which are soluble in camphor and do not react with it chemically, do not the compose below the melting point of camphor.

A known weight of compound is mixed with a known weight of the camphor till a clear homogeneous solution, cooled and then melting point of solid is determined.

By using the formula, the molecular weight M is calculated:-

$$\mathrm{M} = \frac{K_f \times 1000 \times W}{\Delta T \times w}$$

Where

W = weight of the compound,
w = weight of camphor
ΔT = depression in M.Pt
K_f = molal depression constant

CHEMICAL USED:-

1. Liquid paraffin bath
2. Non-volatile solute like naphthalene
3. Resublimed camphor

APPARATUS USED:-

1. 0.1°C thermometer
2. Capillary tubes
3. Beaker
4. Thiele's tube
5. Spatula
6. Weight box
7. Iron stand

PROCEDURE:-

I. **Preparation of homogeneous mixture**

a. Take an ignition tube, dry it and weight it accurately.
b. Mix 0.5 g of powdered camphor into it.
c. Loosely cork the ignition tube and heat it carefully on a low flame until a clear solution is obtained.
d. To ensure complete addition and then allow it to cool to get a solid mixture.

II. Determination of m.pt. of the solid mixture

a. With the help of fine edged spatula, remove the solid mixture from the ignition tube. Powder it and fill it in the capaillary tube.
b. In Thiele's tube determine the m.pt. of this solid mixture.
c. Repeat this procedure with two samples for m.pt. determination.

III. Determination of m.pt. of pure camphor

a. With the help of ether moisten some camphor and powder it carefully in a mortar.
b. By capillary tube method determine its m.pt.
c. Repeat the m.pt. determination with two samples.

OBSERVATION:-

Weight of the ignition tube = W_1 g
Organic compound + weight of ignition tube = W_2 g
Camphor + 0.c + weight of ignition tube = W_3 g
Weight of organic compound = W_2-W_1 = 0.05 g (W)
Weight of camphor = W_3-W_2 = 0.5 g (w)

M.Pt. of solid mixture			**M.Pt. of camphor**		
Experiment No.	Temperature	Mean	Experiment No.	Temperature	Mean
1. 2. 3.		t_1°C $T_1 = t_1 + 273$	1. 2. 3.		t_2°C $T_2 = t_2 + 273$

CALCULATION:-

Molal depression of camphor (k_f) = 39.7°
Depression in m.pt., $\Delta T = (T_2 - T_1)$

Using the formula, M = $\frac{k_f \times 1000 \times W}{\Delta T \times w}$

Using the above relation M, molecular weight of given organic compound.

PRECAUTIONS:-

1. The apparatus used should be clean.
2. The camphor will vapourise during heating if the ignition tube is not corked properly.
3. By giving rotation to the ignition tube following alternate melting and cooling, a homogenous mixer of the compound and camphor can be obtained.
4. When the last crystal of the solid disappears, the temperature should be noted.

EXPERIMENT-6

AIM:- To standardize the given acid solution (like HCl) pH metrically.

THEORY:-

In pH metric titrations, when some alkali is added to an acid there will be an decrease in concentration of H^+ ion and increase in pH of the solution.

pH is the negative logarithm of hydrogen ion or hydronium ion concentration.

$$pH = -\log[H_3O^+)$$

In the beginning, the pH increases slowly but at the end point, the increase is very large due to rapid increase in the fraction of the H^+ ion removed by the addition of constant volume of the alkali. After the end point, pH increases slowly. A plot of pH vs volume of alkali added will give the equivalence point.

CHEMICAL USED:-

1. Standard alkali or $\frac{N}{10}$ NaOH
2. HCl solution
3. A buffer solution of known pH (.05 M potassium hydrogen phthalate, pH = 4)
4. Distilled water

APPARATUS USED:-

1. pH meter
2. Glass and calomel electrodes
3. Burette
4. Beaker

PROCEDURE:-

1. Wash the electrodes with distilled water.
2. Take 20 ml of given acid solution in a beaker and dip the electrodes in it.
3. Measure the pH of the solution using previously calibrated pH meter with a glass electrode.
4. Mix 0.5 ml of NaOH solution from a burette each time and measure the pH after stirring the solution.

5. Mix NaOH solution dropwise near the end point, and measure the pH after each mixing.
6. Repeat the experiment to take some more readings beyond the equivalent point.

OBSERVATION:-

Volume of given HCl taken in beaker = 20 ml

Normality of NaOH solution = $\frac{N}{20}$

Volume of NaOH used for complete neutralization or to get end point = V ml

CALCULATION:-

By ploting the pH vs volume of alkali added, find the equivalence point
Using normality equation,

$$\underset{(HCl)}{N_1V_1} = \underset{(NaOH)}{N_2V_2}$$

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

$\therefore$ N_1 is Normality of HCl solution = $\frac{V}{400}$

Strength of HCl solution = Normality × Eq. wt.

$$= \frac{N}{400} \times 36.5 = x \text{ g/litre}$$

RESULT:-

Strength of given HCl solution = x g/litre

PRECAUTIONS:-

1. When electrodes not in use, should be placed in distilled water.
2. With buffer solution of known pH. pH meter should be calibrated.
3. After each a mixing solution should be stirred.
4. Near the end point, NaOH solution should be mixed dropwise.

EXPERIMENT – 7

AIM:- To determine the strength of given mono basic acid (like HCl) potentiometrically.

THEORY:-

Poteniometric method is applicable to wide range of titration, provided in indicator electrode is available an indicator electrode is the whose potential indicance the change in the concentration of ion to be titrated. The variation of potential of an electrode with the concentration of ion with which it is in equilibrium is used as an indicator in volumemetric analysis. It is not possible to determine the electrode potential separately, hence the indicator electrode is used in conjunction with a reference electrode, in which potential at one of the reference electrode remains constant during the course of titration e.g. saturated calomel electrode (SCE) while potential of other electrode depends on the activity of H^+ ion in the solution.
A solution of acid is titrated with a solution of an alkali, the following cell is setup in the acid solution.

Ag, AgCl/1MHCl || KCl aq.|| calomel electrode

The change of pH will be reflected in the change of cell potential. When a small volume of standard $\left(\frac{N}{10}\right)$ NaOH is mixed to HCl, a little change in e.m.f. is produced in the beginning . After the end point, the fraction of the hydrogen ion removed by a constant volume of the standard alkali increases rapidly.

Plot the e.m.f. against volume of the alkali added.

By plotting graph between $\frac{\Delta E}{\Delta V}$ against the volume of alkali mixed, the sharp end point is obtained.

CHEMICAL USED:-

1. Approx 0.1 N HCl solution
2. 0.1 N NaOH solution
3. KCl salt bridge
4. Pure H_2

APPARATUS USED:-

1. Potentiometer
2. Glass electrode
3. Calomel electrode
4. Beaker
5. Stirrer

6. Burette

PROCEDURE:-

I. Set all the apparatus properly.
II. Take 20 ml of acid solution in a beaker and calomel electrode is dipped into the solution to saturate the solution mix a quinhydrone.
III. Connect two electrodes to a potentiometer.
IV. Immediately above the beaker clamp a burette with $\frac{N}{10}$ NaOH solution.
V. Measure the e.m.f. of the cell.
VI. Mix 0.5 ml of NaOH solution from the burette and determine the e.m.f. of the cell after waiting for one minute.
VII. Mix another 0.5 ml of NaOH and measure e.m.f. of the solution.
VIII. Repeat the experiment similarly, after each addition 0.5 ml of NaOH.
IX. When a sudden jump in the e.m.f. is observed, stop mixing NaOH and find the approximate volume needed for reaching the end point.
X. By mixing 1 or 2 ml of alkali in initial steps, repeat the titration. By adding 1 or 2 drops of alkali at a time expected to involve the end point.
XI. Note down the e.m.f. after each addition.

OBSERVATION:-

Sr.No.	Volume of NaOH(ml)	EMF (Volts)	ΔE (Volts)	ΔV (ml)	$\frac{\Delta E}{\Delta V}$	V (ml)
1.	V_1	E_1				
2.	V2	E_2				
3.	V_3	E_3				
4.	V_4	E_4				
5.	V_5	E_5				
6.	_	_				
7.	_	_				
8.	_	_				

CALCULATION:-

Draw a graph between EMF along y-axis and volume of NaOH along x-axis. The inflexion point correspondes to the equivalence point. Then find the value of V corresponding to inflexion point.

If a clear equivalence point is not obtained then draw a graph between $\frac{\Delta E}{\Delta V}$ against volume of NaOH

By using normality equation, the normality of strength of HCl can be determined

$$\underset{\text{(acid)}}{N_1V_1} = \underset{\text{(Alkali)}}{N_2N_2} \quad N_1=?$$

$$\text{Strength} = (N_1 \times 36.5)\ \text{g/litre}$$

Section – C (Organic)

EXPERIMENT-1

AIM:- To separate green leaf pigments by thin layer chromatography and determine their R_f values.

THEORY:-

In thin layer chromatography where the stationary phase is a polar adsorbent and the mobile phase can be a single solvent or combination of solvent. A thin layer of adsorbent usually silica gel or activated alumina on a smooth surface is used as a stationary phase and chromatogram is developed by upward capillary movement of the solvent through the thin layer of adsorbent.

In principle, the components will differ insolubility and in the strength of their adsorption to the adsorbent and some components will be carried farther up plate then others. When the solvent has reached the top of the plate, the plate is removed from the developing chamber, dried and separated components are visualized. If the compounds are coloured, visualization is straight forward. Various compound on the developed TLC plates are identify through their R_f values. R_f stands for Retention factor or Ratio of Fronts.

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

CHEMICAL USED:-

1. Chloroform
2. Acetone
3. Benzene
4. Silica gel
5. Spinach leaves

APPARATUS USED:-

1. Glass plates
2. Wide mouthed bottles with stoppers
3. Glass jars with lids
4. Tongs
5. Capillary tubes
6. Beaker

7. Filter paper

PROCEDURE:-

I. Slurry of adsorbent is prepared by mixing about 30 g of silica gel in small amount to 100 ml of chloroform in a wide mouth bottle with constant swirling motion, the bottles should be tightly stopperd.
   Similarly, we can also prepare a slurry of alumina in alcohol.
II. By holding the two cleaned and dried glass plate together with tong, dipping them in the slurry of adsorbent and removing the plates quickly in this way, obtain a uniform coating of the slurry on one side of each plate. Do not dip the top and end of the plate now allow the slurry to drain and holding the top edges, separate the two plate and allow it to dry in air by placing these plate on a filter paper with slurry side upwards. These plates are known as chromatographic plates.
III. Prepare the ‘extract of leaves’ by dipping few crushed leaves of spinach in a little quantity of alcohol for 30 minutes in a beaker stirring with a glass rod the contents are filtered and the filtrate is taken as the ‘extract of leaves’.
IV. In the center of the chromatographic plate a drop of he green leaves extract is applied with the help of a capillary tube. Allow the drop dry in air.
V. In chromatography jar place the glass plate in such a way that it does not touch the side of the jar.
VI. Into the chromatography jar lower the glass plate containing the developing solvent (alcohol and water in the ratio 1:1) care should be taken that the spot does not dip into the solvent and the glass strip hangs vertically straight. With the help of a lead cover the jar.
VII. Remove the slide from the jar, when the developing solvent rises on a certain suitable distance and dry it in air.
VIII. Mark the level of the liquid with a pencil.
IX. Mark the central points of each support.
X. The distance travelled by the spots and the developing agent to be measured.
XI. Calculate the R_f values of the spots corresponding to different components.

By using the relation

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

CALCULATION:-

Distance travelled by component 1 = d_1 m

Distance travelled by component 2 = d_2 m

Distance travelled by component 3 = d_3 m

Distance travelled by developing liquid = d_4 cm

R_f value of component 1 = $\frac{d_1}{d_4}$

R_f value of component 2 = $\frac{d_2}{d_4}$

R_f value of component 3 = $\frac{d_3}{d_4}$

PRECAUTIONS:-

I. The glass plate used must be thoroughly clean and dry.
II. Fine capillary tube should be used for applying a spot of solution.
III. The glass plate should be kept erect.
IV. During the experiment, keep the glass jar always covered and undisturbed.
V. The slurry bottle must be tightly Stoppard.
VI. The spots of the solution must not dip in the developing solvent.

EXPERIMENT – 2

AIM:- To separate a mixture of coloured organic compounds (fluorescein and methylene blue) by column chromatography.

THEORY:-

In column chromatography, the stationary phase, a solid adsorbent, is placed a vertical glass, according to the amount of material which needs to be loaded on to the column, the columns may vary in sizes of their diameter and lengths. A plug of cotton wool is inserted in the neck of the tube.

Adsorbents used commonly for column chromatography are silica gel and alumina. The mobile phase, a liquid solvent is added to the top and flows down through the column by either gravity or external pressure.

As the solution passes through the column different components gets adsorbed to different extents at different level producing bands is selected a solvent or eluent is added, as soon as the last portion of the solution and enters the column. It acts as a moving phase. The selected solvent dissolve out the different components from the different zones selectively and thus work out the different band in the form of fraction which are collected separately. To get the components in pure form, the eluent from each fraction is then distilled off.

CHEMICAL USED:-

1. Alumina 100-200 mesh
2. Methylene blue
3. Sodium salt of fluorescein
4. Rectified spirit

APPARATUS USED:-

1. Chromatography column
2. Test tube
3. Beaker

PROCEDURE:-

1. Using rectified spirit prepare a slurry of 50-100 mg of activated alumina and pour this slurry into the column. On the top of the adsorbent leave a space of 4-5 cm for addition of solvent.
2. Mix solvent to the top of the adservent. The solvent flows slowly down the column. At the top level monitor the solvent, it does not go below the top of the adsorbent.
3. As the solvent close through the alumina and its level reaches the bottom of the column, the processes of pre-elution is complete and the column is ready to load.
4. In about 5 ml of rectified spirit dissolve 5 mg of methylene blue and sodium salt of fluorescein, the sample is ready to be loaded in the column. Only a few drops of solvent use to load the sample into the column.
5. Add rectified spirit, when the sample is introduced in the column to develop the chromatogram. After some time, blue colour of methylene blue travels down the column while fluoresecin remains at the top.
6. Continue the process till whole of methylene blue has passed into the receiver placed below the column and the same becomes colourless.
7. Rectified spirit is placed by water has eluent solvent when fluorescein starts moving down the column. change the collection beaker and continue elution till whole of fluorescein is recovered.

PRECAUTIONS:-

1. To prevent the drying out of the column, the upper surface of the column always remains covered with the solvent throughout the experiment.
2. Eluents must be low boiling liquids show as to facilitate the quick removal from the fractions collected.

EXPERIMENT – 3

AIM:- To seprate a mixture of O- and p- nitrophenols by steam distillation.

THEORY:-

The process is use for purification of those substances from no-volatile impurities which themselves are steam volatile but insoluble in water at the same time.

For such a system, where to immiscible liquids are brough to gather which do not mix at all, each liquid behaves independent of the other and exerts a vapour pressure.

O-nitrophenol is steam volatile as it shows intra molecular hydrogen bonding and has a boiling point lower than p-nitrophenol which shows inter molecular hydrogen bonding. So they can be separated by steam distillation.

O-nitrophenol → p-nitrophenol
(intramolecular H-Bonding) (intermolecular H-bonding)

CHEMICAL USED:-

1. Ortho and para nitrophenol
2. 2% HCl solution
3. Powdered charcoal

APPARATUS USED:-

1. Round bottom flask
2. Sand bath
3. Filter paper
4. Buchner funnel
5. Condenser tube
6. Burner
7. Steam generator

PROCEDURE:-

1. Take the mixture of o-and p-nitrophenol in the round bottom flask.
2. While passing steam through it simultaneously, heat it on a sand bath, when ortho-nitrophenol passes over and para-isomer is left behind.
3. In ice cold water cool the distillate.
4. Filter at the pump and allow to drain.

5. Between folds of filter paper dry the compound and determine the melting point.
6. The removal of ortho-nitrophenol by steam distillation, cool the residue in the flask in ice for few minutes and filter.
7. For about five minute boil the crude para-nitropheno, with 2% solution of HCl with a little powdered charcoal.
8. Through a Buchner funnel filter the solution and allow to cool gradually when colourless needle like crystal of para-nitrophenol separate out.
9. Dry them by pressing between folds of filter paper and not its melting point.
10. When ortho-nitrophenol solidifies in the condenser tube, stop passing cold water through the condenser.

PRECAUTION:-

1. All the apparatus used in experiment should be clean and dry.
2. Set all the apparatus properly.
3. Follow all the steps carefully.
4. To avoid the condensation of water, the round bottom flask is being simultaneously heated on a sand bath.

EXPERIMENT – 4

AIM:- To prepare O-chlororobenzoic acid from anthranilic acid.

THEORY:-

First anthranilic acid is diazotized to give desired product of diazonium salt. In a diazonium salt, diazo group is replaced by halogen atom by treating the diazonium salt with cuprous halide in presence of HCl.

CHEMICAL REACTION:-

$$\text{Anthranilic acid} \xrightarrow[0-5^\circ C]{NaNO_2/HCl} \text{diazonium salt} \xrightarrow{Cu_2Cl/HCl} \text{o-Chlorobenzoic acid}$$

CHEMICAL USED:-

1. Anthranilic acid = 6.0 g
2. Sodium nitrite = 3.0 g
3. Copper sulphate = 10 g
4. Sodium chloride = 10 g
5. Conc. HCl = 40 ml
6. Copper turnings = 6.0 g
7. Ice = as needed

APPARATUS USED:-

1. Round bottom flask
2. Ice bath
3. Beaker
4. Burner
5. Iron stand
6. Droper
7. Glass rod
8. Filter paper etc.

PROCEDURE:-

1. Prepare a solution of cuprous chloride as follows :-
   Dissolve 10 g of copper sulphate and 10 g of NaCl (sodium chloride) in 30ml of distilled water taken in a round bottom glass. Boil the solution and add 30ml conc. HCl and 6g copper turnings. Hit the contants till a clear solution is obtained. Filter and full the filterate in an ice bath.

2. Take 6 g anthranilic acid and 10ml conc. HCl in a beaker. Add about 40ml distilled water and hit the contents to get a clear solution. Then, cool the solution to 0-5℃ in an ice bath.
3. To the anthranilic acid solution, add a solution of 3g of sodium nitrite prepared in 10ml of distilled water dropwise. Taking care the temperature should remains below 5℃.
4. Add the above diazonium salt solution in small instalments to the cold cuprous chloride solution prepared in the first step with constant stirring.
5. Allow the reaction mixture to stand for about half an hour at room temperature.
6. Filter the crude product and crystallize from boiling water containing a little amount alcohol.

OBSERVATION:-

I. Colour = white crystalline solid.
II. Yield = 5g
III. M.Pt. = 138℃

PRECAUTIONS:-

1. The colour of cuprous chloride solution should be colourless.
2. During diazotization of anthranilic acid, the temperature should be kept below 5℃.
3. To avoid the decomposition on standing, the diazonium salt solution should be added immediately to the cuprous chloride solution.

EXPERIMENT – 5

AIM:- To prepare p-Bromonaniline from p-Bromoacetanilide.

THEORY:-

p-Bromonaniline can be obtained by the hydrolysis of acetanilide with an alkali.

CHEMICAL REACTION:-

$$\text{p-Bromoacetanilide} + H_2O \xrightarrow{KOH} \text{p-Bromoaniline} + CH_3COOH$$

CHEMICAL USED:-

1. P-Bromoacetanilide = 7.5 g
2. Ethyl alcohol = 25-30 ml
3. Potassium hydroxide = 4.5 g
4. Ice = As needed

APPARATUS USED:-

1. Round bottom flask
2. A reflux water condenser
3. Beaker
4. Water bath
5. Glass rod
6. Iron stand
7. Burner

PROCEDURE:-

1. Take 7.5g of p-bromoacetanilide & 15 ml ethyl alcohol in a 100 ml round bottom flask fitted with a reflux water condenser. Heat the contents to get clear solution.
2. Mix a solution of 4.5g KOH dissolved in 7.8 ml distilled water to this solution, boil the contents of the flask for about 30 minutes.
3. Transfer the contents of round bottom flask to a 250 ml beaker and add 50ml of water. By heating on a water bath concentrate the solution to two – thirds of its volume.
4. In a beaker a containing 60-70 ml ice cold water, cool and pour the contents with constant stirring, when an oily product separates out which soon solidifies.
5. Filter and crystallize the crude product from 50% ethyl alcohol to get white crystalline solid.

RESULT:-

I. Yield = about 5g
II. Colour = white crystalline solid
III. M.Pt. = 66℃

PRECAUTIONS:-

p-Bromoaniline separates as an oily liquid which soon solidifies. In case does not solidify, scratch the sides of the beaker with a glass rod and add more ice.

EXPERIMENT – 6

AIM:- To prepare m-nitroaniline from m-dinitro benzene.

THEORY:-

m-nitroaniline can be obtained by reducing m-dinitrobenzene partially either with sodium polysulphide or with alcoholic solution of ammonium hydrogen sulphide.

CHEMICAL REACTION:-

m-dinitrobenzene + Na_2S_2+ H_2O → m-Nitroaniline + $Na_2S_2O_3$

m-dinitrobenzene + $3NH_4SH$ → m-Nitroaniline +3S + $2H_2O$ + $3NH_3$

CHEMICAL USED:-

1. m-Dinitrobenzene = 7.5g
2. Sodium sulphide = 12.0g
3. Sulphur powder = 4.5g
4. Ice = as needed

APPARATUS USED:-

1. Separating funnel
2. Beaker
3. Glass rod
4. Burner
5. Iron stand

PROCEDURE:-

I. First prepare polysulphide solution is formed as follows:
Dissolve 12g sodium sulphide in 50 ml distilled water. Mix 4.5g of sulphur powder to it and heat the contents gently till a clear solution is obtained.

II. Now take 7.5g of m-dinitrobenzene and 150 ml of water in 400 ml beaker. Heat to boil stir the solution.

III. Transfer the sodium poly sulphide solutions prepared in first step to a separating funnel and clamp it over the beaker containing m-dinitro benzene solution.

IV. Mix sodium poly sulphide solution with constrant stirring to the boiling mixer in small instalments so that mixing is complete in about 10 minutes boil the contains for 20 minutes more.

V. In the beaker, cool and add crushed ice when crude m-nitroaniline separates out.

VI. Filter crude product and wash with cold water.

VII. Now transfer the crude product to a beaker containing in about 50 ml water and 12 ml conc. HCl. Boil for 15 minutes when m-nitroaniline passes into solution as its hydrochloride, leaving behind sulphur. Filter the solution.

VIII. To the filerate mix conc. Aqueous ammonia when m-nitroaniline separates out.

IX. Filter and recrystallise from volume water.

RESULT:-

I. Yield = about 5g

II. Colour = Bright yellow needle like crystals

III. M.Pt. = 114°C

EXPERIMENT -7

AIM:- To prepare S-Benzyl – iso – thiouronium chloride from thiourea.

THEORY:-

By treating benzyl chloride with thiourea in alcoholic solutions, S-Benzyl – iso – thiouronium chloride can be obtained.

CHEMICAL REACTION:-

Benzyl chloride + Thiourea → S-Benzyl – iso – thiouronium chloride

CHEMICAL USED:-

1. Benzyl chloride = 4g
2. Thiourea = 2.5g
3. Reactified spirit = 5ml

PROCEDURE:-

1. Take 4g benzyl chloride, 2.5g thiourea and 5ml rectified spirit in 100 ml round bottom flask which is fitted with a reflux condenser.
2. Warm the contents on a water bath. An exothermic reaction occurs immediately and whole of thiourea passes into the solution.
3. Reflex the resulting solution of round bottom flask for about 30 minutes and cool in ice cold water when white crystal of S-Benzyl is thiouronium chloride separate out.
4. Filter under suction and allow the crystals to dry in air.
5. Recrystallise the crude product from 0.2 N HCl.

RESULT:-

I. Yield = 5g (about)

II. Colour = white crystal

III. M.Pt. = 175℃

EXPERIMENT -7

AIM:- To prepare S-Benzyl – iso – thiouronium chloride from thiourea.

THEORY:-

By treating benzyl chloride with thiourea in alcoholic solutions, S-Benzyl – iso – thiouronium chloride can be obtained.

CHEMICAL REACTION:-

Benzyl chloride + Thiourea → S-Benzyl – iso – thiouronium chloride

CHEMICAL USED:-

1. Benzyl chloride = 4g
2. Thiourea = 2.5g
3. Reactified spirit = 5ml

PROCEDURE:-

1. Take 4g benzyl chloride, 2.5g thiourea and 5ml rectified spirit in 100 ml round bottom flask which is fitted with a reflux condenser.
2. Warm the contents on a water bath. An exothermic reaction occurs immediately and whole of thiourea passes into the solution.
3. Reflex the resulting solution of round bottom flask for about 30 minutes and cool in ice cold water when white crystal of S-Benzyl is thiouronium chloride separate out.
4. Filter under suction and allow the crystals to dry in air.
5. Recrystallise the crude product from 0.2 N HCl.

RESULT:-

I. Yield = 5g (about)
II. Colour = white crystal
III. M.Pt. = 175°C