

LABORATORY MANUAL
ON ENGINEERING CHEMISTRY
OF
1ST/2ND SEMESTER
(COMMON TO ALL BRANCHES)

According to the New Syllabus of SCTE & VT

NEW SYLLABUS (Effective from 2018-19 session)

Pr.2b. Engineering Chemistry Lab
(1st / 2nd semester Common)

Practical: 4 periods/week
Marks Total Periods: 60 Periods
50 Marks Examination: 3 Hours

Sessional : 50
End Sem Exams :
Total Marks :100

| SI No. | NAME OF THE EXPERIMENTS |
|--------|---|
| 01 | Preparation and study of physical and chemical properties CO ₂ gas. |
| 02 | Preparation and study of physical and chemical properties NH ₃ gas |
| 03 | Crystallization of Copper sulphate from copper carbonate |
| 04 | Simple acid-base titrations (i) Acidimetry (ii) Alkalimetry |
| 05 | Tests for acid radicals (Known): (i) Carbonate, (ii) Sulphide, (iii) Chloride, (iv) Nitrate and (v) Sulphate. |
| 06 | Test for Basic radicals (Known): (i) Ammonium, (ii) Zinc, (iii) Magnesium, (iv) Aluminium, (v) Calcium, (vi) Sodium and (vii) potassium |
| 07 | Test for unknown Acid radicals |
| 08 | Test for unknown basic radicals |
| 09 | Test for unknown salt (composed of one basic radical and one acid radical) |

INDEX TABLE

| SI No. | NAME OF THE EXPERIMENTS | PAGE NO. | DATE OF EXPERIMENT PERFORMED | GRADE /MARK | SIGN. OF SUBJECT FACULTY | REMARK |
|--------|---|----------|------------------------------|-------------|--------------------------|--------|
| 01 | Preparation and study of physical and chemical properties CO ₂ gas. | | | | | |
| 02 | Preparation and study of physical and chemical properties NH ₃ gas. | | | | | |
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| 06 | Test for Basic radicals (Known): (i) Ammonium, (ii) Zinc, (iii) Magnesium, (iv) Aluminium, (v) Calcium, (vi) Sodium and (vii) Potassium | | | | | |
| 07 | Test for unknown Acid radicals | | | | | |
| 08 | Test for unknown Basic radicals | | | | | |
| 09 | Test for unknown salt (composed of one basic radical and one acid radical) | | | | | |

EXPERIMENT NO.- 01

AIM OF THE EXPERIMENT:

Preparation and study of physical and chemical properties Carbon dioxide (CO₂) gas.

OBJECTIVES OF THE EXPERIMENT:

At the end of this experiment, students will be able to:

- a. Name and specify the chemicals, which are used to prepare CO₂ gas in the laboratory.
- b. Describe the physical and chemical properties of the gas
- c. Test carbonate radical in the given salt sample.
- d. Describe the procedure for preparation of gas in the laboratory.
- e. Know the precautions and safety measures.

SET UP OF EXPERIMENT:

A. Apparatus Required :

- a. Woulf's Bottle.
- b. Thistle Funnel
- c. Derive Tube.
- d. Rubber Cork,
- e. Gas Jar with Lid.
- f. Few Test Tubes.

B. Chemicals Required :

- a. Marble chips (CaCO₃),
- b. Dilute Hydrochloric Acid (HCl)
- c. Litmus Paper,
- d. Magnesium Ribbon
- e. Lime Water,
- f. Phenolphthalein Solution,
- g. NaOH Solution.

Theory:

In the Laboratory, carbon dioxide (CO₂) gas is prepared by the action of dilute hydrochloric acid (HCl) upon marble chips (CaCO₃) in a Woulf's bottle.

Chemical Equations :



Diagram :

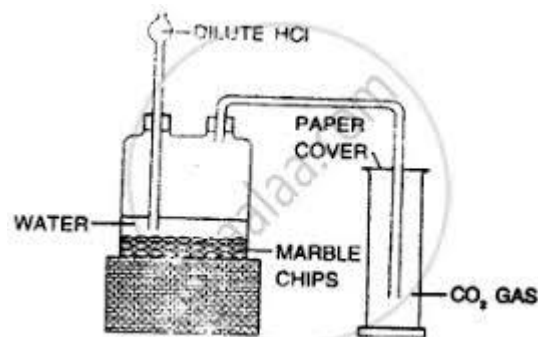


Fig. Preparation of CO₂ gas in laboratory

PROCEDURE:

Take a Woulf's bottle fitted with rubber cork, thistle funnel and delivery tube, Examine that it is perfect airtight. In case of air leakage, use melted paraffinwax or grease.

Introduce few small size marble chips into the Woulf's bottle by Opening one of its mouths

Now pour some water into the Woulf's bottle through the thistle funnel so as to cover the marble chips.

Insert the thistle funnel more into the Woulf's bottle such that its extreme end remains inside the water.

Now add little quantity of the dilute hydrochloric acid through the thistle funnel. Do not add excess amount of acid at a time to exhaust the marble chips before the experiment is completed.

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 the gas jar.

Study the properties of CO₂ gas by collecting the gas in different test tubes.

OBSERVATION:STUDY OF PROPERTIES

Physical Properties

| <u>SL.NO</u> | EXPERIMENT | OBSERVATION | INFERENCE |
|--------------|--|-------------|-----------|
| 01 | Appearance :Observe the colour of the gas | | |
| 02 | Odour : Smell the odour of the gas. | | |
| 03 | Burning Candle : Enter a burning candle into a test tube full of CO ₂ gas | | |
| 04 | Invert test tube full of CO ₂ gas over another empty test tube containing air. Then add little lime to the test tube containing air initially. | | |
| 05 | 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 water in the test tube. | | |

Chemical Properties

| SL NO. | EXPERIMENT | OBSERVATION | INFERENCE |
|--------|--|-------------|-----------|
| 01 | A piece of moist litmus paper is shown to the gas. | | |
| 02 | Pass the CO ₂ gas through the 2-3 ml of very dilute solution of sodium hydroxide containing one drop of phenolphthalein solution. | | |
| 03 | a. Pass the gas through limewater b. Pass the gas in excess c. Boil the solution | | |
| 04 | Introduce a burning magnesium ribbon into a test tube/gas jar containing carbon dioxide gas. | | |

SAFETY AND PRECAUTIONS:

- The fittings should be air tight. The end of the thistle funnel must remain deep inside the solution.
- The shorter end of the delivery tube should remain above the surface of the solution in the Wolf's bottle.
- The longer end of the delivery tube must reach the bottom of the gas jar.
- Addition of excess acid should be avoided.
- The gas should be collected after removing air from the apparatus.

EXPERIMENT NO.- 02

AIM OF THE EXPERIMENT:

Preparation and study of physical and chemical properties of Ammonia gas(NH_3).

OBJECTIVES OF THE EXPERIMENT:

At the end of this experiment, the students will be able to :

- Detect the chemicals and apparatus required for preparation of ammonia gas in the laboratory.
- Identify the formula of Nessler's reagent.
- Know the precautions and safety measures.

SET UP OF EXPERIMENT:

A. Apparatus Required:

- Hard Glass Test Tube
- Delivery tube
- Gas jar with lid
- Clamp Stand
- Bunsen Burner
- Cork
- Cork Borer
- Triangular file
- Test Tube

B. Chemicals Required:

- Solid Ammonium Chloride (NH_4Cl).
- Quick lime (CaO) or dry slakedlime $\text{Ca}(\text{OH})_2$

Theory:

Ammonia gas is prepared in the laboratory by heating an intimate mixture of solid ammonium chloride and quick lime or dry slaked lime in 1:3 ratio. The gas is collected by the downward displacement of air as it is lighter than air.

PREPARATION OF AMMONIA GAS

DIAGRAM

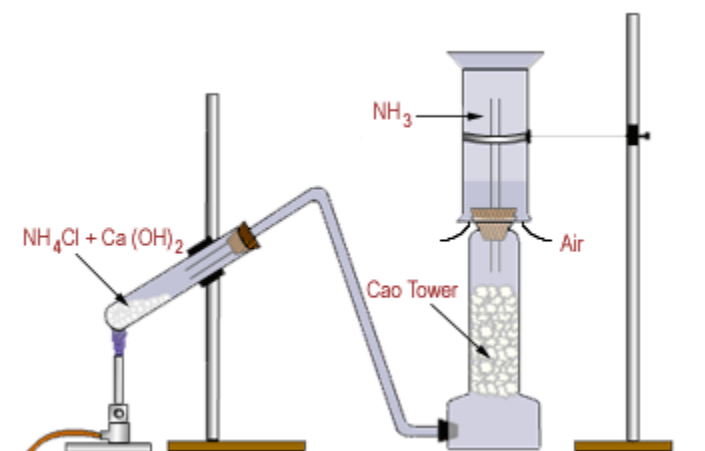
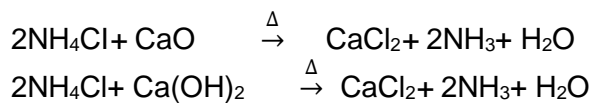


Fig. Preparation of Ammonia (NH_3) gas in laboratory

Chemical Equation



PROCEDURE

- Take a mixture of ammonium chloride and quick lime in 1:3 ratio.
in a mortar and mix them thoroughly and take this mixture in a hard glass test tube.
- The hard glass test tube should be half filled with the mixture.
- Fit the cork along with the delivery tube into the mouth of the hard glass test tube.
- Clamp the hard glass test tube in the clamp stand.
- Heat the hard glass test tube continuously.
- Collect the gas by downward displacement of air.

| SL NO. | EXPERIMENT | OBSERVATION | INFERENCE |
|--------|---|-------------|-----------|
| 01 | Observe the colour of the gas | | |
| 02 | Introduce a burning match stick into the gas jar containing gas | | |
| 03 | .Invert test gas jar containing ammonia gas into the water | | |
| 04 | Show a moist red litmus paper to the gas. | | |
| 05 | Show a glass rod dipped in concentrated HCl to the gas | | |
| 06 | pass the gas through about 2 CC of Nessler's reagent in a clean dry test tube | | |
| 07 | Pass 2 CC of ferric chloride solution in a Clean dry test tube. | | |
| 08 | First Slowly pass the gas through 2 CC of aqueous copper sulphate solution in a clean dry test tube and then in excess. | | |

Safety and Precautions

- i. The mixture should be prepared with proper ratio.
- ii. The hard glass tube should be fitted slightly inclined with the mouth downward so that water droplets, which will be produced during the reaction, are collected at the mouth of the test tube.
- iii. The fittings should be air tight.
- iv. The collected gas should be perfectly dried.
- v. The students should not inhale the gas.

EXPERIMENT NO.- 03

AIM OF THE EXPERIMENT:

Crystallization of copper sulphate from copper carbonate.

OBJECTIVES OF THE EXPERIMENT:

At the end of this experiment, students will be able to know

- a. The preparation of a crystal from its saturated solution.
- b. Purification of a substance through the process of crystallization.
- c. General basic properties of a crystal.

SET UP OF EXPERIMENT:

A. Apparatus Required :

a. Beaker, b. Funnel ,c. Glass rod d. China dish e. Bunsen Burner, f. Tripod stand g. Wire gauge
h. Filter stand, i.Filter paper

B. Chemicals Required:

a. Copper Carbonate (CuCO_3), b. Dilute Sulphuric acid (H_2SO_4)

PROCEDURE

At first, about 40 ml of dil.sulphuric acid is taken in a beaker. The copper carbonate powder is added to the beaker pinch by pinch till a small quantity of copper carbonate solid left undissolved. Then the resulting solution in the beaker is heated for 2-3 minute to drive out carbon dioxide gas. The beaker is cooled and filtered into china dish. A few drop of dil.sulphuric acid is added to the filtrate in order to prevent hydrolysis of salt. The filtrate in the china dish is heated with constant stirring till crystallization point. When the crystallizationw point is reached , the china dish is removed from the flame and placed over a beaker full of water for cooling.After cooling ,crystals are separated from mother liquor by decantation and dried by spreading over a layer of filter paper to retain their shape.The crystals are then kept in a desiccator for some time.

Result

1. Colour of crystal_____.
2. Shape of crystal _____.

PRECAUTIONS

- a. The solution of copper sulphate prepared should be the saturated solution.
- b. The solution should be slightly acidic otherwise the salt may get hydrolysed.
- c. The filtrate should not be heated beyond the crystallization point.
- d. The crystals should never be dried by heating.

EXPERIMENT NO.- 04(A)

AIM OF THE EXPERIMENT:

To determine the strength of supplied acid solution using a standard alkali solution.

OBJECTIVES OF THE EXPERIMENT:

At the end of this experiment, the students will be able to

- a) Perform the different types of titration by using different types of indicators.
- b) Calculate the strength of the solution.
- c) Acquire knowledge about concentration terms like Normality, Molarity etc.

THEORY:

A known volume of standard alkali is titrated against the supplied acid solution of unknown strength in presence of methyl orange indicator till the colour changes from yellow to light pink. Then volume of acid solution is determined. By knowing the volume of both the solutions and strength of acid solution can be determined by using the principle of equivalence. $N_1 \times V_1 = N_2 \times V_2$.

APPARATUS REQUIRED:

The required apparatus are:

- a) Burette
- b) Burette stand
- c) Pipette(10 ml)-1 no. ,
- d) Conical flask(250 ml)-1no.,
- e) Wash bottle
- f. Beaker (500ml)-2nos.
- g. Glazed tile

CHEMICAL REQUIRED:

- a) Acid solution (unknown strength)
- b) Alkali solution (1.02 N/10)

PROCEDURE

At first the burette, pipette, conical flask are washed thrice with tap water. The burette is rinsed with supplied acid solution and the washings are rejected. Then the burette is filled with the acid solution to a convenient level without air bubbles and is clamped in the burette stand. The pipette is rinsed with supplied alkali solution and the washings are rejected. 10 ml of the alkali solution is pipetted out into the conical flask. 1-2 drops of methyl orange indicator is added to the flask. Initial Burette reading (I.B.R) is noted down. Then titration is carried out till the yellow colour just changed to pink. The process is repeated to get at least three concordant readings.

OBSERVATION TABLE

| No. of Observation | Volume of Alkali taken(ml) | Initial Burette Reading (I.B.R) (ml) | Final Burette Reading (F.B.R) (ml) | Difference (ml) | Remark |
|--------------------|----------------------------|--------------------------------------|------------------------------------|-----------------|------------------|
| 1 | 10 | X_1 | X_2 | X_R | Rough Value |
| 2 | 10 | X_2 | X_3 | X_C | Concordant value |
| 3 | 10 | X_3 | X_4 | X_C | |
| 4 | 10 | X_4 | X_5 | X_C | |

Calculation

The volume of acid solution consumed = $V_1 = X_C$ ml

Strength of the acid solution = $N_1 = ?$

Volume of the alkali solution = $V_2 = 10$ ml

Strength of the alkali solution = $N_2 = 1.02 \frac{N}{10}$

Applying the principle of equivalence

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

$$N_1 = \frac{N_2 \times V_2}{V_1} = \frac{1.02 \frac{N}{10} \times 10}{X_C} = Y \frac{N}{10}$$

Conclusion

The strength of acid solution is found to be $Y \frac{N}{10}$.

EXPERIMENT NO.-4(B)

AIM OF THE EXPERIMENT:

To determine the strength of supplied alkali solution using a standard acid solution.

OBJECTIVES OF THE EXPERIMENT:

At the end of this experiment, the students will be able to

- d) Perform the different types of titration by using different types of indicators.
- e) Calculate the strength of the solution.
- f) Acquire knowledge about concentration terms like Normality, Molarity etc.

THEORY:

A known volume of standard alkali is titrated against the supplied acid solution of unknown strength in presence of methyl orange indicator till the colour changes from yellow to light pink. Then volume of acid solution is determined. By knowing the volume of both the solutions and strength of acid solution can be determined by using the principle of equivalence i.e.,

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

APPARATUS REQUIRED:

Required apparatus are:

- b) Burette , b) Burette stand, c) Pipette(10 ml)-1 no. , d) Conical flask(250 ml)-1no., e) Wash bottle, f. Beaker (500ml)- 2nos., g. Glazed tile

CHEMICALS REQUIRED:

Required chemicals are:

- b) Acid solution (unknown strength) b) Alkali solution (1.02 N/10)

PROCEDURE

At first the burette, pipette, conical flask are washed thrice with tap water. The burette is rinsed with supplied acid solution and the washings are rejected. Then the burette is filled with the acid solution to a convenient level without air bubbles and is clamped in the burette stand. The pipette is rinsed with supplied alkali solution and the washings are rejected. 10 ml of the alkali solution is pipetted out into the conical flask. 1-2 drops of methyl orange indicator is added to

the flask. Initial Burette reading (I.B.R) is noted down. Then titration is carried out till the yellow colour just changed to pink. The process is repeated to get at least three concordant readings.

OBSERVATION TABLE

| No. of Observation | Volume of Alkali taken(ml) | Initial Burette Reading (I.B.R) (ml) | Final Burette Reading (F.B.R) (ml) | Difference (ml) | Remark |
|--------------------|----------------------------|--------------------------------------|------------------------------------|-----------------|------------------|
| 1 | 10 | X_1 | X_2 | X_R | Rough Value |
| 2 | 10 | X_2 | X_3 | X_C | Concordant value |
| 3 | 10 | X_3 | X_4 | X_C | |
| 4 | 10 | X_4 | X_5 | X_C | |

Calculation:

The volume of acid solution consumed = $V_1 = X_C$ ml

Strength of the acid solution = $N_1 = 1.01 \frac{N}{10}$

Volume of the alkali solution = $V_2 = 10$ ml

Strength of the alkali solution = $N_2 = ?$

Applying the principle of equivalence

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

$$N_1 = \frac{N_1 \times V_1}{V_2} = \frac{1.01 \frac{N}{10} \times 10}{X_C} = Y \frac{N}{10}$$

Conclusion:

The strength of acid solution is found to be $Y \frac{N}{10}$.

EXPERIMENT NO.- 5 to 9

TEST FOR ACID RADICALS

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|---|---|-------------|--|-----------|---|
| (1) Test for Carbonate (CO_3^{2-}) | | | | | |
| (i) | 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. | (i) | Effervescence takes place with the evolution of a colourless, odourless gas. | (i) | It may be CO_2 from CO_3^{2-} . |
| (ii) | Warm the above reaction mixture to get more gas and pass the gas slowly through lime water. | (ii) | First lime water turns milky and with excess of the gas milkyness disappears but this gas fails to change orange colour of $\text{K}_2\text{Cr}_2\text{O}_7$ solution green. | (ii) | CO_3^{2-} is confirmed. |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|---|---|-------------|--|-----------|---|
| (2) Test for Sulphide (S^{2-}) | | | | | |
| (i) | 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. | (i) | Effervescence takes place With the evolution of colourless gas with rotten egg smell | (i) | It may be H_2S gas from S^{2-} . |
| (ii) | Warm the above reaction mixture and show a filter black paper soaked with lead acetate solution to the evolved gas. | (ii) | The filter paper turns black | (ii) | S^{2-} is confirmed. |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|------------|--|-------------|--|-----------|--|
|------------|--|-------------|--|-----------|--|

| (3) Test for Chloride(Cl⁻) | | | | | |
|---|---|-------|--|-------|---|
| (i) | Take a pinch of the salt in a clean and dry test tube and add 2-3 drops of conc. H ₂ SO ₄ to it. | (i) | Effervescence takes place with the evolution of colourless gas which fumes in moist air. | (i) | It may be HCl from Cl ⁻ . |
| (ii) | Warm the above reaction mixture and show a glass rod dipped in conc. NH ₄ OH to the mouth of the test tube. | (ii) | Dense white fumes are produced and white solid deposited on the tip of the glass rod. | (ii) | It is due to the formation of NH ₄ Cl. Cl ⁻ may present. |
| (iii) | Take a pinch of the salt in a clean and dry test tube. Add a little MnO ₂ and 2-3 drops of conc. H ₂ SO ₄ . Heat the reaction mixture. | (iii) | Greenish yellow gas is evolved which turns filter paper soaked in starch iodide solution blue. | (iii) | Chlorine gas comes out from chloride which liberates iodine from iodide. Cl ⁻ may present. |
| (iv) | Take 1-2 ml of salt solution. Acidify it with 1-2 ml. of dil. HNO ₃ . Add few drops of AgNO ₃ solution to it. | (iv) | A curdy white precipitate is formed. | (iv) | It is due to the formation of AgCl. Cl ⁻ may present. |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|---|--|--------------------|---|------------------|---|
| (4) Test for Nitrate(NO₃⁻) | | | | | |
| (i) | Take a pinch of the salt in a clean and dry test tube. Add few pieces of copper turnings and 4-5 drops of conc. H ₂ SO ₄ and heat it. | (i) | Copious brown fumes are evolved and the solution turns green or bluish green. | (i) | Brown fume is due to NO ₂ from nitrate (NO ₃ ⁻) salt. |
| (ii) | <u><i>Brown-ring Test</i></u> Take 1-2ml of the salt solution. Add equal volume of conc. H ₂ SO ₄ slowly into the test tube. Cool the test tube perfectly under tap water. Then slowly add 2-3 ml of freshly prepared ferrous sulphate solution through the sides of the test tube. | (ii) | A brown ring is formed at the junction of two liquid layers. | (ii) | NO ₃ ⁻ is confirmed. |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|-------------------|--|--------------------|--|------------------|--|
|-------------------|--|--------------------|--|------------------|--|

| (5) Test for Sulphate(SO₄²⁻) | | | |
|---|---|----------------------------------|---|
| (i) | Take about 1-2 ml of salt solution. Acidify with 1-2 ml of dil. HCl. Add about 1 ml. of BaCl ₂ solution. | A white precipitate is obtained. | SO ₄ ²⁻ is confirmed. |
| (ii) | Add about 1 ml of conc. HCl to the above solution. | The precipitate is not soluble. | SO ₄ ²⁻ is confirmed. |

TEST FOR BASIC RADICALS

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|------------------------------|---|--|---|--|--|
| Dry test tube heating | | | | | |
| | In a clean and dry test tube a pinch of the salt is heated. | (i) Water vapours condensed at the cooler part of the test tube. (ii) Decrepitation took place. (iii) Salt is volatilised and white sublimate is formed. (iv) Salt is first melted but finally infusible white mass left. (v) Salt is fused on heating and solidified on cooling. (vi) The colour of the salt is changed. a. Yellow when hot & white when cold. b. Yellow when hot and cold. c. Yellowish brown in hot and yellow when cold d. Black residue. | (i) (ii) (iii) (iv) (v) (vi) | Salt with water of crystallisation. May be crystalline salt. May be Volatile salt of NH ₄ ⁺ , As ³⁺ and Hg ²⁺ . May be Mg ²⁺ , Al ³⁺ , Zn ²⁺ , Ca ²⁺ , Sr ²⁺ , Ba ²⁺ etc. May be alkali or alkaline earth metal salt (Na ⁺ , K ⁺ etc.) May be salt of Pb ²⁺ , Bi ²⁺ , Sn ²⁺ etc. The salt is non-volatile. a. May be Zn ²⁺ salt. b. May be Pb ²⁺ salt. c. May be Sn ²⁺ and Bi ³⁺ salt d. May be Cu ²⁺ , Ni ²⁺ , Mn ²⁺ and Fe ²⁺ . | |
| | | | | | |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|--------------------------------------|--|--------------------|--|------------------|---|
| Heating in a charcoal cavity | | | | | |
| (i) | A pinch of the salt is taken in a charcoal cavity and is heated in oxidising flame with a blow pipe. | (i) | The salt is completely volatilised. | (i) | May be NH_4^+ , As^{3+} , Hg^{2+} (Sodalime test is to be performed) |
| | | (ii) | An infusible incandescent white mass is obtained. | (ii) | May be Mg^{2+} , Al^{3+} , Zn^{2+} , Ca^{2+} , Ba^{2+} , Sr^{2+} , Sn^{2+} . (Cobalt nitrate test is to be performed). |
| | | (iii) | The salt fused and sank into the charcoal cavity and reappeared on cooling. | (iii) | May be alkali or alkaline earth metal salt like Na^+ , K^+ . (Flame test is to be performed.) |
| | | (iv) | Original salt is white and formed a coloured mass. | (iv) | May be Pb^{2+} , Bi^{3+} , Sn^{2+} , Ag^+ etc. (Reduction test is to be performed) |
| | | (v) | Original salt is coloured and is formed a coloured mass. | (v) | May be Cr^{3+} , Mn^{2+} , Ag^+ etc. Co^{2+} , Ni^{2+} , Cu^{2+} . (Borax bead test is to be performed) |
| EXPERIMENT | | OBSERVATION | | INFERENCE | |
| Sodalime test (Volatile Salt) | | | | | |
| (i) | A pinch of salt is taken in a watch glass. A little sodalime is added with a drop of water. Then it is rubbed. | (i) | Ammonia gas is evolved and the colour of the mixture is not changed. | (i) | May be NH_4^+ , |
| | | (ii) | Only colour of the residue is changed to brown and there is no evolution of gas. | (ii) | May be NH_4^+ , |
| | | (iii) | No gas is evolved and no change in colour of residue. | (iii) | May be As^{3+} |

| EXPERIMENT | OBSERVATION | | INFERENCE |
|--|-------------|---|--------------------------------|
| 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 shining mirror is formed | (i) May be Hg^{2+} . |
| | (ii) | A black shining mirror is formed with the evolution of a gas having garlic odour. | (ii) May be As^{3+} . |

| EXPERIMENT | OBSERVATION | | INFERENCE |
|---|-------------|-------------------|---|
| Cobalt Nitrate Test (For infusible salt) | | | |
| A pinch of salt is taken in a charcoal cavity. It is heated in an oxidising flame till an infusible mass is obtained. A drop of cobalt nitrate solution is added and again heated strongly. | (i) | Blue mass. | (i) May be Al^{3+} . |
| | (ii) | Green mass. | (ii) May be Zn^{2+} . |
| | (iii) | Pink mass.. | (iii) May be Mg^{2+} . |
| | (iv) | Dirty green mass. | (iv) May be Sn^{2+} . |
| | (v) | Grey mass. | (v) May be $\text{Ca}^{2+}, \text{Sr}^{2+}, \text{Ba}^{2+}$ (Flame test to be performed) |

| EXPERIMENT | OBSERVATION | | INFERENCE |
|--|-------------|---------------------------------|---|
| Flame Test (For fusible salt) | | | |
| A clean nichrome wire is moisten with conc. HCl . A pinch of a salt is taken by touching this wire to the salt. Then it is shown to the oxidizing flame. The colour of the flame is observed through naked eye and through double blue glass. | | Colour through naked eye | Colour through double blue glass |
| | | (i) Persistent | (i) Colourless |
| | | (ii) golden | (ii) Red |
| | | (iii) yellow | (iii) Light yellow |
| | | (iv) Violet | (iv) Red |
| | | (v) Brick red | (v) Green |
| (vi) Crimson red | (vi) — | | |
| Pea green Green flame with blue centre. | | | |
| | (i) | (i) | (i) May be Na^+ . |
| | (ii) | (ii) | (ii) May be K^+ . |
| | (iii) | (iii) | (iii) May be Ca^{2+} . |
| | (iv) | (iv) | (iv) May be Sr^{2+} . |
| | (v) | (v) | (v) May be Ba^{2+} . |
| (vi) | (vi) | (vi) May be Cu^{2+} . | |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|--|-------|--|-------|---------------------------|--|
| 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 cavity and is heated in a reducing flame. | (i) | White shining malleable bead without incrustation which did not mark on paper. | (i) | May be Ag ⁺ . | |
| | (ii) | White shining malleable bead with lemon yellow incrustation which marked on paper. | (ii) | May be Pb ²⁺ . | |
| | (iii) | Brittle bead with lemon yellow incrustation. | (iii) | May be Bi ³⁺ . | |
| | (iv) | Malleable white bead. | (iv) | May be Sn ²⁺ . | |
| | (v) | Red scales without incrustation. | (v) | May be Cu ²⁺ . | |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|-------------------------|---|-------------|--|-----------|--|
| Group Separation | | | | | |
| (i) | About 2 ml of salt solution is taken in a test tube and | (i) | (a) White ppt. is formed (b) No white ppt. is formed. | (i) | (a) Gr. I radical may be present. (b) Gr. I radicals are absent. |
| (ii) | little dil. HCl is added. The above solution is warmed and H ₂ S gas is passed through it | (ii) | (a) A coloured precipitate is formed. (b) No precipitation formed | (ii) | (a) Gr. II radicals may be present. (b) Gr. II radicals are absent. |
| (iii) | About 2 ml. of the salt solution is taken in a test tube. Solid NH ₄ Cl is added till saturation and dil. NH ₄ OH till faintly ammoniacal. | (iii) | (a) A precipitate is formed. (b) No precipitation formed | (iii) | (a) Gr. IIIA radicals may be present. (b) Gr. IIIA radicals are absent. |
| (iv) | H ₂ S gas is passed through above solution. | (iv) | (a) A coloured ppt. is formed. (b) No ppt. is formed. | (iv) | Gr. IIIA radicals may be present. Gr. IIIB radicals are absent. |
| (v) | About 2ml. of salt solution is taken with solid NH ₄ Cl and dil. NH ₄ OH till ammoniacal. Then a saturated solution of ammonium carbonate is added. | (v) | (a) A white ppt. is formed. (b) No white ppt. | (v) | (a) Gr. IV radicals may be present. (b) Gr. IV radicals are absent. |

| EXPERIMENT | OBSERVATION | | INFERENCE |
|---|-------------|---|---|
| TEST FOR GR-I RADICALS (Pb²⁺, Ag⁺, Hg₂²⁺), Group reagent –dil. HCl | | | |
| About 2 ml. of salt solution is taken in a test tube and treated with a little dil. HCl. The white ppt. is washed with distilled water and divided into two parts. Further distilled water is added with washing. | (i) | A white ppt. is formed. (a)The ppt. is dissolved on heating and reappeared on cooling. (b)The ppt. is not soluble in hot condition. | (i) Group-I radical Pb ²⁺ , Ag ⁺ or Hg ₂ ²⁺ . (a)May be Pb ²⁺ . (b)Pb ²⁺ is absent. |
| | (ii) | (a)The ppt. is soluble and reappeared on acidified with dil.HNO ₃ . (b)The precipitate is turned black. | (ii) (a)May be Ag ⁺ . (b)May be Hg ₂ ²⁺ . |

| EXPERIMENT | OBSERVATION | | INFERENCE |
|---|-------------|---|---|
| TEST FOR GR-IIA RADICALS (Pb²⁺, Hg²⁺, Cu²⁺, Bi³⁺) & GR-IIB(As³⁺, Sb³⁺, Sn²⁺), Group reagent- dil.HCl and H₂S | | | |
| About 2 ml. of salt solution is taken and a little of HCl is added obtained. Then it is warmed and finally H ₂ S gas is passed through it. | (i) | Black precipitate is obtained | (i) May be Pb ²⁺ or Hg ²⁺ or Cu ²⁺ . |
| | (ii) | Dark brown precipitate is obtained. | (ii) May be Bi ³⁺ . |
| | (iii) | Yellow ppt. is obtained. | (iii) May be As ³⁺ . |
| | (iv) | Orange ppt. is obtained. Dirty yellow or light brown ppt. is obtained. | (iv) May Sb ³⁺ . May be Sn ³⁺ . |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|---|--|-------------|------------------------------------|-----------|---------------------------|
| TEST FOR GR-III A RADICALS (Fe^{3+}, Al^{3+}, Cr^{3+}), Group reagent- Solid NH_4Cl and NH_4OH | | | | | |
| | To about 2 ml. of salt solution NH_4Cl is added till saturation and then dii. NH_4OH is added to alkanline. till alkaline. | (i) | Reddish brown ppt is obtained. | (i) | Maybe Fe^{3+} . |
| | | (ii) | Gelatinous white ppt. is obtained. | (ii) | Maybe Al^{3+} . |
| | | (iii) | Greenish white ppt. is obtained. | (iii) | May be Cr^{3+} . |

| EXPERIMENT | | OBSERVATION | | INFERENCE | |
|---|--|-------------|---|-----------|--------------------------------|
| Confirmatory Test for Aluminium(Al^{3+}) | | | | | |
| | To about 2 ml. of salt solution NaOH solution is added drop by drop and then in excess. | | Gelatinous white ppt. is formed.(white ppt. is soluble in excess NaOH). | | Al^{3+} is confirmed. |

| EXPERIMENT | OBSERVATION | | | INFERENCE | |
|--|--|-------|-------------------------|-----------|---------------------------------|
| TEST FOR GR-IIIB RADICALS (Zn^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}), Group reagent- Solid NH_4Cl, NH_4OH and H_2S | | | | | |
| | To about 2 ml. salt solution, solid NH_4Cl is added till saturation and then dil. NH_4OH is added till alkaline. Then H_2S gas is passed through it. | (i) | White ppt. is obtained. | (i) | May be Zn^{2+} . |
| | | (ii) | Buff coloured ppt. | (ii) | May be Mn^{2+} . |
| | | (iii) | Black ppt. is obtained. | (iii) | May be Co^{2+} or Ni^{2+} . |

| EXPERIMENT | OBSERVATION | | | INFERENCE | |
|---|--|--|--|-----------|-------------------------|
| Confirmatory Test for Zinc(Zn^{2+}) | | | | | |
| | To about 2 ml. of salt solution, dil. $NaOH$ is added drop by drop and then in excess. | | White ppt. is formed. (white ppt. is soluble in excess $NaOH$). | | Zn^{2+} is confirmed. |

| EXPERIMENT | OBSERVATION | | | INFERENCE | |
|--|--|--|-------------------------|-----------|--|
| TEST FOR GR-IV RADICALS (Sr^{2+}, Ba^{2+}, Ca^{2+}), Group reagent- Solid NH_4Cl, NH_4OH and $(NH_4)_2CO_3$ | | | | | |
| | To about 2 ml. salt solution, solid NH_4Cl is added till saturation and then dil. NH_4OH is added till alkaline. Then saturated solution of $(NH_4)_2CO_3$ is added. | | White ppt. is obtained. | | Gr.IV radicals (Sr^{2+} , Ba^{2+} , Ca^{2+}) may be present. |

| EXPERIMENT | OBSERVATION | INFERENCE |
|---|-----------------------|--------------------------------|
| Confirmatory Test for Calcium(Ca²⁺) | | |
| To about 2 ml. salt solution, solid NH ₄ Cl is added till saturation and then dil.NH ₄ OH is added till alkaline. Then saturated solution of (NH ₄) ₂ CO ₃ is added. The white ppt. obtained is dissolved in acetic acid and divided into three parts. Take one part of solution in a test tube and add ammonium oxalate solution followed by dil.NH ₄ OH. | White ppt. is formed. | Ca ²⁺ is confirmed. |

| EXPERIMENT | OBSERVATION | INFERENCE |
|---|---|-------------------------------|
| TEST FOR GR-V RADICALS (Na⁺, K⁺, Mg²⁺, NH₄⁺) | | |
| Confirmatory Test for (Na⁺) | | |
| To about 2 ml. of salt solution potassium pyroantimonate solution (K ₂ H ₂ Sb ₂ O ₇) is added. colour in flame test. | White ppt. is obtained, which gave persistent golden yellow colour in flame test. | Na ⁺ is confirmed. |

| EXPERIMENT | OBSERVATION | INFERENCE |
|---|---|------------------------------|
| Confirmatory Test for Potassium (K⁺) | | |
| To about 2ml. of salt solution a few drops of cobalt nitrate solution is added followed by addition of solid NaNO ₂ , and dil. CH ₃ COOH. Then it is allowed to stand for five minutes. | Yellow ppt. of potassium cobaltinitrite is obtained, which gave violet purple colour in flame test. | K ⁺ is confirmed. |

| EXPERIMENT | OBSERVATION | INFERENCE |
|--|-------------------------|--------------------------------|
| Confirmatory Test for Sodium (Mg²⁺) | | |
| To about 2 ml. of salt solution solid NH ₄ Cl is added till saturation and dil. NH ₄ OH is added till ammoniacal. Then disodium-hydrogen phosphate is added. | White ppt. is obtained. | Mg ²⁺ is confirmed. |

| EXPERIMENT | OBSERVATION | INFERENCE |
|--|-------------------------|--|
| Confirmatory Test for Ammonium (NH₄⁺) | | |
| To about 2 ml. of salt solution few drops of NaOH was added followed by a little Nessler's reagent. A brown ppt. is obtained. Then | Brown ppt. is obtained. | NH ₄ ⁺ is confirmed. |

Conclusion:

Acid radical of the salt detected to be _____.

Basic radical of the salt detected to be _____.

Hence, the unknown supplied salt is detected to be _____.