RPS DEGREE COLLEGE

BALANA (MAHENDERGARH)-123029

Lab Manual

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

Department of Chemistry

INORGANIC CHEMISTRY

B.Sc. Hons. Ist Year

INDEX

SEMESTER 1

1. **SEMi MICRO QUALITATIVE ANALYSIS**
   To analyse the given mixture for anions (acid radicals) and cations (basic radicals), including interfering and excluding insoluble:
   Pb^{2+}, Hg^{2+}, Hg_2^{2+}, Ag^{+}, Bi^{3+}, Cu^{2+}, Cd^{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-}

SEMESTER 2

Volumetric Analysis:

1. To determine the strength of a given solution of sodium hydroxide solution by titrating it against a standard solution of oxalic acid.
2. 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
3. Given solution was prepared by dissolving 4g of ferrous oxalate in dil. H_2SO_4 and volume made to one litre. Determine volumetrically.
   (i) % purity of the sample.
   (ii)% of oxalate ions in the sample.

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

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

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 $K_2Cr_2O_7$ and few ml of conc. H_2SO_4 pass the red vapours through NaOH solution.	Red vapours of Chromyl Chloride are formed	Cl^- present
	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	Orange coloure in CS_2 or CCl_4 layer	Br^-confirmed

or CCl_4 and few ml of 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 with conc. H_2SO_4	A mixture of CO and CO_2 evolved	Oxalate ion may be 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	Purple streaks	SO_4^{2-} confirmed

flame till charred mass. Through this mass in sodium nitroprusside 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	White ppt.	PO_4^{3-} confirmed

part of SE add dil. HCl, boil of CO_2 gas and cool. Add NH_4OH solution till alkaline and then add 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	Dil.HCl	H_2S gas in	H_2S gas	NH_4OH	H_2S gas	$(NH_4)_2Co_3$	No

Reagent		presence of dil. HCl	in presence of dil. HCl	in presence of NH_4CL	in presence of dil. HCl	in presence of NH_4Cl & NH_4OH	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	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

II- Supernate or filtrate (for Ag^+) :- To filtrate add few drops of dil. HNO_3 -white ppt. – Ag^+ 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</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>

	mix $SnCl_2$ solution. White ppt. turns grey - Hg^{2+} confirmed	

Coloured ppt.- for Group II	**Filterate – Group III**
Test for As^{3+} :- 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	Ppt for Pb^{2+} 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 colspan="2">Black ppt. for Co^{2+} and Ni^{2+}</th><th colspan="2">Filterate for Mn^{2+} and Zn^{2+}</th></tr>
<tr><td colspan="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 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 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 colspan="2" rowspan="2"></td><td>Dark brown ppt. (for Mn^{2+}):-</td><td>Filterate (for Zn^{2+}):-</td></tr>
<tr><td>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</td><td>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</td><td>Mix few drops of dimethyl gloxime and NH_4OH to second part of the solution
- Bright red colour
- Ni^{2+}</td><td colspan="2"></td></tr>
</table>

alcohol layer - Co^{2+} confirmed	confirmed	- Mn^{2+}confirmed	

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</td></tr>
</table>

		- Ca^{2+}confirmed

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

SEMESTER 2

EXPERIMENT – 1

Aim

To determine the strength of a given solution of sodium hydroxide solution by titrating it against a standard solution of oxalic acid.

Theory

The determination of the strength of a solution of an acid by titration with a standard solution of a base is called acidimetry, whereas when the strength of a solution of an alkali is determined by means of titration with standard solution of an acid is termed as alkalimetry.

This estimation involves titration of a weak acid that is oxalic acid against a strong base is sodium hydroxide and phenolphthalein is the indicator of choice. The reaction between oxalic acid and sodium hydroxide is

$$(COOH)_2 + 2NaOH \rightarrow (COONa)_2 + 2H_2O$$

Since sodium hydroxide is not a primary standard a standard solution of oxalic acid is prepared and used for standardisation of sodium hydroxide.

In acid base titration at the end point the amount of acid becomes chemically equivalent to the amount of base present. In case of strong acid and strong base titration at the end point of solution the solution become neutral.

Materials Required

1. Burette
2. Pipette
3. Conical flask
4. Burette stand
5. Funnel
6. Stirrer
7. White glazed tile
8. Measuring flask
9. Oxalic acid (solid)
10. Sodium hydroxide solution
11. Phenolphthalein indicator

Procedure

(a) Preparation of 0.1M Standard Oxalic Acid Solution

1. Take a watch glass, wash it with distilled water and dry it.
2. Weigh the exact amount of clean and dried watch glass and record its weight in the notebook.
3. Weigh correctly on the watch glass 3.15 g of oxalic acid and record this weight in the notebook.
4. Using a funnel, transfer oxalic acid softly and carefully from the watch glass into a clean and dry measuring flask.
5. Wash the watch glass with distilled water to move the particles that stick to it into the foam with the assistance of a wash bottle.
6. For this purpose, the volume of distilled water should not exceed 50 ml.
7. Wash funnel several times with distilled water to move the sticking particles into the measuring flask using a wash bottle. Add water in tiny quantities while washing the funnel. The distilled water quantity used for this purpose should not exceed 50 mL.
8. Using a wash bottle, wash the funnel carefully with distilled water to pass the solution attached to the funnel into the measuring flask
9. Turn the flask of measurement until the oxalic acid dissolves.
10. Using a wash bottle, thoroughly add enough distilled water to the measuring flask just below the etched mark on it.
11. Add the last few mL of distilled water drop into the measuring flask until the reduced meniscus level just touches the mark.
12. Put the stopper on the mouth of the flask and shake softly to make the entire solution uniform. Calculate it as a solution of oxalic acid M/10.

(b) Titration of Sodium Hydroxide and Oxalic Acid Solution

1. Rinse the burette with the standard oxalic acid solution.
2. Take 10cm^3 of oxalic acid solution in a titration flask. Fill the burette with sodium hydroxide solution.
3. Remove the air gap if any, from the burette by running the solution forcefully from the burette nozzle and note the initial reading
4. Pipette out 20ml of NaOH solution is a conical flask. Add 2-3 drops of phenolphthalein indicator to it.
5. Titrate the base with oxalic acid solution until pink colour disappears.
6. Repeat the titration till three concordant readings are obtained.

Observations

1. Molarity of oxalic acid solution = M10
2. Molarity of sodium hydroxide solution = x
3. Volume of oxalic acid solution = 10cm^3
4. Indicator = Phenolphthalein
5. End point = Light pink colour

S.No	Initial Reading of the Burette	Final Reading of the Burette	Volume of NaOH solution used	Concordant Reading
1	a cm^3	b cm^3	(b-a) cm^3	V cm^3
2	b cm^3	c cm^3	(c-b) cm^3	V cm^3
3	c cm^3	d cm^3	(d-c) cm^3	V cm^3

Calculations

Mass of oxalic acid dissolved in 100ml of standard solution = y g

Strength of oxalic acid = y × 10 g/L
Normality (N) of standard oxalic acid = Strength/ Eq.wt = y×1063.04 = N
Normality (N_1) of sodium hydroxide solution

$N_1 \times V_1 = N \times V$
Therefore,

$N_1 = N \times V/V1$
Normality (N_2) of given oxalic acid solution

$N_2 \times V_2 = N_1 \times V_1$
$N_2 = N1 \times V1/V2$
Strength of given oxalic acid = N_2 x 63.04 g/L

Results and Discussion

The strength of the given sodium hydroxide solution is _____ g/L.

Precautions

1. Weighing of oxalic acid crystals need weights of 2g + 1g + 100mg + 50mg.
2. While weighing do not spill the substance on balance pan.
3. Rotate the knob of balance gently.
4. Keep the weights in weights box at proper places after weighing
5. Wash the watch glass carefully so that even a single crystal is not left on the watch glass.
6. Bring the watch glass close to funnel while transferring weighed substance and transfer it gently. Wash it repeatedly with distilled water.
7. Wash the burette with water after titration is over.
8. Last few drops should be added using pipette to avoid extra addition of distilled water above the mark on the neck of the measuring cylinder.

EXPERIMENT 2

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

APPARATUS REQUIRED:-

Burette, conical flask, dropper, glass rod

CHEMICAL REQUIRED:-

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

THEORY:-

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

Chemical equation:

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

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

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

First titration:- $FeSO_4$ against $KMnO_4$

Indicator:- $KMnO_4$ acts as a self indicator

End point:- Appearance of light pink colour

PROCEDURE:-

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

OBSERVATION:-

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

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

Concordante volume = V ml

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

Indicator:- $KMnO_4$ acts as a self indicator

End point:- Appearance of light pink colour

PROCEDURE:-

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

OBSERVATION:-

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

Concordant volume = X ml

Calculations:-

Ist titration:- Using normality equation

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

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

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

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

2nd titration:- Again using normality equation

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

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

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

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

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

$= N_1 \times 284$

(∴ eq. wt. Of anhydrous salt is 284)

Now using the relation

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

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

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

PRECAUTIONS:-

(i) The apparatus should be cleaned and dried.

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

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

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

RESULT:-

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

EXPERIMENT – 3

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 = $\frac{Y}{4}$ × 100 = a (say)

RESULT:-

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

EXPERIMENT – 4

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

$\therefore$ Weight of solid $K_2Cr_2O_7$ = 0.6125g

Volume of solution made = 250ml

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

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

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

Concordant volume = V ml

CALCULATIONS:-

Using normality equation

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

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

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

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

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

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

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

RESULT:-

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

PRECAUTIONS:-

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

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