LAB MANUAL

OF

Engineering Chemistry Practical

1st/2nd semester of all Engineering Branches

Prepared by

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COURSE: Engineering chemistry lab

Programme: Electrical Engineering

Course code: Pr.2b

Periods/week: 4

Course pre-requisites: Basic concepts of chemistry

Course Objectives:

To develop students understanding through laboratory activities

COURSE OUTCOMES:

After the completion of the course the student will be able to:

CO-1: Understand the physical and chemical properties of carbon dioxide and ammonia gas.

CO-2: Understand the method of crystallisation.

C0-3: Learn and apply basic techniques used in chemistry laboratory for volumetric analysis

CO-4: Identify the constituents of a salt by qualitative analysis.

SYLLABUS

EXPERIMENT NO.	CHAPTER
1	Preparation and Study of Properties of Carbon Dioxide Gas
2	Preparation and Study of Properties of Ammonia Gas
3	Crystallization of Copper Sulphate from Carbonate Powder
4	SIMPLE ACID BASE TITRATION (i) ACIDIMETRY (ii) ALKALIMETRY
5	TEST FOR ACID RADICALS (KNOWN) (i) CARBONATE (ii) SULPHIDE (iii) CHLORIDE (iv) NITRATE (v) SULPHATE
6	TEST FOR BASIC RADICALS (KNOWN) (i) AMMONIUM (ii) ZINC (iii) MAGNESIUM (iv) ALUMINIUM (v) CALCIUM (vi) SODIUM (vii) POTASSIUM
7	TEST FOR UNKNOWN ACID RADICALS.
8	TEST FOR UNKNOWN BASIC RADICALS.
9	TEST FOR UNKNOWN SALT (COMPOSED OF ONE BASIC AND ONE ACID RADICALS).

EXPERIMENT NO-01

AIM OF THE EXPERIMENT:

Preparation and study of physical and chemical properties of carbon dioxide gas.

APPARATUS REQUIRED:

1. Woulf'sbottle.
2. Thistlefunnel.
3. Deliverytube.
4. Rubbercork.
5. Gas jar withlid.
6. Few testtubes.

CHEMICALS REQUIRED:

1. Marblechips ($CaCO_3$).
2. Dil.Hydrochloricacid (HCl).
3. Litmuspaper.
4. Magnesiumribbon.
5. Limewater.
6. Phenolphthaleinsolution.

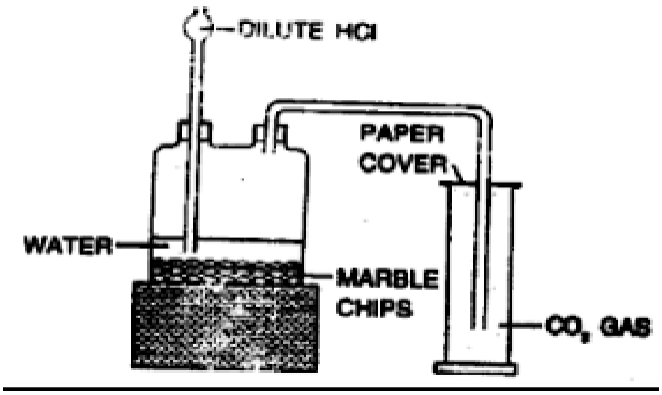
THEORY:

In laboratory carbon dioxide gas is prepared by the action of dilute hydrochloric acid (HCl) upon marble chips ($CaCO_3$) in a woulf's bottle. It is collected by upward displacement of air. Carbon dioxide is heaviour in nature.

CHEMICAL EQUATIONS:

$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2$

LABORATORY DIAGRAM:



PROCEDURE:

1. Take a woulf's bottle fitted with rubber cork, thistle funnel and delivery tube. Examine thatitis perfectly airtight. In case of air leakage, use melted paraffin wax or grease.

2. Introduce few small marble chips into the woulf's bottle by opening one ofitsmouths.

3. Nowpoursomewaterintothewoulf'sbottlethroughthethistlefunnelsoastocover the marble chips.

4. Insert the thistle funnel more into the woulf's bottle such that its extremeendremainsinside the water.

5. Now add little quantity of the dil. Hydrochloric acid through the thistle funnel. Do not add excess amount of acid at a time to exhaust the marble chips before the experiment iscompleted.

6. Then collect the carbon dioxide gas in the gas jar by upward displacement of air. Test the collected gas in the jar by showing a burning splinter at the mouth of gasjar.

7. Study the properties of carbon dioxide gas by collecting the gas in different test tubes.

OBSERVATION:

PHYSICAL PROPERTIES

SL.	EXPERIMENT	OBSERVATION	INFERENCE
1.	Observe the colour ofthe gas		
2.	Observe the odour of the gas		
3.	Enter a glowingsplinter into a test tube full of CO_2gas.		
4.	Invert the test tube full of CO_2 gas over another empty test tube containing air. Then add little lime to the testtube containing air initially.		

5.	Collect the gas in a test tube half-filled with water. Shake the test tube vigorously by putting the thumb at its mouth and remove the thumb and observe the level/volume of waterin the test tube.		

CHEMICAL PROPERTIES

SL NO	EXPERIMENT	OBSERVATION	INFERENCE
1	A piece of moist blue litmus paper is shown to the gas.		
2.	Pass the CO_2 gas through 2-3 ml of dilute solutions of sodium hydroxide (NaOH) containing one dropof phenolphthaleinsolution.		
3.	a) Pass the gas throughlime water. b) Pass the gas in excess. **c)** Boil thesolution**.**		
4.	Introduce a burning magnesium ribbon into a test tube /gas jar containingcarbondioxide gas.		

SAFTY AND PRECAUTION:

1. The fittings should beairtight.

2. The end of thistle funnel must be remaining deep inside thesolution.

3. The shorter end of the delivery tube should remain above the surface of the solutionin

the woulf's bottle.

4. The longer end of the delivery tube must reach the bottom of the gasjar.

5. Addition of excess of dil. hydrochloric acid should beavoided.

6. The gas should be collected after removing air from theapparatus.

ASSIGNMENT QUESTIONS:

(1) What are the apparatus required for this experiment?
(2) Write the chemical formula of marble chip.
(3) Can we use $CaCO_3$ powder for preparation of CO_2 gas?
(4) Why marble chips are used instead of $CaCO_3$ powder?
(5) Write the chemicals used for preparation of CO_2 gas.
(6) How can you prepare dilute HCl?
(7) Hydrochloric acid is a strong or weak acid? Give reason.
(8) Explain the acidic nature of CO_2 gas?
(9) What happens when CO_2 gas is passed through alkaline phenolphthalein solution?
10. What happens when moist blue and red litmus papers are shown to CO_2 gas? How CO_2 gas is collected?
11. What are the apparatus required for preparation of CO_2 gas?
12. How can you prepare CO_2 gas in laboratory?
13. Why moist blue litmus paper turns red on exposure to CO_2 gas?
14. Write two methods of preparation of CO_2 gas.
15. What happens when a burning match stick is introduced into a jar containing CO_2 gas?
16 .What happens when methyl orange indicator is added to aqueous solution of CO_2 gas?
17. What happens when CO_2 gas is passed through lime water first in less amount and then in excess?
18. Write the reactions involved between CO_2 gas and lime water.
19. What is the formula of lime water?
20. Why lime water turns milky when less amount of CO_2 gas is passed through it? Give Equation.
21. Why milky colour disappears on passage of excess CO_2 gas through lime water? Give Equation.
22. What happens when colourless $Ca(HCO_3)_2$ solution will be warmed strongly? Give Equation.
23. What happens when a burning magnesium is introduced into gas jar containing CO_2 gas? Give Equation.
24. Write two uses of CO_2 gas.
25. What is dry ice?
26. How can you test that CO_2 gas is heavier than air?
27. Can sulphuric acid (H_2SO_4) be used in place of HCl for preparation of CO_2 gas?
28. What type of bonding is present in CO_2 ?
29. Why smaller pieces of marble chips are required?
30. Why the thistle funnel is inserted deep to the bottom of the Woulfe's bottle?
31. How can you test the solubility of CO_2 gas?

EXPERIMENT NO-02

AIM OF THE EXPERIMENT:

Preparation and study of properties of NH_3 gas.

APPARATUS REQUIRED:

1.Hard glass test tube
2.Delivery tube
3.Gas jar
4.Card cover
5.Glass jar containing CaO (quick lime)
6.Bunsen burner
7.Rubber cork
8.Clamp stand

CHEMICALS REQUIRED:

1. Solid Ammonium Chloride, NH_4Cl

2. Anhydrous Calcium Hydroxide, $Ca(OH)_2$ or Calcium Oxide (CaO)

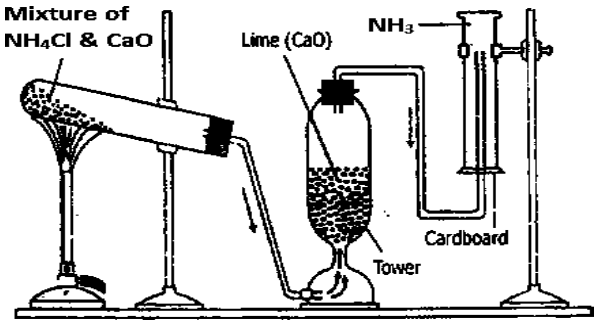
THEORY:

Ammonia gas is prepared in laboratory by heating the mixture of ammonium Chloride (NH_4Cl) & Calcium Hydroxide, $Ca(OH)_2$ paste in 1:3 ratio by weight. The reaction proceeds as:

$$2NH_4Cl + (OH)_2 \rightarrow 2NH_3\uparrow + CaCl_2 + 2H_2O$$

The gas so formed is collected in the gas jar by downward displacement of air because ammonia gas is lighter than air.The gas cannot be collected under water because it is highly soluble in water. Ammonia gas is dried by passing it through the glass bottle containing CaO.

DIAGRAM:



Laboratory preparation of Ammonia Gas.

PROCEDURE:

1. Take a hard glass test tube with rubber cork and deliverytube.

2. Mix 1:3 ratio of ammonium chloride and calcium hydroxide and place the mixture into the testtube.

3. Tilt the test tube at 30 degree angle and clamp it to thestand.

4. Attach the rubber cork to the test tube along with delivery tube connected at one end of the test tube and the other end connected to the glass jar containing CaO. The quicklime is present so as to absorb moisture present in the ammonia gas.

5. Make sure that all the connections are airtight to prevent leakage of ammonia gas.

6. Now carefully and gently heat the mixture in the testtube.

7. Then collect the ammonia gas in the gas jar by downward displacement ofair.

OBSERVATION:

PHYSICAL PROPERTIES

SL.	EXPERIMENT	OBSERVATION	INFERENCE
1	Color of the gas		
2	Odour of the gas		
3	Collect the gas in a test tube half-filled with water. Shake the test tube vigorously by putting the thumb at its mouth and remove the thumb and observe the volume of water in the test tube.		

CHEMICAL PROPERTIES

SL.	EXPERIMENT	OBSERVATION	INFERENCE
1	Show a piece of moist red litmus paper to the gas.		

2	Pass the gas into the test tube containing copper sulphate solution for short timeat first and then in excess.		
3	Pass the gas into the test tube containing ferric chloride solution.		
4	Pass the ammonia gas into the test tube containing Nessler's reagent.		

CONCLUSION:

Ammonia gas is prepared at laboratory by using ammonium chloride (NH_4Cl)&Calcium hydroxide ($Ca(OH)_2$). Ammonia gas is basic in nature. It is highly soluble in water.

SAFETY MEASURES:

1. The apparatus must be airtight.
2. The hard glass test tube should be fixed in inclined position towards its mouth in order to prevent crack in it.
3. Heat should be provideduniformly.
4. The gas jar should bedried.

ASSIGNMENT QUESTIONS:

(1) Write the principle of preparation of ammonia gas in laboratory?
(2) What is the principle of collection of ammonia gas?
(3) Write the apparatus required for preparation of ammonia gas?
(4) What are the chemicals required for ammonia gas?
(5) While clamping the hard glass test tube, its mouth is present slightly downward. Why?
(6) Write the physical properties of ammonia gas?
(7) What is the odour of ammonia gas?
(8) What happens when a glass rod dipped in conc HCl is shown to the ammonia gas?
(9) What happens when a test tube filled with ammonia gas is inverted into a trough of water?
(10) Explain a test to show that ammonia is lighter than air?.
(11) Which compound is required to dry ammonia gas?
(12) Why conc. Sulphuric acid is not used to dry ammonia gas ?
(13) Write two tests to show that ammonia is alkaline in nature?
(14) What happens when ammonia gas is passed through Nessler's reagent? Give equation ?
(15) What happens when ammonia gas is passed through ferric chloride solution? Give equation?
(16) Ammonia gas has rotten egg / pungent/ irritating/ sweet odour.
(17) Ammonia is sparingly / insoluble / highly soluble in water?
(18) What happens when ammonia gas is passed through copper sulphate solution in small quantities?
(19) What happens when ammonia gas is passed through copper sulphate solution in excess?
(20) Ammonia gas turns blue litmus to red / red litmus to blue?
(21) Write the uses of ammonia gas?
(22) What happens when ammonia gas is passed through phenolphthalein solution?
(23) What is the combustibility property of ammonia?
(24) Can NaOH or KOH be used in place of CaO or $Ca(OH)_2$ for preparation of ammonia?
(25) What is that compound (brown precipitate) which forms when ammonia gas is passed through nessler's reagent?

EXPERIMENT NO- 03

AIM OF THE EXPERIMENT:

Crystallization of copper sulphate from copper carbonate.

APPARATUS REQUIRED:

1.Beaker

2.Funnel

3.Glass rod

4.Porcelain basin

5.Tripod stand

6.Wire gauze

7.Bunsen burner

8.Filter paper

9.Filter stand

CHEMICALS REQUIRED:

1. Copper carbonate($CuCO_3$)
2. Dilute sulphuric acid(H_2SO_4)

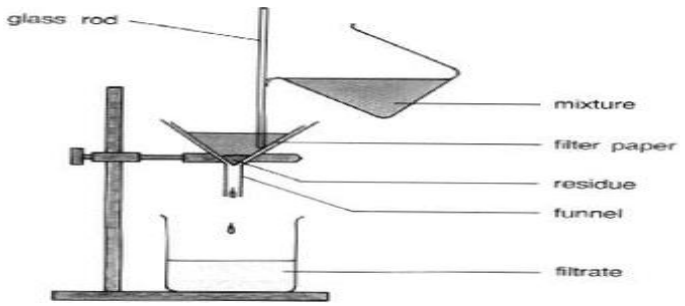
THEORY:

Copper carbonate reacts with dilute sulphuric acid to form soluble copper sulphate with evolution of carbon dioxide gas. The resulting solution is concentrated by evaporation till the point of crystallization is reached and then cooled to get crystals of copper sulphate pentahydrate ($CuSO_4.5H_2O$) called blue vitriol.

$$CuCO_3 + H_2SO_4 \rightarrow CuSO_4 + CO_2\uparrow + H_2O$$

$$CuSO_4 + 5H_2O \rightarrow \quad CuSO_4.5H_2O$$

(Blue vitriol)



Filtration

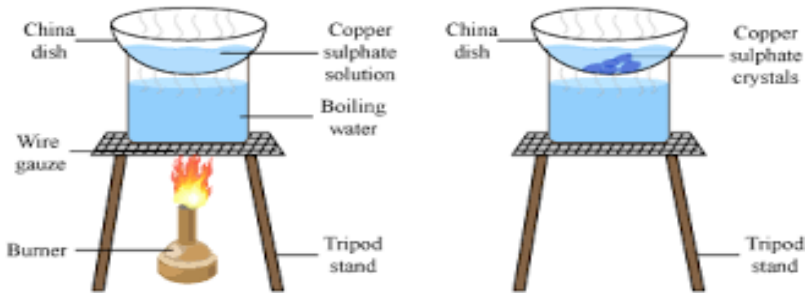


Fig 3.2 Crysta n of copper sulphate .

Procedure :- The preparation of Copper Sulphate crystals from Copper Carbonate involves following steps.

(A) **Preparation of Saturated solution** :-

- Take about 20 ml of dilute Sulphuric acid in a beaker.
- Add supplied Copper Carbonate ($CuCO_3$) power gradually to this acid in small quantities with constant stirring.
- Continue addition of the of the powder till a small quantity of Copper Carbonate ($CuCO_3$) is left behind.
- Heat the resulting solution slightly to expel the dissolved CO_2 gas.
- Take the filter paper and four fold it.
- Prepare a cone of filter paper by taking three folds in one side and one fold in the other side.
- Take a funnel and insert the cone made by the filter paper in to it.
- Filter the solution from beaker to the porcelain basin .
- The solution must be transfered from the beaker to the filter paper cone slowly with the help of a glass rod.
- Wash the insoluble component present in the cone with the distilled water so as to make it free from soluble component.

(B) **Concentrating the Filtrate** :-

- Evaporate the filtrate in the porcelain basin with constant stirring.
- Continue the process of evaporation till a drop of the liquid solution forms crystals on the tip of glass rod when blown on it. This state is termed as **Crystallisation point**.

(C) **Crystallisation** :-

- Cool the hot solution (after reaching crystallisation point) slowly in air to start the process of crystallisation.
- If required, keep the hot porcelain basin containing the solution over a beaker full of water for quicker cooling.

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(D) **Drying and Crystals** :-

- Decant off the saturated mother liquor present over the crystals after the crystallisation is over.
- Transfer the deep blue crystals present in the porcelain basin to a filter paper and spread to dry.

Precautions :-

- Minimum amount of dilute sulphuric acid (H_2SO_4) should be used to prepare the solution .
- The solution should be slightly acidic , other wise the salt may get hrdrolysed
- The solution should not be heated beyond crystallisation point.
- The concentration of solution must be carried with constant stirring
- Crystals should never be dried by heating.

RESULT:

Color : .....

Shape:.....

Yield: .....gm

ASSIGNMENT QUESTIONS:

1. Define crystallisation.
2. Define solubility.
3. Define filtration.
4. Why the solution is not concentrated or heated to dryness during crystallisation?
5. What is decantation?
6. Decantation and filtration which is a better process and why?
7. Why the saturated solution be cooled slowly?
8. What is blue vitriol?
9. What are hydrates?
10. What are anhydrous salts?
11. What is seeding ?
12. Aqueous solution of Copper Sulphate solution acidic or basic or neutral?
13. What is efflorescence?
14. What is the colour and structure of anhydrous Copper Sulphate ?
15. Why excess H_2SO_4 acid is not used for dissolving $CuCO_3$ powder ?
16. What is crystallisation point ?
17. Define mother liquor .
18. Write the reaction between anhydrous $CuCO_3$ and dilute H_2SO_4 .
19. Why the $CuSO_4$ solution be prepared slightly acidic ?
20. Can CuO be used instead of $CuCO_3$ powder for preparation of blue vitriol? If yes, then write the reaction.
21. Write two uses of $CuSO_4.5H_2O$?
22. Mention two other salts which can be prepared by this method .
23. Why a glass rod is used to transfer the solution from beaker to the filter paper cone in the funnel?
24. How can one check the crystallisation point ?
25. How a filter paper cone be prepared?

EXPERIMENT NO- 4(i)

AIM OF THE EXPERIMENT:

Acidimetry: To determine the strength of unknown acid using standard alkali..

APPARATUSREQUIRED:

1.Burette (50 ml.)

2.Burette Stand with clamp

3.Pipette (10 ml.)

4.Conical flask (100ml.)

5.Measuring flask (250ml.)

6.Glazed porcelain

CHEMICALS REQUIRED:

1.Acid Solution (Unknown Strength)

2.N/10 Alkali Solution (Known Strength)

3.Indicator: Methyl Orange

THEORY:

A known volume of standard alkali solution is titrated against the supplied acid solution of unknown strength in the presence of methyl orange indicator till the colour just changes from pale yellow/ straw yellow to light pink. The volume of the acid required for neutralization is determined. Knowing the volume of both the solutions and the strength of alkali, the strength of acid solution can be calculated by using the principle of equivalency.

$$\mathbf{N_a V_a = N_b V_b}$$

Where,

N_a=Normality of the acid solution.

V_a=Volume of acid solution.

N_b=Normality of alkali solution.

V_b=Volume of alkali solution.

PROCEDURE:

1. Wash the burette, pipette and conical flask thrice with tap water and then rinse with distilled water.

2. Rinse the burette thrice with a few ml. of the given acid solution and reject the washings.

3. Fill the burette with the given acid solution to a convenient level without air bubbles. 4.Remove air bubble (if any) present in it.

5. Rinse the pipette with the alkali solution thrice and reject the washing.

6. Pipette out 10 ml. of alkali solution is into the conical flask.

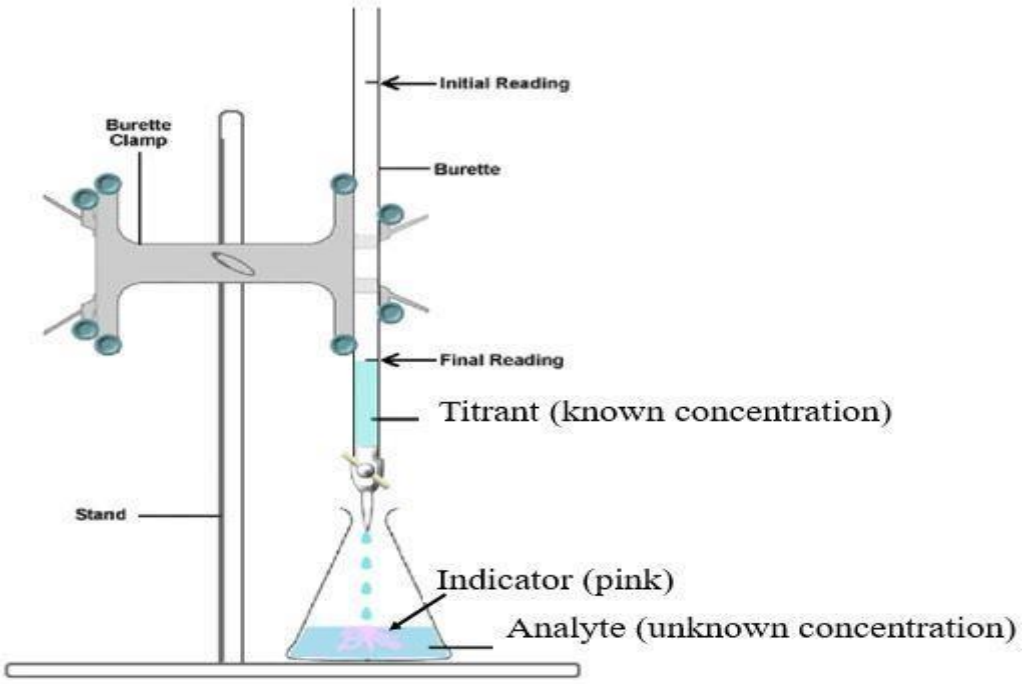
7. After transferring the acid solution, touch the tip of the pipette to the inner side of the conical flask thrice. Wash the inner sides of the conical flask with a little distilled water.

8. Add one drop of methyl orange indicator to it. Keep the conical flask over a white glazed porcelain tile under the burette.

9. Note the initial burette reading avoiding parallax error.

10. Carry out titration by running alkali from the burette with constant stirring till the colour of the solution just changes from colorless to light pink.

11. Note the final burette reading. Repeat the titration till three concordant values are obtained.



- **Titration**

OBSERVATION:

No of Observations	Volume of alkali (ml.)	Initial burette reading (ml.)	Final burette reading (ml.)	Difference (ml.)	Concordant

CALCULATION:

We know that $\mathbf{N_aV_a=N_bV_b}$

Where,

N_a=Normality of acid solution

V_a=Volume of acid solution consumed

N_b=Normality of alkali solution

V_b=Volume of alkali solution

$N_a=N_bV_b/V_a$= --------- N/10

CONCLUSION:

From the above titration the strength of unknown acid solution is found to be--------.

PRECAUTIONS:

1. Care should be taken while handling the acid and base.
2. Always rinse the burette and the pipette with the solution which is to be taken in them.
3. Remove the air gap if any, from the burette before titration.
4. Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
5. Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
6. Never use burette and pipette with a broken nozzle.
7. Never suck a strong acid or an alkali with the pipette, use pipette bulb.
8. Always keep the lower end of the pipette dipped in the liquid while sucking the liquid

ASSIGNMENT QUESTIONS:

(1) What do you mean by volumetric analysis?
(2) What is titration?
(3) Define titrant and titrate?
(4) What are acidimetry and alkalimetry ?
(5) Define a standard solution?
(6) What do you mean by concentration / strength of solutions?
(7) Define an indicator?
(8) Name few indicators which are used during acid – base titration?
(9) Define gram equivalent weight and gram equivalent?
(10) What is molality and normality?
(11) 10 grams of caustic soda is how much gram equivalents?
(12) What is the principle of titration?
(13) Why rinsing is necessary?
(14) Why conical flask is not rinsed with acid or alkali?
(15) What is an anti parallax card?
(16) Why one should not hold the pipette from its bulb?
(17) Why the last drop of the solution be not blown from its bulb?
(18) Why one or two drops of indicator should always be used?
(19) Define the end point or neutralisation point?
(20) How can you detect the neutralisation point?
(21) Why mostly decinormal(N/10) solutions are used rather than normal(N) or centinormal(N/100) solutions?
(22) Which solutions are required to rinse the burette and pipette?
(23) To prepare 2 litres of solution, how much amount of sodium carbonate is required?
(24) Why presence of air bubbles is not preferred in the burette while filling the acid solution?
(25) Why stirring of conical flask and slow addition of acid is required?
(26) What are the colours of methyl orange and phenolphthalein solutions in acidic, alkali and neutral mediums?
(27) How the strength of the solution can be determined?

EXPERIMENT NO- 4(ii)

AIM OF THE EXPERIMENT:

Alkalimetry: To determine the strength of unknown alkali using standard acid..

APPARATUSREQUIRED:

1.Burette (50 ml.)

2.Burette Stand with clamp

3.Pipette (10 ml.)

4.Conical flask (100ml.)

5.Measuring flask (250ml.)

6.Glazed porcelain

CHEMICALS REQUIRED:

1. N/10 Acid Solution (known Strength)

2. Alkali Solution (unknown Strength)

3.Indicator: Methyl Orange

THEORY:

A known volume of standard alkali solution is titrated against the supplied acid solution of unknown strength in the presence of methyl orange indicator till the colour just changes from pale yellow/ straw yellow to light pink. The volume of the acid required for neutralization is determined. Knowing the volume of both the solutions and the strength of alkali, the strength of acid solution can be calculated by using the principle of equivalency.

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Where,

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N_b=Normality of alkali solution.

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PROCEDURE:

1. Wash the burette, pipette and conical flask thrice with tap water and then rinse with distilled water.

2. Rinse the burette thrice with a few ml. of the given acid solution and reject the washings.

3. Fill the burette with the given acid solution to a convenient level without air bubbles. 4.Remove air bubble (if any) present in it.

5. Rinse the pipette with the alkali solution thrice and reject the washing.

6. Pipette out 10 ml. of alkali solution is into the conical flask.

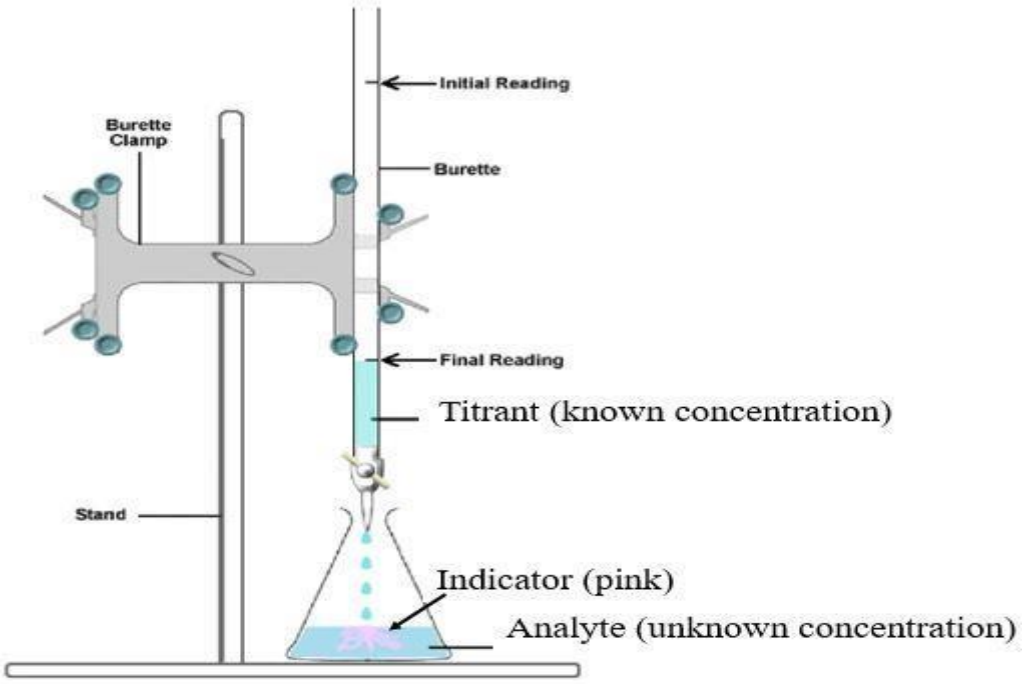
7. After transferring the acid solution, touch the tip of the pipette to the inner side of the conical flask thrice. Wash the inner sides of the conical flask with a little distilled water.

8. Add one drop of methyl orange indicator to it. Keep the conical flask over a white glazed porcelain tile under the burette.

9. Note the initial burette reading avoiding parallax error.

10. Carry out titration by running alkali from the burette with constant stirring till the colour of the solution just changes from colorless to light pink.

11. Note the final burette reading. Repeat the titration till three concordant values are obtained.



Titration

OBSERVATION:

No of Observations	Volume of alkali (ml.)	Initial burette reading (ml.)	Final burette reading (ml.)	Difference (ml.)	Concordant

CALCULATION:

We know that $\mathbf{N_aV_a=N_bV_b}$

Where,

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N_b=Normality of alkali solution

V_b=Volume of alkali solution

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CONCLUSION:

From the above titration the strength of unknown acid solution is found to be--------.

PRECAUTIONS:

1. Care should be taken while handling the acid and base.
2. Always rinse the burette and the pipette with the solution which is to be taken in them.
3. Remove the air gap if any, from the burette before titration.
4. Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
5. Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
6. Never use burette and pipette with a broken nozzle.
7. Never suck a strong acid or an alkali with the pipette, use pipette bulb.
8. Always keep the lower end of the pipette dipped in the liquid while sucking the liquid

ASSIGNMENT QUESTIONS:

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(7) Define an indicator?
(8) Name few indicators which are used during acid – base titration?
(9) Define gram equivalent weight and gram equivalent?
(10) What is molality and normality?
(11) 10 grams of caustic soda is how much gram equivalents?
(12) What is the principle of titration?
(13) Why rinsing is necessary?
(14) Why conical flask is not rinsed with acid or alkali?
(15) What is an anti parallax card?
(16) Why one should not hold the pipette from its bulb?
(17) Why the last drop of the solution be not blown from its bulb?
(18) Why one or two drops of indicator should always be used?
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(23) To prepare 2 litres of solution, how much amount of sodium carbonate is required?
(24) Why presence of air bubbles is not preferred in the burette while filling the acid solution?
(25) Why stirring of conical flask and slow addition of acid is required?
(26) What are the colours of methyl orange and phenolphthalein solutions in acidic, alkali and neutral mediums?
(27) How the strength of the solution can be determined?

EXPERIMENT NO-5

AIM OF THE EXPERIMENT:

Test for carbonate, sulphide, chloride, nitrate, sulphate radicals (Known)

APPARATUS REQUIRED:

1. Test tube holder
2. Watch Glass
3. Test tubes
4. Bunsen Burner

CHEMICALS REQUIRED:

1.Given salts

2.Various Reagent

3. Litmus paper

THEORY AND PROCEDURE:

TEST FOR ACID RADICALS:

Test for Carbonate (CO_3^{2-}):

EXPERIMENT	OBSERVATION	INFERENCE
1.Take 2 ml of dil. HCl or dil. H_2SO_4, in a clean test tube. Warm it and add a little of the salt into it.	1. 1.Effervescences takes place with the evolution of a colourless, odourless gas.	1.It may be CO_2 from CO_3^{2-}
2.Warm the above reaction mixture to get more gas and pass the gas slowly through limewater.	2. First lime water turns milky and with excess of the gas milkiness disappears.	2. $CO_3^{2-}$is confirmed.

Explanation for Carbonate Test:

1. $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$

$$Na_2CO_3 + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + CO_2$$

2. Milkyness is due to the formation of $CaCO_3$, and with excess of the gas milkyness disappears due to the formation of water soluble $Ca(HCO_3)_2$

$$Ca(OH)_2 + CO_2 \rightarrow \underset{\text{White ppt.}}{CaCO_3\downarrow} + H_2O$$

$$CaCO_3 + H_2O + CO_2 \rightarrow \underset{\text{Soluble}}{Ca(HCO_3)_2}$$

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

EXPERIMENT	OBSERVATION	INFERENCE
1.Take 2 ml of dil. HCl or dil. H_2SO_4, in a clean test tube. Warm it and add a little of the salt into it.	1. Effervescence takes place with the evolution of a colourless gas with rotten egg smell.	1. It may be H_2S from S^{2-}
2.Warm the above reaction mixture and show a filter paper soaked with lead acetate to the evolved gas.	2. The filter paper turns black	2. $S^{2-}$is confirmed.

Explanation for Sulphide Test:

1.

$$Na_2S + 2HCl \rightarrow 2NaCl + H_2S$$

$$Na_2S + H_2SO_4 \rightarrow Na_2SO_4 + H_2S$$

2. The black colour is due to the formation of PbS

$$(CH_3COO)_2Pb + H_2S \rightarrow 2CH_3COOH + \underset{\text{Black}}{PbS\downarrow}$$

Test for Chloride (Cl^-):

EXPERIMENT	OBSERVATION	INFERENCE
1.Take a pinch of salt in a clean and dry test tube and add 2 to 3 drops of conc. $H_2SO_4$to it.	1. Effervescence takes place with the evolution of a colourless gas which fumes in moist air.	1. It may be HCl from Cl^-
2.Warm the above reaction mixture and show a glass rod dipped in conc. NH_4OH to the	2. Dense white fumes are produced and white solid deposited on the tip of the glass	2. It is due to the formation of NH_4Cl Cl^-may be present.

mouth of test tube. 3. Take a pinch of salt in a clean and dry test tube add a little MnO_2&2 to 3 drops of conc. $H_2SO_4$to it and heat the reaction mixture. 4.Take 1-2 ml of the supplied salt solution. Acidify it with 1-2 ml of dilute HNO_3 and add few drops of $AgNO_3$ solution to it. 5.Wash the above of precipitate with distilled water and divided into two parts. Part I- Add dil $HNO_3$and shake well. Part II- Add dil NH_4OH and shake well.	rod. 3.Greenish yellow gas is called which turns filter paper soaked in starch iodide solution blue. 4.A curdy white precipitate is formed 5. Part I- The precipitate does not dissolve Part II- The precipitate does dissolve.	 3.Chlorine gas comes out from chloride which liberates iodine from iodide 4. It is due to the formation of AgCl Cl^-may be present. 5. AgCl is not soluble in dil HNO_3 AgCl is soluble in dil NH_4OH due to the formation of silver diamino complex. Cl^-may be present.

Explanation for Chloride Test:

1. $NaCl + H_2SO_4 \rightarrow NaHSO_4 + HCl \uparrow$ (Colorless gas)

$NaCl + NaHSO_4 \rightarrow Na_2SO_4 + HCl \uparrow$

2. $NH_4OH + HCl \rightarrow NH_4Cl + H_2O$
White fumes

3. $2NaCl + MnO_2 + 2H_2SO_4 \rightarrow Na_2SO_4 + MnSO_4 + Cl_2 \uparrow + 2H_2O$
Greenish yellow gas

$2KI + Cl_2 \rightarrow 2KCl + I_2$

4. $NaCl + AgNO_3 \rightarrow NaNO_3 + AgCl$
White ppt.

5. $AgCl + 2\ NH_4OH \rightarrow [Ag(NH_3)_2]\ Cl + 2H_2O$
Diamino Silver (I) Chloride
(Water soluble complex)

Test for Nitrate (NO_3^-):

EXPERIMENT	OBSERVATION	INFERENCE
1.Take a pinch of salt in a clean and dry test tube. Add few pieces of copper turnings and 4 to 5 drops ofconc. $H_2SO_4$and heat it.	1.Copious brown fumes are evolved and the solution turns green or bluish green.	1. Brown fume is due to NO_2 from NO_3^- salt.

2. Show a filter paper soaked in freshly prepared $FeSO_4$ Solution to the above brown gas. 3.**Brown Ring Test:**Take 1-2 ml. of the salt solution. Add equal volume ofconc. H_2SO_4slowly into the test tube. Cool the test tube perfectly under tap. Then slowly add 2-3 ml of freshly prepared $FeSO_4$ Solution through the sides of test tube.	2. The paper turns black. 3.A brown ring is formed at the junction of the two liquid layers.	2. May be NO_3^- 3.The brown ring is due to the formation of $[Fe(NO)]SO_4$ $NO_3^-$is confirmed

Explanation for Nitrate Test:

1. $$Cu + 4HNO_3 \xrightarrow{\Delta} Cu(NO_3)_2 + 2H_2O + 2NO_2\ t$$ Brown gas

2. $$2NaNO_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2HNO_3$$

3. $$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + 2NO + 4H_2O$$

$$FeSO_4 + NO + 5H_2O \rightarrow [Fe(NO)(H_2O)_5]SO_4$$

Brown Complex

Test for Sulphate (SO_4^{2-}):

EXPERIMENT	OBSERVATION	INFERENCE
1.Take about 1-2 ml of salt solution. Acidify with 1-2 ml of dil. HCl add about 1 ml of $BaCl_2$ solution. Add about 1 ml of conc. HCl to the above solution and warm it.	1. A white precipitate is obtained. The precipitate is not soluble.	1. $SO_4^{2-}$is confirmed.

Explanation for Sulphate Test:

The white precipitate is due to the formation of $BaSO_4$ which is insoluble in conc. HCl.

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4\downarrow + 2NaCl$$

White

ASSIGNMENT QUESTIONS:

1.What is acid radicals ?
2.How do u test for carbonate ?
3.What is the chemical equation involved in the test for sulphate ion ?
4.How do you test for sulphide ion ?
5. How can you test for nitrate ion ?
6. How does a salt become crystalline and amorphous?
7.How do you detect the presence of phosphate ion ?
8.Give the example of chemical reaction which involved for carbonate radicals ?
9.The acid radical carries --------ion.
10.What is brown ring test ?

EXPERIMENT NO-6:

AIM OF THE EXPERIMENT:

Test for ammonium, zinc, magnesium, aluminium, calcium, sodium & potassium (Known)

APPARATUS REQUIRED:

1. Test tube holder
2. Watch Glass
3. Blow pipe
4. Nichrome wire
5. Blue glass
6. Charcoal cavity
7. Test tubes

CHEMICALS REQUIRED:

1. Given salts
2. Various Reagent
3. Litmus paper

THEORY & PROCEDURE:

A.DRY TEST FOR BASIC RADICALS:

I. Heating in a dry Test Tube.

EXPERIMENT	OBSERVATION	INFERENCE
Heat a small quantity of supplied salt in a clean, dry test tube first slowly and then strongly for about 3 to 4 minutes.	a) Water particles condense at the cooler part of the test-tube.	a) Salt contains water of crystallisation.
	b) A sublimate is formed.	b) Volatile Salts
	White sublimate NH_4NO_3 is volatile but produces no white sublimate.	May be NH_4^+ , Hg^{2+} or As^{3+} salts
	c) The salt decrepitates. (produces cracking sound)	c) Crystalline Salts.
	d) Deflagation takes place. (Catches fire)	
	e) Infusible mass left.	d) Some nitrate or nitrite salts.

	f) The salt changes colour: i) Yellow when hot and white when cold. ii) Red to black when hot and brown when cold. g) The salt is fused on heating and solidified on cooling. h) The salt is swelled up on heating. A gas or vapour is evolved. (i) A colourless, odourless gas (CO_2) which turns lime water milky. (ii) A colourless gas (NH_3) with pungent odour which turns red litmus paper blue.	e) May be Mg^{2+}, Al^{3+}, Zn^{2+} etc. f) (i) May be Zinc (Zn^{2+}) salt ii) May be Fe^{2+} or Fe^{3+} g) May be alkali or alkaline earth metal salts. h) May be Al^{3+} (i) May be carbonate salts. (ii) May be ammonium salts.

. Heating in a Charcoal Cavity.

EXPERIMENT	OBSERVATION	INFERENCE
Make a small cavity on a charcoal block. Fill the with the supplied salt. Moisten the salt with a drop of water. Heat the salt strongly with the oxidising flame for about three minutes with help of a blow pipe.	(i) The salt decrepitates or produces cracking sound. (ii) The salt volatilises completely with white fumes. (iii) The salt deflagrates (iv) The salt may or may not melt but finally leaves a white infusible incandescent (giving light) residue. (v) The salt melts and sinks into the charcoal cavity on heating and reappears on cooling.	(i) Crystalline salts. **(ii)** May be ammonium, arsenic or mercury salts **(Perform soda lime and bulb tube test).** (iii) May be nitrate or nitrite (iv) Aluminium, zinc, magnesium,

	(vi)The salt is coloured and leaves a coloured residue.	tin or alkaline earth metal salts. (**Perform cobalt nitrate test**). (v) May be alkali or alkaline on earth metal salts (**Perform flame test**). (vi) May be chromium, manganese, iron, cobalt, nickel or copper salts. **(Perform borax bead test).**

III. Soda lime Test: (For Volatile Salts)

EXPERIMENT	OBSERVATION	INFERENCE
Take a pinch of the salt in a watch glass. Add a little sodalime (NaOH + CaO) and few drops of water to it. Rub it with the thumb.	a) A colourless gas with smell of ammonia is evolved which produces a dense white fume with concentrated HCl.	a) $NH_4^+$is present.

IV. Cobalt Nitrate Test: (For Infusible Salts)

EXPERIMENT	OBSERVATION	INFERENCE
Heat a small quantity of the salt in charcoal cavity in the oxidizing flame with the help of a blow pipetill an infusible and incandescent residue is left. Moisten in the Residue with a drop of Cobalt Nitrate solution and heat in the oxidising flame. Note the colour of the residue	a) Blue (infusible) mass. b) Green mass. c) Pink mass d) Grey mass	a) May be aluminium salt. b) May be Zinc Salt. c) May be Magnesium Salt. d) May be Calcium Salt.

V. Flame Test: (For fusible Salts)

EXPERIMENT	OBSERVATION		INFERENCE
Clean a nichrome wire or a Platinum wire by robbing it with Sandpaper.Dip in concentrated HCl taken in a watch glass.Show it to the non-luminous Bunsen flame till it imparts no colour to the Bunsen flame. Moisten the clean where with concentrated HCl and touch it with a little of the powdered salt. Show it to the non-luminous flame. Observe the colour of the flame in naked eye and through double blue glass.	Colour of the flame		
	In naked eye	Through Double blue glass	
	i) Golden Yellow ii) Violet iii) Brick Red iv) Yellowish Green	i) Colourless ii) Crimson red iii) Light Green iv) Bluish Green	(i) May be Sodium salt. ii) May be Potassium Salt. iii) May be Calcium Salt. iv) May be Barium Salt.

VI. Charcoal Cavity Reduction Test: (For White salts changing Colour):

EXPERIMENT	OBSERVATION	INFERENCE
Prepare an intimate mixture of salt charcoal powder and fusion mixture in 1: 3: 1 proportion. Take a little of this mixture in a charcoal cavity and heat it in reducing flame with the help of a blow pipe.	i) White shinning malleable bead with lemon yellow incrustation and the bead marks paper.b) Red Scale without incrustation.	a) May be lead salt. b) May be Copper Salt.

❖ **B.WET TEST FOR BASIC RADICALS:**

Test For NH_4^+:

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution is treated with dil. NaOH and boiled.	1. Ammonia gas is evolved producing white fumes with a glass rod dipped in conc. HCl.	1. NH_4^+ is confirmed.
2. To 1-2 ml. of the salt solution, about 1ml dil. NaOH solution is added followed with a little Nessler's reagent.	2. Brown ppt. is obtained.	2. NH_4^+ is confirmed. [This is due to formation of $I-Hg-O-Hg-NH_2$]

Explanation for NH_4^+Test:

1. $NH_4Cl + NaOH \rightarrow NH_3\ t + NaCl + H_2O$
2. $NH_4Cl + 2K_2[HgI_4] + 4KOH \rightarrow I - Hg - O - Hg - NH_2 \downarrow + KCl + 7KI + 3H_2O$

Brown ppt

Test For Zn^{2+}:

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution solid NH_4Cl is added till saturation and dil. NH_4OH is added till ammoniacal and H_2S is passed through it.	1. A white precipitate is obtained.	1. May be Zn^{2+}. [It is due to the formation of ZnS]
2. To about 1-2 ml. of the salt solution, dil. NaOH solution is added drop by drop and then in excess.	2. A white precipitate is first obtained which is soluble in excess of the reagent.	2. Zn^{2+} is confirmed. [The white ppt. is due to the formation of $Zn(OH)_2$ which gets dissolved with excess of the reagent due to the formation of Na_2ZnO_2.
3. To about 1-2 ml of salt solution few drops of $K_4[Fe(CN)_6]$ solution is added.	3. A white precipitate is obtained.	3. Zn^{2+} is confirmed. [This is due to the formation of Zinc ferrocyanide]

Explanation for Zn^{2+}Test:

1. $ZnSO_4 + H_2S \rightarrow ZnS \downarrow (White) + H_2SO_4$
2. $ZnSO_4 + 2NaOH \rightarrow Zn(OH)_2 \downarrow (White) + Na_2SO_4$
3. $Zn(OH)_2 + 2NaOH \rightarrow Na_2ZnO_2\ (Sodium\ Zincate) + 2H_2O$
4. $2ZnSO_4 + K_4[Fe(CN)_6] \rightarrow Zn_2[Fe(CN)_6] + 2K_2SO_4$

Zinc ferrocyanide (White)

Test for Mg^{2+}

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution solid NH_4Cl is added till saturation and dil. NH_4OH is added till alkaline and disodium hydrogen phosphate solution is added to it.	1. A white precipitate is obtained.	1. May be Mg^{2+}. [This is due to the formation of $Mg(NH_4)PO_4$].
2. To about 1-2 ml. of the salt solution about 1 ml of dil. HCl is added followed with 2-3 drops of Magneson reagent and the solution is made strongly alkaline with NaOH solution.	2. A blue precipitate is obtained.	2. Mg^{2+} is confirmed. [This is due to the formation of magnesium salt of magneson reagent.]

Explanation for NH_4^+Test:

3. $NH_4Cl + NaOH \rightarrow NH_3\ t + NaCl + H_2O$
4. $NH_4Cl + 2K_2[HgI_4] + 4KOH \rightarrow I - Hg - O - Hg - NH_2 \downarrow + KCl + 7KI + 3H_2O$

Brown ppt

Test For Zn^{2+}:

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution solid NH_4Cl is added till saturation and dil. NH_4OH is added till ammoniacal and H_2S is passed through it.	1. A white precipitate is obtained.	1. May be Zn^{2+}. [It is due to the formation of ZnS]
2. To about 1-2 ml. of the salt solution, dil. NaOH solution is added drop by drop and then in excess.	2. A white precipitate is first obtained which is soluble in excess of the reagent.	2. Zn^{2+} is confirmed. [The white ppt. is due to the formation of $Zn(OH)_2$ which gets dissolved with excess of the reagent due to the formation of Na_2ZnO_2.
3. To about 1-2 ml of salt solution few drops of $K_4[Fe(CN)_6]$ solution is added.	3. A white precipitate is obtained.	3. Zn^{2+} is confirmed. [This is due to the formation of Zinc ferrocyanide]

Explanation for Zn^{2+}Test:

5. $ZnSO_4 + H_2S \rightarrow ZnS \downarrow (White) + H_2SO_4$
6. $ZnSO_4 + 2NaOH \rightarrow Zn(OH)_2 \downarrow (White) + Na_2SO_4$
7. $Zn(OH)_2 + 2NaOH \rightarrow Na_2ZnO_2\ (Sodium\ Zincate) + 2H_2O$
8. $2ZnSO_4 + K_4[Fe(CN)_6] \rightarrow Zn_2[Fe(CN)_6] + 2K_2SO_4$

Zinc ferrocyanide (White)

Test for Mg^{2+}

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution solid NH_4Cl is added till saturation and dil. NH_4OH is added till alkaline and disodium hydrogen phosphate solution is added to it.	1. A white precipitate is obtained.	1. May be Mg^{2+}. [This is due to the formation of $Mg(NH_4)PO_4$].
2. To about 1-2 ml. of the salt solution about 1 ml of dil. HCl is added followed with 2-3 drops of Magneson reagent and the solution is made strongly alkaline with NaOH solution.	2. A blue precipitate is obtained.	2. Mg^{2+} is confirmed. [This is due to the formation of magnesium salt of magneson reagent.]

Test For Na^+:

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution about 1 ml of potassium pyroantimonate solution is added and the inner side of the test tube is scratched with glass rod.	1. A White precipitate is obtained.	1. Na^+ is confirmed. [The ppt. is due to the formation of $Na_2H_2Sb_2O_7$]

Explanation for Na^+Test:

1. $2NaCl + K_2H_2Sb_2O_7 \rightarrow Na_2H_2Sb_2O_7 + 2KCl$

White ppt. of Sodium Pyroantimonate

Test For K^+:

EXPERIMENT	OBSERVATION	INFERENCE
1. To about 1-2 ml of salt solution solid $NaNO_2$ is added till saturation followed with few drops $Co(NO_3)_2$ solution. About 1 ml of dil. CH_3COOH is added to it and the solution is kept for sometime.	1. A Yellow precipitate is obtained.	1. K^+ is confirmed. [This is due to the formation of $K_3[Co(NO_2)_6]$].

Explanation for K^+Test:

1. $KCl + NaNO_2 \rightarrow KNO_2 + NaCl$
2. $Co(NO_3)_2 + 2NaNO_2 \rightarrow Co(NO_2)_2 + 2NaNO_3$
3. $Co(NO_2)_2 + 2KNO_2 + 2CH_3COOH \rightarrow Co(NO_2)_3 + 2CH_3COOK + NO\uparrow + H_2O$
4. $Co(NO_2)_3 + 3KNO_2 \rightarrow K_3[Co(NO_2)_6]$

ASSIGNMENT QUESTIONS:

1. What is dry test?
2. Which type of salt is subjected to sodalime test?
3. Which type of salt is subjected to flame test?
4. What type of wire can be used in the flame test?
5. What is wet test of salt
6. What are the group-I radicals and how they precipitated?
7. For what type of salts charcoal cavity reduction test is performed ?
8. For what type salt cobalt nitrate test is carried out?
9. What is the principle of charcoal cavity reduction test ?
10. Which dry test is applied for infusible salt to identify its basic radicals

EXPERIMENT NO-7

AIM OF THE EXPERIMENT:

Test for unknown acid radicals.

APPARATUS REQUIRED:

1. Test tube holder
2. Bunsen Burner
3. Spatula

CHEMICALS REQUIRED:

1.Given salts

2.Various Reagent

3. Litmus paper

THEORY & PROCEDURE:

PRELIMINARY TEST:

1.Salt No----------

2.Colour of the salt----------

3.Structure----------

4.Solubility of the salt--------

B. TEST FOR ACID RADICALS:

EXPERIMENT	OBSERVATION	INFERENCE
I. Test with dil. HCl (For CO_3^{2-}, S^{2-})		
About 2ml of dil. HCl is taken in a clean test tube. It is warmed and small amount of the supplied salt is added to it.	(a) Effervescence takes place with the evolution of a colourless, odourless gas. (b) Effervescence takes place with the evolution of a colourless gas with the smell of rotten egg. (c) No effervescence and no gas is evolved.	(a) It may be CO_2 from CO_3^{2-} (b) May be H_2S from S^{2-}. (c) CO_3^{2-},S^{2-} are absent.

Confirmatory Test for CO_3^{2-}		
The gas evolved from the reaction of dilute HCl and the salt is passed through lime water	Lime water is turned milky and with excess of gas disappeared.	CO_3^{2-} is confirmed.
Confirmatory Test for S^{2-}		
The gas is passed through lead acetate solution or a filter paper dipped in lead acetate solution is shown to the gas	It is turned into black.	S^{2-} is confirmed.

EXPERIMENT	OBSERVATION	INFERENCE
II.(a) Test with conc. H_2SO_4 For Cl^-		
A Pinch of salt is taken in a clean and dry test tube. About 2 to 3 drops at conc. H_2SO_4 was added. Then it was warmed slightly	(a) Effervescence takes place with the evolution of a colourless gas with pungent odour. White fumes were produced when a glass rod dipped in conc. NH_4OH is shown to the gas. (d) No effervescence and no gas is evolved.	(a) May be Cl^- (Confirm by $AgNO_3$ test.) (d) Cl^- is absent.
(b)Confirmatory Test for Cl^-(Silver Nitrate Test)		
About 1 ml of salt solution taken in a test tube is acidified with dilute HNO_3 and $AgNO_3$ solution is added	(i) Curdy white precipitate soluble in dilute NH_4OH which reappeared on addition of dil.HNO_3.	(i) Cl^- is confirmed.

EXPERIMENT	OBSERVATION	INFERENCE
III.(a) Test for NO_3^- (Conc. H_2SO_4 and Copper turning)		
A little of the salt is heated with concentrated H_2SO_4 and a few copper turnings.	Brown fumes are evolved and solution in test tube is turned green.	(a) May be NO_3^-

(b) Brown Ring Test for NO_3^- (Confirmatory Test for NO_3^-)		
To about 1 ml of salt solution taken in a test tube equal volume of concentrated H_2SO_4 is added. It is cooled under tap water. Then freshly prepared $FeSO_4$ solution is added slowly.	A brown ring is formed at the junction of the two rings.	NO_3^- is confirmed.

EXPERIMENT	OBSERVATION	INFERENCE
IV. $BaCl_2$ Test for SO_4^{2-} (Confirmatory Test for SO_4^{2-})		
About 1 ml of salt solution taken in a test tube is acidified with dilute HCl and $BaCl_2$ solution is added	White precipitate is obtained which is insoluble in concentrated HCl even on boiling	SO_4^{2-} is confirmed.

CONCLUSION:

Acid radical of the salt is detected to be----------

EXPERIMENT NO-8

AIM OF THE EXPERIMENT:

Test for unknown basic radicals.

APPARATUS REQUIRED:

1. Test tube holder

2. Watch Glass

3. Blow pipe

4. Nichrome wire

5. Blue glass

6.Charcoal cavity

CHEMICALS REQUIRED:

1.Given salts

2.Various Reagent

3. Litmus paper

THEORY & PROCEDURE:

PRELIMINARY TEST:

1.Salt No----------

2.Colour of the salt----------

3.Structure----------

4.Solubility of the salt------

A.DRY TEST FOR BASIC RADICALS:

I. Dry Test tube heating

EXPERIMENT	OBSERVATION	INFERENCE
In a clean and dry test tube. A pinch of the salt is heated.	(a) Water vapours condensed at the cooler part of the test tube. (b) Decrepitation took place.	(a) Salt with water crystallisation. (b) May be crystalline salt

III. Sodalime Test.(for Volatile Salt)

	(c) Salt is volatilised and white sublimate is formed. (d) Salt is first melted and finally infusible white mass left. (e) Salt is fused on heating and solidified on cooling. (f) The colour of the salt is changed (i) Yellow when hot and white when cold. (ii) Yellow when Hot and Cold (iii) Yellowish brown in hot and yellow when cold. (iv) Black residue.	(c) May be Volatile salt of NH_4^+, As^{3+} and Hg^{2+}. (d) May be Mg^{2+}, Al^{3+}, Zn^{2+}, Ba^{2+}, Ca^{2+}, Sr^{2+} etc. (e) May be alkali or alkaline earth metal. (f) May be salt of Pb^{2+}, Bi^{2+} ,Sn^{2+} etc. The salt is non-volatile. (i) May be Zn^{2+}salt. (ii) May be Pb^{2+}salt. (iii) May be Sn^{2+}or Bi^{3+} salt. (iv) May be Cu^{2+} ,Ni^{2+} ,Mn^{2+} or Fe^{2+} salt.

II. Heating in a Charcoal Cavity.

A pinch of the salt is taken in a charcoal cavity and is heated in oxidising flame with a blow pipe.	(a) Salt is completely volatilised. (b) An infusible incandescent white mass is obtained. (c) The salt is fused and sank into the charcoal cavity and reappeared on cooling. (d) Original salt is white and formed a coloured mass. (e) Original salt is coloured and formed a coloured mass.	(a) May be salt of NH_4^+, As^{3+} and Hg^{2+}. (Sodalime test is to be performed). (b) May be Mg^{2+}, Al^{3+}, Zn^{2+}, Ba^{2+}, Ca^{2+},Sr^{2+},Sn^{2+}etc.(Cobalt nitrate test is to be performed). (c) May be alkali or alkaline earth metal salt. (Flame test is to be performed). (d) May be salt of Pb^{2+}, Bi^{2+}, Sn^{2+} Ag^+ etc. (Reduction test is to be performed). (e) May be Cr^{3+}, Ag^+, Mn^{2+} etc. (Borax bead test is to be performed).

A pinch of salt is taken in a watch glass. A little soda lime is added with a drop of water. Then it is rubbed.	(a) Ammonia gas is evolved and the colour of the mixture is not changed. (b) Only colour of the residue is changed to brown and there is no evolution of gas. (c) No gas is evolved and no change in colour of residue.	(a) May be NH_4^+ (b) May be Hg^{2+}. (c) May be As^{3+}

IV. Bulb tube test.(for Volatile Salt)

A mixture of salt, anhydrous Na_2CO_3 and charcoal powder in the proportion of 1: 3: 1 was prepared. A little of the mixture is taken in a bulb tube and heated.	(i) a white shinning mirror is formed (ii) A black shinning mirror is formed with the evolution of a gas having garlic colour.	(a) May be Hg^{2+}. (b) May be As^{3+}

V. Cobalt Nitrate test. (For infusible Salt)

A Pinch of salt is taken in a charcoal cavity. It is heated in an oxidizing flame till an infusible mass is obtained. A drop of cobalt nitrate solution is added and again heated strongly.	(i)Blue Mass (ii)Green Mass (iii)Pink Mass (iv)Grey Mass	(a) May be Al^{3+}. (b) May be Zn^{2+} (c) May be Mg^{2+} (d) May be Ca^{2+} (Flame test to be performed.)

VI. Flame test. (For fusible Salt)

A clean nichrome wire is moisten with concentrated HCl and touch it with a little of the powdered salt. Show it to the non-luminous flame. Observe the colour of the flame in naked eye and through double blue glass.	Colour through naked flame i) Golden Yellow ii) Violet iii) Brick Red	Colour through double blue glass. i) Colourless ii) Red iii) Light Yellow	(a) May be Na^+. (b) May be K^+ (c) May be Ca^{2+}

VII. Charcoal Reduction Test. (For white salt changing colour)

A mixture of salt and fusion mixture in the proportion of 1:1 is prepared. A little of this mixture is taken in a charcoal	(i) White shining malleable bead without incrustation which did not mark on paper. (ii) White shining malleable bead	(i) May be Ag^+.

cavity and is heated in a reducing flame.	with lemon yellow incrustation which marked on paper.	(ii) May be Pb^2

.WET TEST FOR BASIC RADICALS:

1. WET TESTS FOR BASIC RADICALS (Group Analysis)

Experiment	Observation	Inference
1. To 1ml. of salt solution in a clean test tube 1 cc. of dil HCI is added.	a. A white precipitate is formed.	a. One of the Gr. I basic radicals (**Pb^{2+}, Ag^{+}, Hg_2^{2+}**) may be present (Analysis of Gr. I basic radicals should be performed)
	b. No white precipitate is formed	b. Gr. I basic radicals are absent.
2. To 1ml. of the supplied salt solution in a clean test tube solid NH"CI is added till saturation followed by addition of dil NH4OH till alkaline.	a. A precipitate is obtained, (colour should be noted)	a. One of the Gr III A basic radicals (**Fe^{3+}, Al^{3+}, Cr^{3+}**)may be present (Analysis of Gr III A basic radicals should be performed)
	b. No precipitate is formed.	b. Gr III A basic radicals are absent.
3. Through the contents of the above test tube H_2S gas is passed under pressure.	a. Precipitate is formed (colour should be noted).	a. One of the Gr III B basic radicals (**Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}**) may be present(analysis of Gr III B radicals should be performed)
	b. No precipitate is formed	b. Gr III B basic radicals are absent.
4. To 1 cc of the salt solution is taken in a clean test tube solid NH_4Cl is added till saturation followed by addition of dil NH_4OH till alkaline. To this saturated solution of ammonium carbonate is added.	a. Precipitate is formed (colour should be noted).	a. One of the Gr IV basic radicals (**Ba^{2+}, Sr^{2+}, Ca^{2+}**) may be present(analysis of Gr IV radicals should be performed)
	b. No precipitate is formed.	b. Gr. IV basic radicals are absent.

The above basic radicals are absent indicating that one of the Gr. V basic radicals may be present. As there is no specific group reagent for Gr. V test for individual radicals should be performed

2. ANALYSIS OF BASIC RADICALS (GROUP WISE)

i) Analysis of Gr. IIIA Basic Radicals (Al^{3+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline.	A white ppt. is formed.	May be Al^{3+}
2. 1 – 2 cc of the supplied salt solution is treated with dil NaOH solution drop wise and then in excess.	A white ppt. of $Al(OH)_3$ is formed which dissolved in excess of the reagent.	May be Al^{3+}
3. 1 cc of the supplied salt solution, disodium hydrogen phosphate solution is added.	A gelatinous white ppt. of $AlPO_4$ is formed which is soluble in dil. HCl solution.	**Al^{3+} confirmed.**

ii) Analysis of Gr. IIIB Basic Radicals (Zn^{2+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline. Then H_2S gas is passed through it.	A white ppt. is formed.	May be Zn^{2+}
2. 1 -2 cc of the supplied salt solution is treated with potassium ferrocyanide solution drop by drop and then in excess.	A white ppt is obtained.	May be Zn^{2+}
3. Dil. NaOH solution is added to 1 cc of the salt solution drop by drop and then in excess.	A gelatinous white ppt. is formed which is soluble in excess of NaOH solution.	**Zn^{2+} confirmed.**

iii) Analysis of Gr. IV Basic Radicals (Ca^{2+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl and then made alkaline with dil	A white ppt. of $CaCO_3$ is formed.	May be Ca^{2+}

NH_4OH solution. Then saturated solution of ammonium carbonate $[(NH_4)_2CO_3]$ is added.		
2. The above ppt. is dissolved in a minimum quantity of dil CH_3COOH. The solution is boiled to remove CO_2 and then ammonium oxalate solution is added to it.	A white ppt. of CaC_2O_4 is formed which is soluble in dil. HCl but insoluble in CH_3COOH.	May be Ca^{2+}

ii) Analysis of Gr.V Basic Radicals (NH_4^+, Na^+, K^+)

Tests for NH_4^+

Experiment	Observation	Inference
1. A small quantity of the salt is treated with soda lime and two drops of water and then the mixture is rubbed in a mortar.	A colourless gas having smell of ammonia which produced dense white fumes with a glass rod dipped in conc. NH_4OH. There is no change in the colour of the residue.	**NH_4^+ confirmed.**
2. Nessler's reagent is added to 1 cc of the salt solution.	A brown ppt. is obtained.	**NH_4^+ confirmed.**

Tests for Mg^{2+}

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline. Then dihydrogen sodium phosphate solution is added to it.	A white ppt. is formed.	May be Mg^{2+}
2. 1 cc of the salt solution is acidified with dil. HCl and then treated with a few drops of magneson reagent followed by the addition of excess of dil NaOH solution.	A blue ppt. is obtained.	**Mg^{2+} confirmed.**

Tests for Na^+

Experiment	Observation	Inference
1. Potassium pyroantimonate solution is added to 1 cc of the supplied salt solution.	A white crystalline ppt. is formed.	**Na^+ confirmed.**

Tests for K^+

Experiment	Observation	Inference
1. 1 cc of the salt solution is treated with two drops of cobalt nitrate solution followed by the addition of solid $NaNO_3$ and dil. CH_3COOH solution.	A yellow ppt. is formed.	**K^+ confirmed.**

Hence, the basic part of the supplied salt is_____.

EXPERIMENT NO. 09

(Tests for Unknown Salt)

AIM OF THE EXPERIMENT:

Test for unknown basic radicals.

APPARATUS REQUIRED:

1. Test tube holder
2. Watch Glass
3. Blow pipe
4. Nichrome wire
5. Blue glass
6. Charcoal cavity

CHEMICALS REQUIRED:

1. Given salts
2. Various Reagent
3. Litmus paper

THEORY & PROCEDURE:

1. Preliminary Test:

(a) Salt No:
(b). Colour of the Salt : Colourless / name of the colour
(c) . Structure of Salt : Crystalline/ Amorphous
(d). Solubility:
i) Soluble in cold water (if not)
ii) Soluble in hot water (if not)
iii) Soluble in dilute HCl (if not)
iv) Soluble in hot dilute HCl
v) If not then salt is insoluble (*Salt soluble in dil HCl implies Gr.I basic radicals absent)

2. DRY TEST FOR BASIC RADICALS

Dry Test Tube heating:

Experiment	Observation	Inference
A small quantity of salt is taken in a clean and dry test tube and heated strongly in the hottest part of the non-luminous flame.	(a) A sublimate is formed (Note the colour of the sublimate)	(a) It is volatile salt, (Soda lime test and bulb tube test should be performed.)
	b)Water particles condense at the cooler part of the test	(b) Salt contains water of crystallisation.
	(c) Decripitation or cracking sound is produced.	(c) May be crystalline salt.
	(d) Deflagration takes place.	(d) The salt may be nitrate of alkali or alkaline earth metal.
	(e) The salt changes colour. Yellow when hot and white when cold.	(e) It may be Zinc salt.

	(f) Salt fuses on heating and solidifies on cooling.	(f) May be alkali or alkaline earth metal salt.

3. SODALIME TEST:

Experiment	Observation	Inference
A little of the salt is taken in a clean watch glass along with soda-lime and it is rubbed by adding two drops of water.	A colourless gas evolved with strong smell of ammonia and colour of the mixture is unchanged.	NH_4^+ may be present. (To be confirmed in the wet test)

4. CHARCOAL CAVITY HEATING (OXIDISING FLAME)

Experiment	Observation	Inference
A little of the Salt is taken in the charcoal cavity and heated by oxidizing flame with the help of a blow pipe.	a. The salt decrepitates.	a. Maybe crystalline salt.
	b. The salt deflagrates.	b. May be NO_3^- salt
	c. The salt fuses and sinks into the charcoal cavity.	c. Salt contains alkali or alkaline earth metal. (Flame test should be performed).
	d. Infusible incandescent white residue.	d. Cobalt nitrate test should be performed.

5. COBALT NITRATE TEST

Experiment	Observation	Inference
The salt is taken in the charcoal cavity and heated in the oxidizing flame with the help of a blow pipe till an infusible, incandescent white mass is obtained. Then one drop of cobalt nitrate solution is added to it and heated strongly.	a. Blue mass is obtained.	a. Al^{3+} may be present.
	b. Green mass is obtained.	b. Zn^{2+} may be present.
	c. Rosy mass is obtained.	c. Mg^{2+} may be present.
	d. Grey mass is obtained.	d. Ca^{2+} may be present.

6. FLAME TEST

Experiment	Observation	Inference
The nichrome wire is cleaned with sand paper and dipped in conc. HCl and shown to non-luminous flame. This process is repeated till no colour is imparted to the flame. Then the wire is moistened with conc. HCl and a little of the salt is taken by touching to the salt is taken by touching to the salt and shown to the oxidizing flame.	a. Persistent golden Yellow coloured flame is seen in naked eye and colourless through double blue glass. b) Violet flame is seen in naked eye and red through a pair of blue glass.	a. Na^+ may be present.
	b. Violet flame is seen in naked eye and red through a pair of blue glass.	b. K^+ may be present.
	c. Brick red flame is observed.	c. Ca^{2+}may be present.

7. IDENTIFICATION OF ACID RADICAL

Test for Gr- I acid radicals (Carbonate and Sulphide)

Experiment	Observation	Inference
1 cc dilute HCl taken in d test tube and slightly warmed. To this a pinch of the supplied salt is added.	1. Effervescence took place with the evolution of a colourless odourless gas is evolved.	a. Carbonate (CO_3^{2-}) may be present (other test should be performed for its confirmation.)
	2. Effervescence took place with the evolution of a colourless odourless gas with rotten egg smell is evolved.	a. Sulphide (S^{2-}) may be present (other test should be performed for its confirmation.)

Test for Gr- II acid radicals (Chloride)

Experiment	Observation	Inference
A few drops of conc. H_2SO_4 is taken in a clean and dry test tube, a pinch of the supplied salt is added in to it and is gently warmed.	A colourless fuming gas with pungent odour is evolved.	Cl^- may be present. (Other test should be performed for its confirmation).

Test for Gr- III acid radicals (Nitrate and Sulphate)

TESTS FOR NITRATE (NO_3^-)

Experiment	Observation	Inference
A pinch of the supplied salt is moistened with a few drops of conc. H_2SO_4 is taken in a clean and dry test tube and is gently warmed.	A brown fume with pungent smell is observed.	May be **NO_3^-.** (Other test should be performed for its confirmation).

TESTS FOR SULPHATE (SO_4^{2-})

Experiment	Observation	Inference
1-2 cc of the salt solution is taken in a clean test tube and is acidified with dil HCl. A few cc of Barium chloride ($BaCl_2$) solution is added into it.	A white ppt. is obtained which is insoluble in conc. HCl even on boiling.	**SO_4^{2-} confirmed.**

CONFIRMATORY TESTS FOR CARBONATE (CO_3^{2-})

Experiment	Observation	Inference
1. A burning match stick is shown to the evolved gas.	The burning stick extinguished.	CO_3^{2-} may be present.
2. A little more salt is added to the above test tube and the evolved gas is passed through lime water with the help of a delivery tube.	At first white turbidity (milk colour) appeared which disappeared with excess passing of the gas.	CO_3^{2-} may be present
3. A little more salt is added to the above test tube and the evolved gas is passed through acidified potassium dichromate solution with the help of a delivery tube.	No change of the colour took place.	**CO_3^{2-} confirmed**

CONFIRMATORY TESTS FOR SULPHISE (S^{2-})

Experiment	Observation	Inference
A filter paper soaked with Lead acetate solution is shown to the mouth of the test tube.	The filter paper turned black.	**S^{2-} Confirmed.**

CONFIRMATORY TESTS FOR CHLORIDE (Cl^-)

Experiment	Observation	Inference
1. A glass rod dipped in conc. NH_4OH solution is shown to the gas evolved.	A white dense fume is formed.	Cl^- may be present.
2. A pinch of MnO_2 is added to the above test tube and is warmed gently.	A greenish yellow gas is formed which turned starch iodide paper blue.	Cl^- may be present.
3. A pinch of the given salt is taken in a clean and dry test tube and is acidified with dil HNO_3 solution. And a few drops of silver nitrate ($AgNO_3$) solution is added into it.	A curdy white ppt. is formed which is soluble in dil NH_4OH and is insoluble in dil HNO_3.	**Cl^- confirmed.**

CONFIRMATORY TEST FOR NITRATE (NO_3^-)

Experiment	Observation	Inference
1. A pinch of the supplied salt and a few copper turnings are taken in a clean test tube. 1 – 2 cc of 50% conc. H_2SO_4 is added into it and is heated gently.	Deep brown vapours are formed and the solution turned bluish green or green.	May be **NO_3^-.**
2. A piece of filter paper soaked in $FeSO_4$ solution is shown to the	It turned black.	May be **NO_3^-.**

evolved gas.		

3. 1 cc of the supplied salt solution in water is taken in a clean test tube. Equal volume of conc. H_2SO_4 is added in to the test tube. The test tube is cooled under tap water. And equal volume of freshly prepared ferrous sulphate ($FeSO_4$) solution is added from the side of the test tube.	A brown ring is formed at the junction of the two liquids. The ring disappeared on shaking.	**NO_3^- confirmed.**

8. WET TESTS FOR BASIC RADICALS (Group Analysis)

Experiment	Observation	Inference
1. To 1ml. of salt solution in a clean test tube 1 cc. of dil HCl is added.	a. A white precipitate is formed.	a. One of the Gr. I basic radicals (**Pb^{2+}, Ag^+, Hg_2^{2+}**) may be present (Analysis of Gr. I basic radicals should be performed)
	b. No white precipitate is formed	b. Gr. I basic radicals are absent.
2. To 1ml. of the supplied salt solution in a clean test tube solid NH"Cl is added till saturation followed by addition of dil NH4OH till alkaline.	a. A precipitate is obtained, (colour should be noted)	a. One of the Gr III A basic radicals (**Fe^{3+}, Al^{3+}, Cr^{3+}**)may be present (Analysis of Gr III A basic radicals should be performed)
	b. No precipitate is formed.	b. Gr III A basic radicals are absent.
3. Through the contents of the above test tube H_2S gas is passed under pressure.	a. Precipitate is formed (colour should be noted).	a. One of the Gr III B basic radicals (**Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}**) may be present(analysis of Gr III B radicals should be performed)
	b. No precipitate is formed	b. Gr III B basic radicals are absent.
4. To 1 cc of the salt solution is taken in a clean test tube solid NH_4Cl is added till saturation	a. Precipitate is formed (colour should be noted).	a. One of the Gr IV basic radicals (**Ba^{2+}, Sr^{2+}, Ca^{2+}**) may be

followed by addition of dil NH_4OH till alkaline. To this saturated solution of ammonium carbonate is added.		present(analysis of Gr IV radicals should be performed)
	b. No precipitate is formed.	b. Gr. IV basic radicals are absent.

The above basic radicals are absent indicating that one of the Gr. V basic radicals may be present. As there is no specific group reagent for Gr. V test for individual radicals should be performed.

9. ANALYSIS OF BASIC RADICALS (GROUP WISE)

ii) Analysis of Gr. IIIA Basic Radicals (Al^{3+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline.	A white ppt. is formed.	May be Al^{3+}
2. 1 – 2 cc of the supplied salt solution is treated with dil NaOH solution drop wise and then in excess.	A white ppt. of $Al(OH)_3$ is formed which dissolved in excess of the reagent.	May be Al^{3+}
3. 1 cc of the supplied salt solution, disodium hydrogen phosphate solution is added.	A gelatinous white ppt. of $AlPO_4$ is formed which is soluble in dil. HCl solution.	**Al^{3+} confirmed.**

iv. Analysis of Gr. IIIB Basic Radicals (Zn^{2+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline. Then H_2S gas is passed through it.	A white ppt. is formed.	May be Zn^{2+}
2. 1 -2 cc of the supplied salt solution is treated with potassium ferrocyanide solution drop by drop and then in excess.	A white ppt is obtained.	May be Zn^{2+}
3. Dil. NaOH solution is added to 1 cc of the salt solution drop by drop and	A gelatinous white ppt. is formed which is soluble in excess of NaOH solution.	**Zn^{2+} confirmed.**

then in excess.		

iv) Analysis of Gr. IV Basic Radicals (Ca^{2+})

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl and then made alkaline with dil NH_4OH solution. Then saturated solution of ammonium carbonate $[(NH_4)_2CO_3]$ is added.	A white ppt. of $CaCO_3$ is formed.	May be Ca^{2+}
2. The above ppt. is dissolved in a minimum quantity of dil CH_3COOH. The solution is boiled to remove CO_2 and then ammonium oxalate solution is added to it.	A white ppt. of CaC_2O_4 is formed which is soluble in dil. HCl but insoluble in CH_3COOH.	May be Ca^{2+}
3. Flame test is performed with the white ppt. formed above.	Brick red flame is noticed.	**Ca^{2+} confirmed.**

iii) Analysis of Gr.V Basic Radicals (NH_4^+, Na^+, K^+)

Tests for NH_4^+

Experiment	Observation	Inference
1. A small quantity of the salt is treated with soda lime and two drops of water and then the mixture is rubbed in a mortar.	A colourless gas having smell of ammonia which produced dense white fumes with a glass rod dipped in conc. NH_4OH. There is no change in the colour of the residue.	**NH_4^+ confirmed.**
2. Nessler's reagent is added to 1 cc of the salt solution.	A brown ppt. is obtained.	**NH_4^+ confirmed.**

Tests for Mg^{2+}

Experiment	Observation	Inference
1. 1 – 2 cc of the supplied salt solution is saturated with solid NH_4Cl followed by the addition of dil NH_4OH solution till alkaline. Then dihydrogen sodium phosphate solution is added to it.	A white ppt. is formed.	May be Mg^{2+}
2. 1 cc of the salt solution is	A blue ppt. is obtained.	**Mg^{2+} confirmed.**

acidified with dil. HCl and then treated with a few drops of magneson reagent followed by the addition of excess of dil NaOH solution.		

Tests for Na^+

Experiment	Observation	Inference
1. Potassium pyroantimonate solution is added to 1 cc of the supplied salt solution.	A white crystalline ppt. is formed.	**Na^+ confirmed.**

Tests for K^+

Experiment	Observation	Inference
1. 1 cc of the salt solution is treated with two drops of cobalt nitrate solution followed by the addition of solid $NaNO_3$ and dil. CH_3COOH solution.	A yellow ppt. is formed.	**K^+ confirmed.**

Hence, the basic part of the supplied salt is_____and the acid part of the salt is_____.

Thus, the salt supplied is_____.