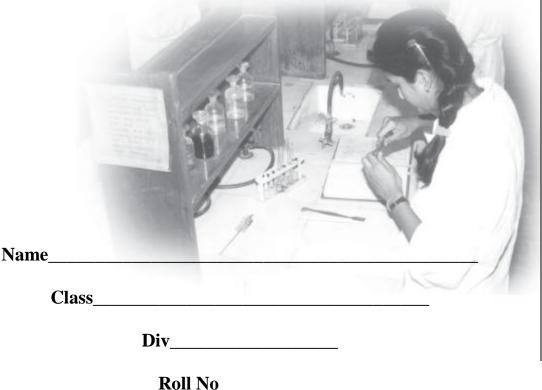
Chemistry chart

For

Standard XI Science



Batch No

INORGANIC VOLUMETRIC ANALYSIS

Acid-Base titration

Experiment No-1

Date: _____

XI Science

Aim: You are provided with two solutions as follows

Flask X - HCl Flask Y - (0. 1050N) NaOH Titrating these solutions determine Normality and strength in terms of g/litre. of HCl solution.

Requirements:

(i). Apparatus: Burette. Stand Pipette. Conical flask. White tile(ii)Chemicals: NaOH (0. 1050 N). HCl and Phenolphthalein

Theory: This is a titration of strong acid HCl and strong base NaOH, where they react with each other and fonn salt and water. The process is called neutralization. Using the normality of NaOH. The strength of HCl can be calculated in terms of normality and grams/litre

Procedure: (i) Wash all the apparatus with water.

(ii) Rinse the pipette with HCl solution. Pipette out 10 ml of HCl solution in conical flask. Add a drop of phenolphthalein indicator.
(iii) Rinse the burette with NaOH solution and fill it with NaOH.
Remove the air bubble if any. Adjust zero mark with lower meniscus.
(iv) Titrate HCl solution against NaOH by adding NaOH from burette one ml at a time till the solution becomes light pink by constant stirring. Note this reading as pilot reading.

(v) Repeat the titration to get constant reading.

Solution	Normality	g/Litre.
HCI	N	
NaOH	0.1050 N	

Result:

Observations and Calculations.

Pilot Reading -----ml to -----ml

Given: 0.1050N NaOH solution

To find : Normality & strength of HCI solution in terms of grams/litre

- (i) Solution in Burette: NaOH (0. 1050N)
- (ii) Solution in Pipette: HCI Solution
- (iii) Solution in conical flask:10 ml of HCI + two drops of indicator
- (iv) Indicator: Phenolphthalein
- (v) End point: Colourless to light pink

Equation: $NaOH + HCI \rightarrow NaCI + H_2O$

Observation Table:

Burette **Constant Burette** I Ш ш Reading Reading (C.B.R.) Final mL ... mL mL Initial 0mL 0mL 0mL mL Difference mL mL mL

Calculations:

1. To calculate Normality of HCI Solution (NaOH)

n (NaOH) (HCI) $N_1V_1 = N_2V_2$ N_1 = Normality of NaOH V_1 = Volume of NaOH (i.e. C.B.R) N_2 = Normality of HCI V_2 = Volume of HCI

0.1050 x C.B.R = N₂? x 10

N₂ = <u>0.1050 x C.B.R.</u> 10 =-----'N Normality of HCI =------N

2. To calculate g/L of HCI = Normality of HCI x Eq.wt. Of HCI -----N x 36.5 = ------ g/L

Note:-(Show log calculations in pencil)

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Date:----

INORGANIC VOLUMETRIC ANALYSIS REDOX TITRATIONS

Experiment No:-

Aim:- You are provided with two solutions as follows.

Container A: ----- N Solution of Hydrated Mohr's Salt.

Container B: - <u>KMnO₄</u> Solution.

Titrating these solutions determine the

- 1. Normality of the solution in container B.
- 2. Strength of less /more concentrated solution in container B/C in terms of gms/_____ml.

Apparatus & Chemicals Required:-

Burette, Pipette conical flask Burette stand white tile, KMnO₄ Solution , Hydrated Mohr's Salt and Dil. sulphuric acid

Theory:-

This is a Redox titration where Neutralization point is due to Reduction and Oxidation reaction occurring simultaneously. In this titration - $\underline{KMnO_4}$ undergoes reduction with change in oxidation number of manganese ion from +7 to +2 and Mohr's Salt undergoes oxidation with change in oxidation number of iron+2(Ferrous) to +3(Ferric) Thus acts as a - $\underline{KMnO_4}$ Oxidizing agent and Mohr's Salt acts as reducing agent and at the end point colour change is from colourless to light pink

(b) Ionic equation

Reduction half reaction : $MnO_4^- + 5e^- + 8H^+ \longrightarrow Mn^{2+} + 4H_2O$ Oxidation half reaction : $Fe^{2+} \longrightarrow Fe^{3+} + e^-] \times 5$

 $MnO_4^- + 5Fe^{2+} + 8H^+ \longrightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

Procedure:

- 1. Rinse the pipette with the given _____ N Mohr's Salt solution and pipette out 10 ml of it in a washed titration flask.
- 2. Rinse and fill the burette with the given $KMnO_4$ solution.
- 3. Add one test-tube (15 ml) or One and half (if test tube size is small) test tube of dilute sulphuric acid (2 N) to the solution in the titration flask.
- 4. Note the initial reading of the burette.
- 5. Add KMnO₄ solution from the burette till a permanent light pink colour is imparted to the solution in the titration flask on addition of the last single drop of KMnO₄ solution.
- 6. Note the final reading of the burette.
- 7. Repeat the above steps to get three concordant readings.

Result:

Solution	Normality	Grams/ml
<u>KMnO₄</u>		
Mohr's Salt		

____Solution is Less/More Concentrated in terms of Normality/ gms per -----ml

- 1. Given:-
- 2. To find:

Observations:

- 1. Solution in Burette : KMn0₄solution.
- 2. Pipette Solution: Mohr's Salt Solution
- 3. Solution in Flask: 10 ml of ----- N /M Mohr's Salt+ 1test tube di!. H₂SO₄,
- 4. Indicator: KMnO₄ acts as self indicator.
- 5. End point: Colourless to light pink.

Chemical equations:

$$\begin{split} 2\text{KMnO}_4 + 8\text{H}_2\text{SO}_4 + 10\text{FeSO}_4\ (\text{NH}_4)_2\ \text{SO}_4 \cdot 6\text{H}_2\text{O} \longrightarrow \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 5\text{Fe}_2(\text{SO}_4)_3 \\ &\quad + 10(\text{NH}_4)_2\text{SO}_4 + 68\text{H}_2\text{O} \end{split}$$

Observation Table:

Pilot Reading	ml To	ml.
-		

Burette Reading	I	н		Constant Burette Reading (C.B.R.)
Final	mL	mL	mL	
Initial	OmL	OmL	OmL	mL
Difference	mL	mL	mL	

Calculations:

- 1. To calculate Normality of : $KMn0_4$ solution = N_1V_1 = N_2V_2
- 2. To calculate Grams per -----ml

=N X Eq.Wt X -----/1000

Physical Experiments

Determination of pH values

Aim: To determine the pH value of the given solutions namely washing soda, lemon juice, vinegar, milk, tomato juice, tap water, etc

Apparatus required: 5 droppers, test tubes, test-tube stand, and glazed tile.

Chemicals required: Universal indicator paper, distilled water, washing soda, vinegar, lemon juice, milk, tomato juice, tap water.

Theory: The pH value of a solution is defined as the negative logarithm of hydronium ion concentration in mol/L.

 $pH = -\log [H^+]$

If the hydronium ion concentration is 1×10^{-7} mol/L, it may be stated that its pH is 7 and the solution is neutral solution.

Pure water $[H_3O^+] = 1 \times 10^{-7} \text{ mol/L}$

Therefore, the pH = 7

The pH of an acidic solution is below 7

The pH of a basic solution is above 7

Since $[OH^{-}] = 1 \times 10^{-14}$

Therefore, Hydroxide ion concentration can vary from $1 \ge 10^{-7}$ to $1 \ge 10^{-14}$ and Hydronium ion concentration can vary from $1 \ge 10^{-1}$ to $1 \ge 10^{-7}$.

Thus pH values vary from 1 to 14. This is known as pH scale.

Procedure:

- 1. Prepare solutions in small quantities of lemon juice, milk, tomato juice etc.
- 2. Take the solution of the sample and arrange them on a test tube stand.
- 3. Place a drop of the test solution on the indicator paper with the help of a dropper.
- 4. Observe the colour produced and compare it with the colour on the chart.
- 5. Read the pH value from the chart given against the colour obtained on the indicator paper.
- 6. Repeat the experiment with other solutions.

Result:

The sample of solution____, ____ are acidic, sample _____ and _____ are basic, and the ______ sample is neutral.

pH Scale

[H ₃ O ⁺] Mol/L	10^{0}	10-1	10 ⁻²	10-3	10-4	10-5	10-6	1()-7	10-8	10-9	10-10	10-11	10 ⁻¹²	10-13	10-14
pН	0	1	2	3	4	5	6		7	8	9	10	11	12	13	14
								Neu	ıtral					5		
	Acidic			Basic												

Observation Table

With pH paper

Sr.	Sample of Solution	Shade of Colour	pН	Acidic, Basic or Neutral
No.				.0.9
1	Vinegar			
2	Tomato Juice		0	
3	Lemon Juice			
4	Milk			
5	Tap water			
6	Washing soda	S.		

Observation Table

With universal indicator

Sr. No.	Sample of Solution	Shade of Colour	pН	Acidic, Basic or Neutral
1	Vinegar			
2	Tomato Juice			
3	Lemon Juice			
4	Milk			
5	Tap water			
6	Washing soda			

Crystallization

Aim: To obtain pure crystals of copper sulphate from an impure sample.

Apparatus: Pestle and mortar, beaker, evaporating dish, sand bath and filter papers.

Chemicals: Impure sample of copper sulphate.

Theory:

Crystallization is the (natural or artificial) process of formation of solid crystals precipitating from a solution, melt or more rarely deposited directly from a gas. Crystallization is also a chemical solid–liquid separation technique, in which mass transfer of a solute from the liquid solution to a pure solid crystalline phase occurs. In chemical engineering crystallization occurs in a crystallizer. Crystallization is therefore an aspect of precipitation, obtained through a variation of the solubility conditions of the solute in the solvent, as compared to precipitation due to chemical reaction.

Procedure:

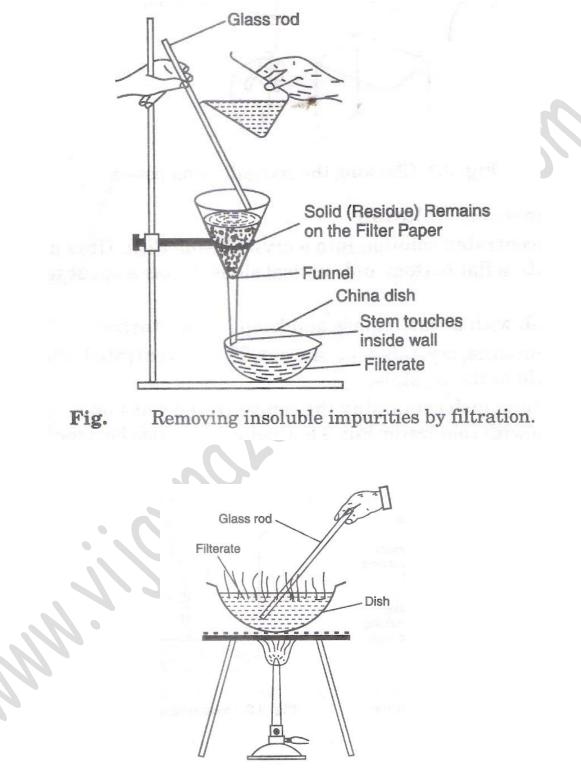
- (i) Grind some of the impure sample of copper sulphate in a pestle and weigh out roughly 5 g. Take 50 ml of distilled water in a beaker and add the weighed sample of impure copper sulphate. Warm gently and stir it with a glass rod. The copper sulphate will dissolve leaving the insoluble impurities in suspension. Filter the solution into an evaporating dish.
- (ii) Heat the solution gently on a sand bath and concentrate it to the saturation point. Test the attainment of the saturation point. If it has been reached, stop heating the solution and keep it aside for crystallization.
- (iii) After some time the crystals will appear. Remove the mother liquor by decantation and wash the crystals to remove the mother liquor sticking to the crystals. Dry the crystals by pressing them between filter papers.

Result :

i. Colour of the crystals: _____

- ii. Shape: _____
- iii. Yield: _____Grams
- iv. Percentage yield: _____%

Diagram:





CHEMICAL EQUILIBRIUM

Aim

Study of the shift in equilibrium in the reaction between $[Co(H2O)_6]^{2+}$ and Cl^- ions, by changing the concentration of any one of these ions.

Theory

In the reaction between $[Co(H2O)_6]^{2+}$ and Cl^- ions, the following displacement reaction takes place.

$$[Co(H_2O)_6]^{2+} + 4Cl^- \rightleftharpoons [CoCl_4]^{2-} + 6H_2O$$

Pink Blue

This reaction is known as ligand displacement reaction and the equilibrium constant, *K*, for this is written as follows:

$$K = \frac{[[CoCl_4]^{2^-}]}{[[Co(H_2O)_6]^{2^+}][Cl^-]^4}$$

Since the reaction occurs in the aqueous medium, it is believed that concentration of H2O is almost constant and is included in the value of *K* itself and is not shown separately in the expression for equilibrium constant.Now if at equilibrium the concentration of either $[Co(H_2O)_6]^{2+}$ ion or Cl⁻ ions is increased, then this would result in an increase in $[CoCl4]^{2-}$ ion concentration thus, maintaining the value of *K* as constant. In other words we can say that equilibrium will shift in the forward direction and will result in a corresponding change in colour.

Chemicals and Material Required

- Conical flask (100 mL) : One
- Beakers (100 mL) : Three
- Burettes : Three
- Test tubes : Six
- Test tube stand : One
- Glass rod : One
- Acetone/alcohol : 60 mL
- Concentrated hydrochloric acid : 30 mL
- Cobalt chloride : 0.6000 g

Procedure

(i) Take 60 mL of acetone in a 100 mL conical flask and dissolve 0.6000 g CoCl2 in it to get a blue solution.

(ii) Take 5 test tubes of same size and mark them as A, B, C, D and E. Add 3.0 mL of cobalt chloride solution in each of the test tubes from 'A' to 'E' respectively. Now add 1.0 mL,

0.8 mL, 0.6 mL, 0.4 mL and 0.2 mL of acetone respectively in these test tubes. Add 0.2 mL, 0.4 mL, 0.6 mL and 0.8 mL of water to test tubes B, C, D and E respectively, so that the total

volume of solution in each of the test tubes is 4.0 mL.

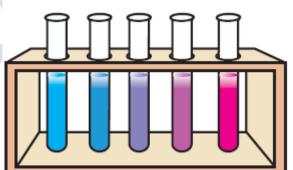
(iii) Note the gradual change in colour of the mixture from blue to pink with an increase in the amount of water. Record your observations in tabular form (Tables 4.3).

(iv) Take 10 mL cobalt chloride solution in acetone prepared above and add 5 mL distilled water to it. A solution of pink colour will be obtained.

(v) Take 1.5 mL of pink solution from step (iv) in five different test tubes labeled as A'B', C', D'and E'. Add 2.0 mL,1.5 mL, 1.0 mL and 0.5 mL of water to the test tubes labeled from A'to D'and 0.5 mL, 1.0 mL, 1.5 mL, 2.0 mL and 2.5 mL

concentrated HCl respectively in the test tubes A'to E'so that total volume of solution in the test tubes is 4 mL.

(vi) Note the gradual change in colour of pink solution to light blue with increasing amounts of hydrochloric acid. Record your observations in tabular form (Tables 4.4).



(SI. No.	Test tube	Volume of acetone added in mL	Volume of CoCl ₂ solution added in mL	Volume of water added in mL	Colour of mixture
	1.	А	1.0	3.0	0.0	
	2.	В	0.8	3.0	0.2	
	3.	С	0.6	3.0	0.4	
	4.	D	0.4	3.0	0.6	
	5.	Е	0.2	3.0	0.8	

Table 4.3 : Shift in equilibrium on adding water



Table 4.4 : Shift in equilibrium on adding Cl[−] ions

Sl. No.	Test tube		Volume of aquo complex solution added in mL	Volume of water added in mL	Colour of mixture
1.	A′	0.5	1.5	2.0	
2.	B′	1.0	1.5	1.5	
3.	C'	1.5	1.5	1.0	
4.	D′	2.0	1.5	0.5	
5.	E'	2.5	1.5	0.0	

Note : • In the first set of experiments concentration of chloro complex is constant and concentration of water is changing. • In the second set concentration of aqua complex is constant and concentration of chloride ions is increasing.

Result:- At equilibrium when the concentration of Cl^- ions is increased, then this results in an increase in $[CoCl_4]^{2-}$ ion concentration thus, maintaining the value of *K* as constant and equilibrium shifts in the forward direction and results in a change in colour from pink to blue. Similarly when the concentration of H₂O is increased, then this results in an increase in $[Co(H_2O)_6]^{2+}$ ion concentration thus, maintaining the value of *K* as constant and equilibrium shifts in the backward direction and results in a change in colour from blue to pink.

Determination of Boiling Point (B.P):

Aim :- To determine the boiling point of an organic compound (liquid)

Apparatus :- Thiele's tube , capillary tube , Stand , thread, Bunsen burner.

Chemicals :- Pure given organic compound, liquid paraffin.

Theory :- The boiling point of a liquid compound is the temperature at which the vapour pressure of the liquid becomes equal to the atmospheric pressure. The presence of impurities raises its B.P. The B.P. of the given organic liquid is generally determined by using Thiele's tube or open oil bath.

Procedure :-

1) Take a small tube (usually sodium fusion tube) and fill it by pure organic liquid whose boiling point is to be determined.

2) Seal a capillary tube at one end and place it in the liquid with the sealed end above the surface of the liquid.

3) Fasten the glass tube to the thermometer using a thread or a rubber band.

4) Immerse partly the thermometer in the paraffin liquid taken in a small beaker or Thiele's tube taking care to see that thread is above the level of the liquid bath i.e. it is not coming in contact with liquid bath.

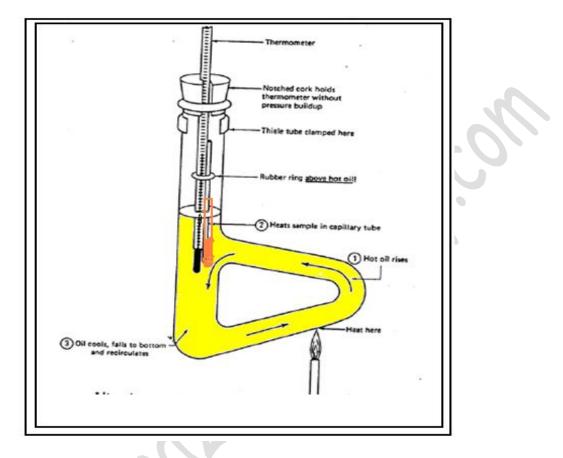
5) Heat the Thiele's tube gently at the lower bent arm and no stirring is required as convection currents maintain uniform temperature. As rapid and continuous stream of air bubbles start coming out from the lower end of the capillary tube, remove the burner but continue stirring note down the temperature at which the air bubble stop and the liquid rushes into the capillary tube. This is the boiling point of the liquid.

Result:- I) The Boiling point of given organic Liquid _____is ____⁰C

2) The Boiling point of given Unknown organic Liquid is ______⁰C

3) The Unknown organic liquid is_____

<u>Diagram</u>



Observation Table

Sr.No	Name of the compound	Boiling point in Degree Celsius	Boiling point in Kelvin
1	Acetic acid	118	
2	Unknown compound		

Boiling point of some common organic compounds

Sr.No	Name of the compound	Boiling point in Degree Celsius	Boiling point in Kelvin
1	Nitrobenzene	210	
2	Benzene	80	
3	Benzaldehyde	179	
4	Acetone	56	

Determination of Melting Point (M.P):

Aim :- To determine the Melting point of an organic compound (Solid)

Apparatus :- Thiele's tube , capillary tube , sodium fusion tube ,Stand ,thread, Bunsen burner.

Chemicals :- Pure given organic compound, liquid paraffin.

Theory :- The Melting point of a compound is the temperature at which the solid state of a compound changes into liquid state. It is defined as the temperature at which solid phase is in equilibrium with the liquid phase at a given temperature. The presence of impurities lowers its M.P.

Procedure :-

1) Take a capillary tube and seal it from one end.

2) Fill the capillary tube with given finely powered organic solid from the non sealed end whose Melting point is to be determined.

3) Fasten this capillary tube to the thermometer using a thread or a rubber band.

4) Immerse partly the thermometer in the paraffin liquid taken in a Thiele's tube taking care to see that thread is above the level of the liquid bath i.e. it is not coming in contact with liquid bath.

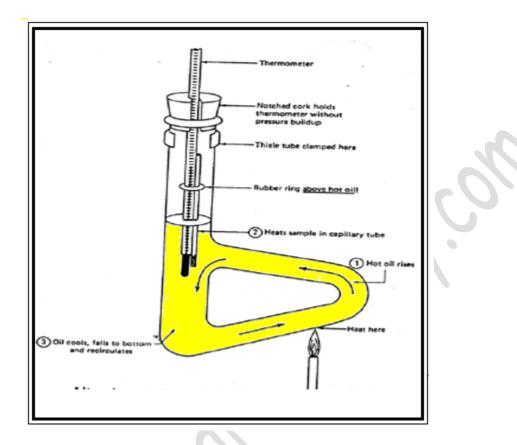
5) Heat the Thiele's tube gently at the lower bent arm and no stirring is required as convection currents maintain uniform temperature. As you see the solid changing in to liquid from the lower end of the capillary tube, that is when you see partly powered compound changed to liquid and still some in the solid state. Note down this temperature, this is the Melting point of the solid.

Result:-	 The Melting point of given organic compound 	is	0C
----------	---	----	----

2) The Melting point of given Unknown organic compound is _____ ⁰C

3) The Unknown organic compound is______

Diagram



Observation Table

 $\mathcal{T}\mathcal{P}$

Sr.No	Name of the compound	Melting point in Degree Celsius	Melting point in Kelvin
1	Naphthalene	80	
2	Unknown		

Melting point of some common organic compounds

Sr.No	Name of the compound	Melting point in Degree Celsius	Melting point in Kelvin
1	Oxalic acid	101	
2	Urea	132	
3	Benzoic acid	121	
4	m-Dinitrobenzene	90	

Analysis of Salts

PRELIMINARY TESTS:

PRELIMINARY TESTS:			
TEST	OBSERVATION	INFERENCE	
DRY TEST OF CATION			
(BASIC RADICALS)			
1 Colour	(a) Plue or Pluish green	Cu ²⁺ or Ni ²⁺ may be	
1. <u>Colour</u>	(a) Blue or Bluish green	present	
	(b) Green	Ni ²⁺ may be present	
	(c) Reddish Brown or	Fe^{3+} may be present	
	Brown		
	(d) Pink	Co ²⁺ may be present	
	(e) Light Pink, Flesh	Mn ²⁺ may be present	
	colour or earthy colour		
	(f) Colourless (white)	$Cu^{2+},Ni^{2+},Fe^{3+},Co^{2+},$	
		Mn^{2+} may be absents.	
		$Pb^{2+}, Al^{3+}, Zn^{2+}, Ca^{2+}, Sr^{2+},$	
		$Ba^{2+}, Mg^{2+}, K^{+}, NH_{4}^{+}$	
2. <u>Heating in a dry test tube</u>		may be present	
Little of compound	(a) Decrepitation	Pb(NO ₃) ₂ ,KCl,Ba(NO ₃) ₂	
heated in a dry	(Crackling sound)	KNO_3 may be present	
heating tube	(b) Coloured residue	$Cu^{2+}, Co^{2+}, Ni^{2+}, Fe^{3+}$	
N.B (1) Observe the	(including black)	salts may be present	
evolution Of gases also			
	(c) White infusible residue	$Ba^{2+}, Sr^{2+}, Ca^{2+}$ may be	
(2) If original compd is		present	
coloured then only a	(d) White sublimate	.: Ammonium halides may	
black residue obtained	(a) Velley, residue when	be present $7e^{2+}$ may be present	
(3)Observe the changes by first slowly heating	(e) Yellow residue when hot which turns white	Zn^{2+} may be present	
followed by strong	when cold		
heating	(f) White compd gives	$: K_2 C_2 O_4$ may be present	
	black residue on strong		
	heating		
	(g) Evolution of gases		
	(1) Colourless gas with	\therefore NH ₄ ⁺ may be present	
	Smell of Ammonia		
	turning red litmus		
	paper blue	C^{1-} C^{2-} C^{1-} C^{2-}	
	(2) Colourless gas turning blue litmus paper red	$::Cl^{-}, SO_{4}^{2-}, CH_{3}COO^{-}$	
	(3) Colourless gas with	may be present $CH_3 COO^-$ may be present	
	having vinegar like	CH3 COO may be present	
	smell		
	1	1	

TEST	OBSERVATION	INFERENCE	
	(4) Colourless gas	$C_2O_4^{2-}$ may be present	
	turning lime water		
	milky		
	(5)Brown funes give	NO_3 , Br may be present	
	out		
	(6)Greenish yellow	C1 ⁻ may be present	
	gas turning starch		
	iodide paper blue		
	(7)Reddish brown	Br ⁻ may be present	
	gas turning starch		
	iodide paper orange		
	yellow (8)Dark violet	I may be present	
	vapours gives out	I may be present	
	turning starch iodide		
	paper blue		
3. Flame test	paper blue		
<i>Clean the Pt.loop/Nichrome</i>			
wire by dipping it in a t.t	(1) Lilac or Violet	K ⁺ may be present	
containing a little conc. HCl	flame	in the present	
and placing it in the blue	(2) Bluish white	Pb ²⁺ may be present	
part of the flame. moisten a	flame		
little compd on the watch	(3) Bluish green	Cu ²⁺ may be present	
glass with conc. HCl. Dip	flame		
the Pt.loop/Nichrome wire	(4) Apple green	Ba ²⁺ may be present	
in this mixture and hold it in	flame		
the blue part of the flame.	(5) Brick red (Dull	Ca ²⁺ may be present	
Observe the flame colour	red) flame	2	
	(6) Crimson red	Sr ²⁺ may be present	
	(dark red) flame		
	(7) No characteristic	$K^{+} Pb^{2+} Cu^{2+} Ba^{2+} Ca^{2+} Sr^{2+}$	
	colour to the	May be absent.	
	flame		
4 Tost for NII +	(1) Gas evolved with	$.: NH_4^+$ may be present	
4. <u>Test for NH_4^+</u> Compound + dil NaOH.	smell of ammonia	INIT ₄ may be present	
Warm	forming dense white		
,, am	fumes with a glass rod		
	dipped in conc. HCl.		
	(2) No smell of ammonia	.: NH4 ⁺ may be absent	
DRY TEST FOR ANION			
(ACIDIC RADICALS)			
5. Action of dil. HCl	(1) Effervescence of a	$: CO_3^{2-}$ may be present	
Compd + dil HCl	gas turning lime water		
(Observe without heating.)	milky		
	(2) No Effervescence	$: CO_3^{2-}$ may be Absent	
		-	

TEST	OBSERVATION	INFERENCE
6. <u>Action of Conc.H₂SO₄</u> Compd + Conc.H ₂ SO ₄	(1) Colourless fumes of HCl given out forming dense white	.: Cl ⁻ may be present
NB: Test for C1 ⁻ in cold. If C1 ⁻ absent, warm and observe	 fumes with a glass rod dipped in NH₄OH. (2)Colourless vapours given out on warming, having the smell of vinegar. (3) Reddish brown or yellowish brown vapour given out on warming the test tube. (4) Brownish fumes obtained above (3) increases on addition of Cu foil. (5) Violet vapours given out on warming the test tube. (6) No characteristic fumes / vapours 	 .: CH₃COO⁻ may be present .: Br⁻,NO₂⁻, NO₃⁻ may be present .: Br⁻ may be absent NO₂⁻, NO₃⁻ may be present .: Γ may be present CΓ, Br⁻, Γ, NO₃⁻, CH₃ COO⁻ may be absent
7. <u>Test for PO₄</u> ³⁻ Compd.+ Conc HNo ₃ (<i>Boil to drive out any</i> <i>coloured fumes if given.</i>) Add excess of ammonium molybdate soln 8. <u>Test for C₂O₄</u> ²⁻ (<u>KMnO₄ Test)</u> Compd. + 2 mldil.H ₂ SO ₄ Boil off any gas evolved, add little more dil.H ₂ SO ₄ and then KMnO ₄ soln dropwise.	 Canary yellow ppt No Canary yellow ppt KMnO₄ Soln is decolourised KMnO₄ Soln is not decolourised 	.: PO_4^{3-} may be present .: PO_4^{3-} absent. .: $C_2O_4^{2-}$ may be present .: $C_2O_4^{2-}$ absent.

NB: If the tests (5),(6),(7) and (8) do not give positive results, the compound may contain SO_4^{2-} anion.

PREPARATION OF SOLUTION (W.E)

Take a 10 ml (approx) of distilled water in a 25 ml beaker, warm and add pinch by pinch the compound, stir till it dissolves, then add more. Solution obtained is (W.E) **Water Extract** (*Use this solution to perform the wet tests for cation and anion*).

1. TEST FOR HALIDES		
$\frac{(\mathbf{Cl}^{*}, \mathbf{Br}^{*}, \mathbf{I}^{*})}{W.E + Ag NO_{3}}$	No ppt or Ppt soluble in dil HNO ₃	Halides absent.
DETECTION OF	Ppt insoluble in dil HNO ₃	Halides (Cl ⁻ , Br ⁻ , I ⁻) are present
HALIDES (Cl', Br', J') Note the colour of the		
appt obtained above in	(a) White ppt soluble in NH ₄ OH	Cl ⁻ is present
	(b) Pale yellow ppt(c) Yellow ppt	Br ⁻ is present I ⁻ is present
$\frac{\textbf{C.T FOR Cl'/Br'/I}^{-}}{(i) W.E + Conc H_2 SO_4 +}$		
MnO ₂ . Warm gently	(a) Faint greenish yellow fumes turning moist blue litmus paper red and finally bleaches it	Cl ⁻ is confirmed
	(b) Reddish brown fumes(c) Violet fumes	Br ⁻ is confirmed I- is confirmed
ii) W.E + dil. H_2SO_4 +CCl ₄ or CHCl ₃ + excess of strong chlorine water. Shake well	(a) Colourless CCl ₄ /CHCl ₃ layer	Cl ⁻ is confirmed
and allow to stand	(b) Reddish yellow CCl ₄ / CHCl ₃ layer	Br ⁻ is confirmed
2. <u>TEST FOR</u> CARBONATE (CO ₃ ²⁻)	(c)Violet CCl ₄ /CHCl ₃ layer	I is confirmed
W. $E + CaCl_2$	White ppt soluble in dil HNO ₃ with effervescence.	ao ² -1
$\frac{\text{C.T for CO}_3^{2-}}{\text{(i) W.E + dil HCl}}$	Effervescence of a gas turning lime water milky.	CO_3^{2-} is present
(ii) W.E + phenolphthalein	Pink colour	CO_3^{2-} is confirmed
3. <u>TEST FOR</u> <u>SULPHATE (SO4²⁻)</u>		CO_3^{2-} is confirmed
$\frac{\text{SOLTHATE}(SO_4)}{\text{W.E} + \text{di.l HNO}_3}$ $+ \text{Ba(NO}_3)_2$	White ppt	
C. T FOR SULPHATE W.E + Pb (CH ₃ COO) ₂	White ppt insoluble in dil. HNO ₃	SO_4^{2-} is present SO_4^{2-} is confirmed

4. <u>TEST FOR NITRATE</u> (<u>NO₃</u>) W.E Conc H ₂ SO ₄ +Cu filings. <i>Warm Carefully</i> .	Brown fumes of NO ₂ and Bluish green soln	NO_3^- is present
$\frac{C.T \ For \ NO_3}{W.E + Conc \ H_2SO_4 \ Cool}$ under tap. then add freshly prepared saturated FeSO ₄ soln Carefully along the sides of the test tube .	Brown ring (of FeSO ₄ .NO) at the junction of the two solutions	NO_3^- is confirmed
5. <u>TEST FOR</u> <u>PHOSPHATE</u> (PO_4^{3-}) W.E + Conc. HNO ₃ boil gently. Add ammonium Molybdate in excess and warm again.	Canary yellow ppt	PO_4^{3-} is present
$\frac{\text{C.T For PO_4}^{3-}}{\text{W.E + NH_4Cl + NH_4OH +}}$ Mg SO ₄ solution 6. <u>TEST FOR</u> <u>ACETATE (CH_3COO^-)</u>	White crystalline ppt of Magnesium ammonium phosphate	PO_4^{3-} is confirmed
$\frac{ACETATE}{CH_{3}COO}$ W.E + neutral FeCl ₃ soln	Reddish coloured ppt or soln	$CH_3 COO^-$ is present
$\frac{\text{C.T for CH}_3 \text{ COO}^{-}}{\text{To the decanted solution from above add dil HCl}}$	Reddish colour disappears and a white ppt is formed in a yellow coloured solution	$CH_3 COO^-$ is confirmed
 (2) ESTER TEST W.E + Conc. H₂SO₄ (2ml) Heat. Add ethyl alcohol (1ml). Shake warm pour this soln into a beaker containing water. 7. TEST FOR OXALATE 	Pleasant fruity smell of ester.	CH ₃ COO ⁻ is confirmed.
$\frac{(C_2O_4^{2-})}{W.E + dil CH_3COOH. Boil to}$ remove CO ₂ . Add CaCl ₂ soln. $\frac{C.T FOR C_2O_4^{2-}}{W.E + dil H_2SO_4 warm. Add 2-3}$ drops of KMnO ₄	White ppt formed that dissolves on adding dil HNO ₃ and warming. Pink colour of KMnO ₄ is decolourised with the evolution of CO ₂ gas.	$C_2O_4^{2-}$ is present. $C_2O_4^{2-}$ is confirmed

<u>WET TEST FOR CATION</u> <u>DETECTION OF CATION OF GROUP ZERO (NH4⁺)</u>

TEST	OBSERVATION	INFERENCE
W.E + 2ml dil NaOH shake	(1) No smell of Ammonia/	.: Group O is absent
well and warm gently	NH ₃ gas not evolved	$.: \mathbf{NH}_4^+$ is absent
	(2)NH ₃ gas evolved recognized by its smell turning most red litmus paper blue or moist Turmeric Paper brown or Evolution of dense white fumes when a glass rod dipped in conc.HCl is held over the mouth of the test tube	.: Group O is Present .: NH4 ⁺ is Present
<u>C.T. for NH4</u> ⁺ 1ml of Nessler's reagent + 2 drops of W.E	Brown ppt or Colouration	.: NH4 ⁺ is confirmed

SEPARATION OF CATIONS INTO GROUPS (I - VI)

TEST	OBSERVATION	INFERENCE
1)W.E + dil. HCl	White ppt	Group I is present
	5	.: Pb ²⁺ is present (<i>Refer table No 2</i>)
	No white ppt	Group I is absent
2)W.E + dil. HCl + warm	Black ppt	\therefore Group II is present
and add H_2S water.		.: Cu ²⁺ is present (<i>Refer Table No 3</i>)
	No black ppt	Group II is absent
3) W.E + Solid NH ₄ Cl +	Coloured ppt	.: Group III is present
NH ₄ OH till alkaline	(white or Brown)	:: Al ³⁺ , Fe ³⁺ , Mn ²⁺ are present (<i>Refer Table No 4</i>)
	No coloured ppt	.: Group III is absent
		•

4) W.E + solid NH_4Cl	Coloured ppt	Group IV is present
+NH ₄ OH till alkaline +	(Black, White, Pink)	Co^{2+} , Ni^{2+} , Zn^{2+} and Mn^{2+}
H_2S gas / H_2S water		are present
		(Refer Table No 5)
	No coloured ppt	.: Group IV is absent
5) W. E + Solid $NH_4Cl +$	White ppt	Group V is present
NH_4OH till alkaline +		$: Ba^{2+}, Sr^{2+}, Ca^{2+}$ are
$(NH_4)_2$ CO ₃ . Warm		present
		(Refer Table No 6)
	No white ppt	.: Group V is absent
		.: Group VI is present
		\therefore K ⁺ ,or Mg ²⁺ may be
		present
		(Refer Table No 7.)

TABLE NO. 2 <u>Analysis of Group I (Pb²⁺)</u> The colour of the group I ppt (PbCl₂) is white $\therefore Pb^{2+}$ is present.

C.T. for Pb²⁺

TEST	OBSERVATION	INFERENCE
1) W.E $+^{1}/_{2}$ ml KI	Yellow ppt of Pb I ₂	.: Pb ²⁺ is confirmed
2) W.E + $\frac{1}{2}$ ml K ₂ CrO ₄	Yellow ppt	.: Pb ²⁺ is confirmed
3) W.E + $\frac{1}{2}$ ml dil H ₂ SO ₄	White ppt of PbSO ₄	.: Pb ²⁺ is confirmed

$\frac{TABLE NO 3}{Analysis of Group II (Cu²⁺)}$ The colour of the group II ppt (CuS) is black .: Cu²⁺ is present.

<u>C.T. for</u> Cu^{2+}		
Test	OBSERVATION	INFERENCE
1 . W.E + KI soln	Brown ppt of CuI ₂	.: Cu ²⁺ is confirmed
2. W.E + dil NaoH	Blue ppt of Cu(OH) ₂ turning black on heating	.: Cu ²⁺ is confirmed

<u>TABLE NO. 4</u> Analysis of Group III (Al³⁺, Fe³⁺, Mn²⁺)

The colour of the ppt of Group III hydroxides is white / Reddish brown

White gelatinous ppt.	Reddish brown or brown or pink ppts	
.: Al ³⁺ is present.	\therefore :Fe ³⁺ or Mn ²⁺ are present.	
-		
C.T. for Al ³⁺ :		
1) W.E + NaOH soln		
dropwise \rightarrow gelatinous	: W.E +K ₄ Fe(CN) ₆ \rightarrow Deep blue ppt / no deep blue ppt.	
1 0		
white ppt soluble in		
excess of NaOH.	Deep blue ppt or	No deep blue ppt
2) W.E +Na ₂ CO ₃ solution	colouration	
\rightarrow white ppt.		
3) W.E + ammonium	Fe ³⁺ is present.	Mn ²⁺ is present
acetate soln. \rightarrow No ppt in	re is present.	ivin is present
cold but on boiling a		
white gelatinous ppt.		
white genuinous ppi.		
Al ³⁺ is confirmed.	C.T for Fe ³⁺	C.T for Mn ²⁺
Al 18 commined.	W.E. +	1) W.E.+ NH_4OH till alkaline
	KSCN/NH ₄ CN	\rightarrow white ppt turning brown on
	-	
	\rightarrow Blood red	heating
	colouration.	2) W.E +5ml of dil HNO ₃ +pinch of
		solid Sodium bismuthate. Shake,
		allow to stand \rightarrow violet colouration.
	Fe ³⁺ is confirmed	Mn ²⁺ is confirmed

Note: With concentrated W.E the Mn^{2+} is precipitated In Group III as $Mn(OH)_2$ (brown or buff ppt) Otherwise Mn^{2+} will be precipitated as MnS (Pinkish ppt) in Group IV. Thus, Mn can analysed either in **Group III** or in **Group IV**.

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<u>Analysis of Group IV (Co²⁺, Ni²⁺, Zn²⁺, Mn²⁺)</u>

The colour of the ppt.of Group IV sulphides is Black / White / Pinkish

Black ppt.		White ppt.	Pinkish ppt.	
: Co^{2+} or Ni ²⁺ are present		2	•	
		Zn^{2+} is present.	: Mn ²⁺ is present.	
W.E + equal volume acetone + NH ₄ SCN \rightarrow Blue colour / no blue colour		 <u>C.T for Zn²⁺:</u> W.E. + W.F. + K₄Fe (CN)₆ → White ppt. 2) W.E.+NaOH soln. dropwise → White ppt. Soluble in excess of NaOH and is reppt. as ZnS on addition of H₂S gas. 	<u>C.T. for Mn²⁺:</u> 1) W.E + NH ₄ OH → White ppt.turning brown on heating. 2) W.E +5ml dil HNO ₃ pinch of sodium bismuthate. Shake and allow to stand → Violet colouration. 3) W.E + PbO ₂ (0.1g) +1/2 ml of dil.(1:1)	
Blue colour	No Blue colour	addition of 1125 gas.	HNO ₃ ; boil and	
Co^{2+} is present.	Ni ²⁺ is present.		allow to settle \rightarrow	
<u>C. T. for Co²⁺</u>	C.T. for Ni ²⁺		Violet colouration.	
1) W.E.+1ml of a-	1) W.E. +			
nitroso β - napthol	NH ₄ Cl +NH ₄ OH			
→Brown ppt.	till alkaline + dimethyl glyoxime.	\therefore Zn ²⁺ is confirmed	.:Mn ^{2+ is} confirmed	
2) W.E.+NaOH	\rightarrow Scarlet red			
\rightarrow blue ppt changing to brown on heating.	ppt.			
	2) W.E. + NH ₄ OH in			
3) W.E.+	excess.			
NH ₄ Cl+NH ₄ OH till	\rightarrow Pale green ppt.			
alkaline	soluble in excess			
$+K_4Fe(CN)_6 \rightarrow$	giving blue soln.			
Red soln.				
Warm →Reddish				
brown ppt.	NI:2+ :-			
$C = 2^{+}$	$.:Ni^{2+}$ is			
.:Co ²⁺ is confirmed	confirmed			
•				

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TABLE NO 6 Analysis of Group V (Ba ²⁺, Sr ²⁺, Ca ²⁺)

The colour of the ppt. of Group V carbonates is white therefore Ba ²⁺/. Sr²⁺ or Ca²⁺ are present W.E +Acetic acid+ K₂CrO₄ \rightarrow yellow ppt / no yellow ppt.

Yellow ppt.	No yellow ppt. Sr^{2+} or Ca^{2+}]	
Ba ²⁺ is present.	. W.E +Acetic acid + NH_4OH till alkaline + solid		
	$(NH_4)_2 SO_{4+} boil \rightarrow white ppt. / No white ppt$		
	White ppt.	No white ppt	
	Sr^{2+} is present	Ca ²⁺ is present	
C.T.for Ba ²⁺ :	<u>.C.T.for</u> Sr ²⁺ :	<u>.C.T. for Ca²⁺</u>	
1) W.E +Acetic acid.+	1) W.E +Acetic acid	1) W.E +Acetic acid.+	
ammonium oxalate soln.	Ammonium oxalate	$CaSO_4 \rightarrow No ppt.$	
\rightarrow White ppt. insoluble in	\rightarrow White ppt. Insolube		
acetic acid.	in acetic acid.	2) W.E +Acetic acid.+	
		$(NH_4)_2C_2O_4 \rightarrow White ppt.$	
2) W.E +Acetic acid. + dil.	2) W.E +Acetic acid.+ dil.	Insoluble in acetic acid.	
$H_2SO_4 \rightarrow White ppt.$	$H_2SO_4 \rightarrow White ppt.$		
3) Flame test \rightarrow Apple green	3) Flame test \rightarrow Crimson	3) Flame test \rightarrow Brick red	
flame.	red flame.	Flame.	
.:Ba ²⁺ is confirmed.	a^{2+1}		
	.:Sr ²⁺ is confirmed.	.:Ca ²⁺ is confirmed.	

TABLE NO 7

Analyses of Group VI (Mg^{2+,} K⁺)

TEST FOR K^+	TEST FOR Mg ²⁺
W.E $+ 1$ -2ml of fresh and clear solution	W.E add NH ₄ Cl and NH ₄ OH till alkaline
of sodium cobaltinitrite. \rightarrow	(in slight excess) and then add ammonium
	phosphate solution. Scratch the inner side
Yellow ppt	of the test tube with a glass $rod \rightarrow$
	1. white crystalline ppt .: Mg^{2+} is
\therefore K ⁺ is present.	present
	2. No white ppt .: Mg^{2+} is Absent
C. T for K ⁺	C.T for Mg ²⁺
(1) W.E + picric acid \rightarrow yellow ppt.	W.E + NaOH solution
\therefore K ⁺ is confirmed	\rightarrow White ppt soluble in NH ₄ Cl
	solution
Note:-If K^+ is absent test for Mg^{2+}	
	.: Mg ²⁺ is confirmed

Result:-

- a. Cation_____
- b. Anion_____
- c. Formula_____
- d. Name of the Compound_____

Instructions:

1. Always be in time for your practical's.

2. Always corne prepared for the experiment. This will help in understanding the experiment better.

3. Always listen to the teacher's instructions carefully and note down the important points and precautions to be followed.

4. Do only the experiments assigned, unallotted experiments should not be done.

5. Do your experiment honestly without caring for the final result. Record the observations on a rough note-book instead of writing on pieces of paper.

6. Be economical with the reagents. Only small quantities of the reagent are to be used.

7. Handle the glass apparatus very carefully. In case of any breakage, report it to your teacher at once.

8. Dispose of all waste liquids in the sink and allow the water to run for sometime by opening the water tap.

9. Keep your work place clean. If an acid or other corrosive chemical is spilled, wash it off with water.

10. In case of any injury or accident or breakage of the apparatus, report it to the teacher immediately.

11. Wash your hands with soap after the experiment.

Precautions:

1. Do not touch any chemical with the hand as some of them may be corrosive.

2. Never taste a chemical. It may be poisonous.

3. Do not place the chemical on the palm of your hand.

- 4. Do not keep the reagent bottles open.
- 5. Do not roam here and there in the laboratory uselessly.
- 6. Do not put any object into the reagent bottle.
- 7. Do not bring inflammable liquids such as alcohol or ether near the flame.

8. Do not take the reagent from the shelf to your work place.

9. Do not disturb the arrangement of the reagents placed on the shelf.

10. Do not use cracked glass apparatus such as beakers for heating purposes.

11. Do not keep the water tap running when not required.

12. Do not throw solid waste materials like pieces of filter paper, broken test-tubes, etc in the sink. Throw them in the waste box only.

13. Do not heat beakers or a china dish directly on a flame. Always make use of wire gauze.

Always wear: 1.Safety gloves 2.Safety glasses 3.Gas masks and 4.Lab coat (Apron) in the Lab