RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029

Lab Manual

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

Department of Chemistry

CHEMISTRY

B.Sc. IIIrd Year

INDEX

SECTION – A: INORGANIC CHEMISTRY

1. **SEMMICRO QUALITATIVE ANALYSIS**
   To analyse the given mixture for anions (acid radicals) and cations (basic radicals).
   Pb^{2+}, Hg^{2+}, Ag^{+}, Bi^{3+}, Cu^{2+}, As^{3+}, Sb^{3+}, Sn^{2+}, Fe^{3+}, Cr^{3+}, Al^{3+}, Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+}, Ba^{2+}, Sr^{2+}, Ca^{2+}, Mg^{2+}, NH_4^{+}, CO_3^{2-}, S^{2-}, SO_3^{2-}, $S_2O_3^{2-}$, NO_2^{-}, CH_3COO^{-}, Cl^{-}, Br^{-}, I^{-}, NO_3^{-}, SO_4^{2-}, $C_2O_4^{2-}$, PO_4^{3-}, BO_3^{3-}

SECTION – B: PHYSICAL CHEMISTRY

1. 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.
2. To determine the strength of given weak acid (CH_3COOH) solution by titrating it against standard base (NaOH) solution conductometrically.
3. To determine the normality and strength of given dibasic acid (oxalic acid) solution by titrating against standard NaOH solution conductometrically.
4. To determine the solubility and solubility product of a given sparingly soluble salt conductometerically.
5. To determine the molecular weight of a non-volatile solute by Rast method.
6. To standardize the given acid solution (like HCl) pH metrically.
7. To determine the strength of given mono basic acid (like HCl) potentiometrically.

SECTION – C: ORGANIC CHEMISTRY

1. **Thin Layer Chromatography**

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

2. **Laboratory Techniques**
   - **a) Colum Chromatography**

     To separate a mixture of coloured organic compounds (fluorescein and methylene blue) by column chromatography.
   - **b) Steam distillation**

     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.
   - **b)** To prepare p-Bromonaniline from p-Bromoacetanilide.
   - **c)** To prepare m-nitroaniline from m-dinitro benzene.
   - **d)** To prepare S-Benzyl – iso – thiouronium chloride from thiourea.

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>Black ppt. for Co^{2+} and Ni^{2+}</th><th colspan="2">Filterate for Mn^{2+} and Zn^{2+}</th></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>

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+}:-		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	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$_2$S gas - Dirty white ppt. - Zn^{2+}confirmed
Mix few crystal of ammonium sulphocyanide and amyl alcohol with shaking - Blue colour in alcohol layer - Co^{2+} confirmed	Mix few drops of dimethyl gloxime and NH$_4$OH to second part of the solution - Bright red colour - Ni^{2+} confirmed		

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

From filterate of group IV, boil off H$_2$S gas mix solid ammonium nitrate. Boil the content, cool and then mix NH$_4$Cl and few drops of NH$_4$OH. 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 ,

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

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

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

$\therefore$ 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.

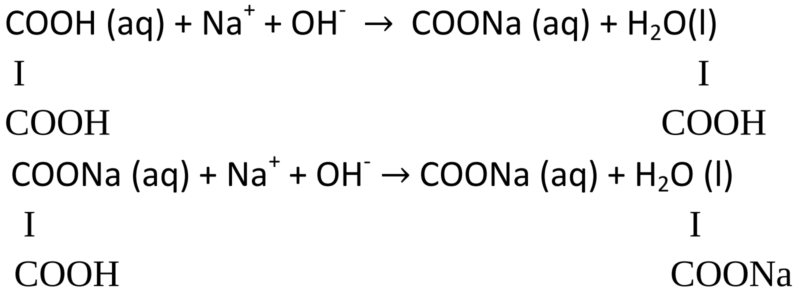
EXPERIMENT – 3

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 $\underset{\substack{|\\ \text{COOH}}}{\text{COOH}}\ (aq) + Na^+ + OH^- \rightarrow \underset{\substack{|\\ \text{COOH}}}{\text{COONa}}\ (aq) + H_2O(l)$

Step – 2 $\underset{\substack{|\\ \text{COOH}}}{\text{COONa}}\ (aq) + Na^+ + OH^- \rightarrow \underset{\substack{|\\ \text{COONa}}}{\text{COONa}}\ (aq) + H_2O\ (l)$

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

Mean value = ………………ohm^{-1}

Cell constant = $\frac{Specific\ \ conductance}{observed\ \ conductance}$ (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.	......ohm^{-1}	…………
2.	......ohm^{-1}	…………
3.	......ohm^{-1}	Constant value of K =….

By using the relation

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

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

Equivalent weight of $PbSO_4$ = $\frac{Molecular\ \ weight}{2} = \frac{207+32+64}{2} = \frac{303}{2} = 151.5$

$$\Lambda_0 \ (PbSO_4) = \Lambda_{0(Pb^{2+})} + \Lambda_{0(so_4{}^{2-})}$$

$$= 73 + 79.8$$

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

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

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

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

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

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:-

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

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℃