Engineering
CHEMISTRY
LABORATORY MANUAL

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CERTIFICATE

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Lecturer Principal

CONTENTS:

EXPERIMENT NO. 1

Preparation of carbon dioxide gas

OBJECTIVE OF THE EXPERIMENT:

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

a) Specify the chemicals, which are being used for preparation of carbon dioxide gas in the laboratory.

b) Know the physical and chemical properties of the gas.

1. AIM OF THE EXPERIMENT: To prepare carbon dioxide gas in the laboratory and study its properties.

2. APPARATUS REQUIRED:

a) Woulf's bottle
b) Thistle funnel
c) Delivery tube
d) Cork
e) Gas jar with lid
f) Test tubes

3. CHEMICALS REQUIRED

a) Marble chips
b) Dilute Hydrochloric acid (HCl)
c) Litmus Paper
d) Magnesium ribbon
e) Lime water

4.THEORY: Carbon dioxide gas is prepared in the laboratory by the action of dilute hydrochloric acid on marble ($CaCO_3$).

5. PROCEDURE

(i) Take a Woulf's bottle fitted with cork, thistle funnel and delivery tube.
(ii) Puts some marble pieces into the woulf's bottle.
(iii)Pour some water into woulf's bottle through the thistle funnel such that its lower end dips under water and marble pieces covered by it.

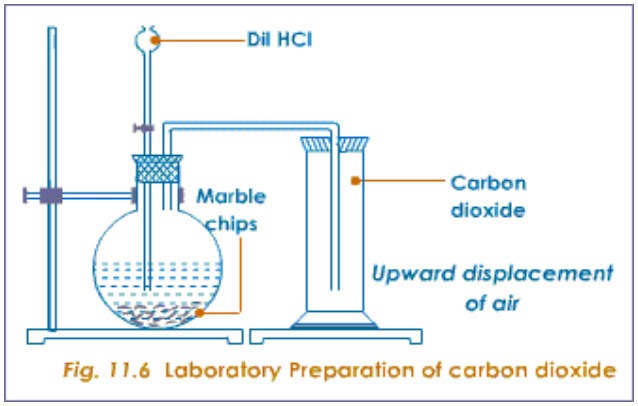


Fig. 11.6 Laboratory Preparation of carbon dioxide

(Preparation of CO_2 gas)

(iv)Now pour some dilute hydrochloric acid down the thistle funnel.
(v)Allow the gas to escape for some time so that the air is driven out.
(vi)Collect the carbon dioxide gas in the gas jar by upward displacement of the air. Test the gas collected in the gas jar by showing a burning splinter at the mouth of the gas jar.
(vii)Study the properties of CO_2 by collecting the gas by in different test tubes.

6 . PHYSICAL PROPERTIES

Experiment	Observation	Inference
(i) Color of the gas Note the color of the gas (ii) Odour of the gas Note the odour of the gas (iii) Solubility Invert the gas jar in a through of water (iv)Density Place a gas jar inverted over an empty jar		

7. CHEMICAL PROPERTIES

(i)Introduce a burning splinter into the gas jar (ii)Action towards litmus A moist blue litmus paper is shown to the gas (iii)a-pass the gas coming out of the delivery tube into lime water taken in a test tube b-pass the gas in excess (iv)magnesium ribbon test Introduce a burning magnesium ribbon into a gas jar containing CO_2 gas (v)Add few drops of water into the gas jar and shake it and put a red litmus paper into the gas jar		

7. PRECAUTIONS:

1. Apparatus should be air tight.
2. Thistle funnel must dip inside the acid.
3. Acid should be added a little at regular interval.

EXPERIMENT NO-2

Preparation of ammonia gas

OBJECTIVE OF THE EXPERIMENT:

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

a) Specify the chemicals, which are being used for preparation of carbon dioxide gas inthe laboratory.
b) Know the physical and chemical properties of the gas.

1.AIM OF THE EXPERIMENTS: To prepare and study the physical and chemical properties of ammonia gas in the laboratory.

2. APPARATUS REQUIRED:

a) Rubber Cork
b) Hard glass test tube
c) Gas jar
d) Clamp stand
e) Delivery tube
f) Test tubes

3. CHEMICALS REQUIRED:

a) Solid ammonium chloride (NH_4Cl)
b) Slaked lime $Ca(OH)_2$
c) Litmus Paper
d) Conc. HCl
e) Nessler's reagent
f) Ferric chloride
g) $CuSO_4$solution

4. THEORY:

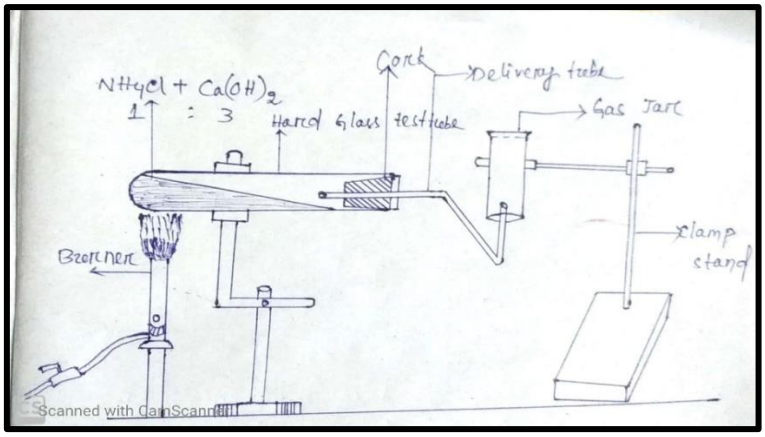
Ammonia gas is prepared in the laboratory by a mixture of solid ammonium chloride and slaked lime in 1:3 ratio. The gas is collected by the downward displacements of air as it is lighter than air.

CHEMICAL EQUATION:

$2NH_4CL + Ca(OH)_2 \rightarrow CaCl_2 + NH_3 + 2H_2O$

5. PROCEDURE:

(i)Take ammonium chloride and slaked limein1:3 ratio in a mortar and mix thoroughly.
(ii) Take the mixture in a hard glass test tube to the half of the test tube.
(iii) Fit the cork with delivery tube in to the mouth of the test tube and clamp the hard glass test tube into the clamp stand.
(iv)Heat the hard glass test tube continuously.
(v)Allow the gas to escape for some time so that the air is driven out.
(vi)Collect the NH3 gas in the gas jar by downward displacement of the air.
(vii)Study the properties of NH_3 by collecting the gas by in different test tubes.



(Preparation of NH_3 gas)

6. PHYSICAL PROPERTIES

Experiment	Observation	Inference
(i)Color of the gas Note the color of the gas (ii)Odour of the gas Note the odour of the gas (iii) Solubility Invert the gas jar in a through of water		

7. CHEMICAL PROPERTIES

Experiment	Observation	Inference
(i)Introduce a burning splinter into the gas jar (ii)Action towards litmus A moist red litmus paper is shown to the gas (iii)Dip a glass rod Conc. HCl and show to the NH_3 gas (iv)Pass theNH3 gas coming out of the delivery tube into Nessler's reagent taken in a clean dry test tube. (v) Pass the NH_3 gas coming out of the delivery tube into 2ml of ferric chloride solution. (vi) Pass theNH_3 gas into 2ml of aque-ous$CuSO_4$ solution slowly and then in excess.		

7. PRECAUTIONS:

(i) Apparatus must be made by proper air tight.
(ii) Heat should be provided uniformly.
(iii) The hard glass test tube should be fixed in inclined position towards it mouth in order to prevent crake on it.
(iv) Gas jar should be dried.

EXPERIMENT NO-3

Preparation of Copper Sulphate Crystal from Copper Carbonate

1. AIM OF THE EXPERIMENTS: To prepare copper sulphate crystals from copper carbonate.

2. APPARATUS REQUIRED:

a) Beaker
b) Glass rod
c) Tripod stand
d) Wire gauze
e) Bunsen burner
f) Filter stand
g) Filter paper
h) Porcelain basin

3. CHEMICALS REQUIRED:

a) Copper carbonate (CuCO3)
b) Dilute H2SO4

4. THEORY:

When Copper carbonate (CuCO3) reacts with Dilute H2SO4, soluble Copper Sulphate if formed. Then the Copper Sulphate solution is heated till the crystallization point is reached. On cooling the resulting solution, the crystal of Copper Sulphate separate out.

CHEMICAL EQUATION:

$CuCO_3 + dil.H_2SO_4 \rightarrow CuSO_4 + CO_2 + H_2O$

5. PROCEDURE:

Preparation of Solution

(i)Take 25ml of dil.H2SO4in a beaker.
(ii) Add CuCO3 gradually to dilute H2SO4.
(iii) Addition of CuCO3 is continued till a little quantities of CuCO3 is left behind.
(iv)Heat the resulting solution to remove CO2.
(v)Filter the solution into a porcelain basin
(vi)Add a few drops of dil.H2SO4 to the filtrate to check hydrolysis of salt

Concentrating the Filtrate

(i)Evaporate the filtrate in the basin with constant stirring.
(ii) The process of evaporation is continued till a drop of the solution forms crystal on the glass rod, when blown on it.

Crystallization

(i)The filtrate of hot saturated solution is cool down slowly to start the process of crystallization.
(ii)For getting better quality of crystal, a crystal of CuSO4 is added so that it finds a place in the middle of the solution. If the beaker undisturbed and let it cool.

Filtration and drying of crystal

(i) When the process of crystallization is over decant the mother liquor.
(ii)The crystals of CuSO4 are then washed with cooled water.
(iii) Dry the crystal keeping those in between two folds of filter paper.
(iv) Submit required quantity of CuSO4 crystal.

6. PRECAUTIONS:

a) Minimum point of dil.H2SO4 should be used to prepare the solution.
b) The solution should be slightly acidic otherwise the salt may get hydrolyzed.
c) The solution should not be heated beyond its crystallization point.
d) The crystal should never be dried by heating.

EXPERIMENT NO- 4

SIMPLE ACID BASE TITRATION

4(a).ACIDIMATRY

AIM OF THE EXPERIMENT: To find out the strength of acid by using a standard alkali solution of strength 1.0 (N/10) in the laboratory.

APPARATUS REQUIRED:

a) Burette (50ml)-1no.
b) Pipette (10ml)-1no
c) Conical flask (250ml)-1no.
d) Beaker (500ml)-2no.
e) Wash bottle-1no
f) Burette stand with clamp-1 set
g) Funnel-2no.

CHEMICALS REQUIRED:

a) Sodium carbonate solution (Na_2CO_3)-Alkali solution.
b) Sulphuric acid solution (H_2SO_4)

T H E O R Y :

The principle of Acidimetry is

VA.SA=VB.SB

Where VA=Volume of acid used (Burette reading)
SA=Strength of acid (Unknown)
VB= Volume of alkali used (Pipette reading)
SB= Strength of alkali (standard solution)

PROCEDURE:

1. Clean the apparatus with water.
2. Wash the burette thoroughly with water then rinse with a little amount of acid.
3. Fill the burette with acid solution to a little above the ‘‘zero mark’’. Open the stopcock for a moment in order to fill the jet with the acid that no air bubble will remain in the burette. Then clamp the burette vertically to the burette stand.
4. Take a clean pipette of 10ml capacity. Rinse the pipette with the standard alkali solution thrice.
5. Suck the alkali solution in to the pipette just a little above the mark. Close the upper open end immediately with index figure firmly. Wipe out the adhering liquid from the

outside of lower stem with filter paper. Now release the index figure slightly and transfer the alkali in to a conical flask slowly but continuously. Touch the tip of the stem thrice slowly with the bottom of the flask.

6. Add one drop of ethyl orange indicator to the alkali solution and shake well. The color of the solution becomes straw yellow.
7. Now place the conical flask containing alkali on the white glazed tile below the burette. Note down the initial reading of the burette.
8. Slowly add the acid solution from the burette to the alkali solution in the conical flask until the color of the solution becomes pale yellow.
9. Continue the addition of the acid solution drop wise while swirling the solution in the flask continuously. Stop adding acid at the point when the color of the solution just changes in to light pink. This the end point. Note down final burette reading (F.B.R). This will be the rough reading.
10. 10.Repeat the process of addition of acid solution to the alkali solution thrice. All the three readings should be concordant.

OBSERVATION:

No. of observation	Volume of alkali in ml.	Initial burette reading in ml.	Final burette reading in ml.	Difference in ml.	Mean in ml.	Remark
1	10					
2	10					
3	10					
4	10					

CALCULATION:

We know that VA.SA=VB.SB
Where VA=Burette reading (diff in ml)
VB= Pipette reading (volume of alkali)
SB= 0.1 N or N/10
SA=???
Thus SA= VB.SB/VA (N/10)

CONCLUSION: From the above titration, the strength of unknown acid solution is found to be ---------- (N/10).

Precautions

1. The air bubbles in the nozzle of the burette must be removed before taking the initial reading.
2. To take the correct burette reading, use anti parallax card.
3. Alkali should be taken in a conical flask and acid in the burette, because if we take acid in the conical flask, during pipetting out of the acid, it may enter into the mouth thus by causing injury.
4. The small amount of alkali which remains inside the pipette during transferring the solution from pipette to conical flask, should not be blown in to the conical flask.
5. Indicator should not be added in excess.

6. The conical flask should always be placed under the burette on a white glazed tile.
7. Acid must be added to the alkali drop by drop as the end point approaches.
8. The solution in the conical flask should be continuously shaken while acid is added to alkali from the burette.

4(b).ALKALIMATRY

AIM OF THE EXPERIMENT: To find out the strength of alkali by using a standard acid solution of strength 1.01(N/10) in the laboratory.

APPARATUS REQUIRED:

1. Burette (50ml)-1no.
2. Pipette (10ml)-1no
3. Conical flask (250ml)-1no.
4. Beaker (500ml)-2no.
5. Wash bottle-1no
6. Burette stands with clamp-1 set
7. Funnel-2no.

CHEMICAL REQUIRED:

1) Sodium carbonate solution(Na_2CO_3)-Alkali solution.
2) Sulphuric acid solution (H_2SO_4)-Acid solution.

Theory:

The principle of Alkalimetry is

$V_A.S_A=V_B.S_B$

Where V_A=Volume of acid used(Burette reading)

S_A=Strength of acid standard solution

V_B= Volume of alkali used (Pipette reading)

S_B= Strength of alkali (unknown)

PROCEDURE:

1. Clean the apparatus with water.
2. Wash the burette thoroughly with water then rinse with a little amount of acid.
3. Fill the burette with acid solution to a little above the ‘‘zero mark’’. Open the stopcock for a moment in order to fill the jet with the acid that no air bubble will remain in the burette. Then clamp the burette vertically to the burette stand.
4. Take a clean pipette of 10ml capacity. Rinse the pipette with the standard alkali solution thrice.
5. Suck the alkali solution in to the pipette just a little above the mark. Close the upper open end immediately with index figure firmly. Wipe out the adhering liquid from the outside of lower stem with filter paper. Now release the index figure slightly and transfer the alkali in to a conical flask slowly but continuously. Touch the tip of the stem thrice slowly with the bottom of the flask.
6. Add one drop of ethyl orange indicator to the alkali solution and shake well. The color of the solution becomes straw yellow.
7. Now place the conical flask containing alkali on the white glazed tile below the burette. Note down the initial reading of the burette.
8. Slowly add the acid solution from the burette to the alkali solution in the conical flask until the color of the solution becomes pale yellow.

9. Continue the addition of the acid solution drop wise while swirling the solution in the flask continuously. Stop adding acid at the point when the color of the solution just changes in to light pink. This the end point. Note down final burette reading (F.B.R). This will be the rough reading.
   Repeat the process of addition of acid solution to the alkali solution thrice. All the three readings should be concordant.

OBSERVATION:

No. of obs	Volume of alkali in ml	Initial burette reading in ml	Final burette reading in ml	Difference in ml	Mean in ml	Remark
1	1 0					
2	1 0					
3	1 0					
4	1 0					

CALCULATION:

We know that VA.SA=VB.SB
Where VA=Burette reading (diff in ml)
VB= Pipette reading (volume of alkali)
SA= 0.1 N or N/10
SB=???
Thus SB= VA.SA/VB (N/10)

CONCLUSION: From the above titration, the strength of unknown alkali solution is found to be ---------- (N/10).

PRECAUTIONS:

a. The air bubbles in the nozzle of the burette must be removed before taking the initial reading.
b. To take the correct burette reading, use anti parallax card.
c. Alkali should be taken in a conical flask and acid in the burette, because if we take acid in the conical flask, during pipetting out of the acid, it may enter into the mouth thus by causing injury.
d. The small amount of alkali which remains inside the pipette during transferring the solution from pipette to conical flask, should not be blown in to the conical flask.
e. Indicator should not be added in excess.
f. The conical flask should always be placed under the burette on a white glazed tile.
g. Acid must be added to the alkali drop by drop as the end point approaches.
h. The solution in the conical flask should be continuously shaken while acid is added to alkali from the burette.

EXPERIMENT NO- 5

Test for acid radical (known)

STUDY OF PHYSICAL PROPERTIES OF SALT

Experiment	Observation	Inference
(a)Note the color	Colorless or white	Most of Na+,K+,Mg2+,Ca2+,Al3+,Zn2+,NH4+ salt etc.,
(b)structure	(i) crystalline	Most of the chlorides, nitrates, sulphates etc.
	(ii) amorphous	Carbonates and sulphides of Ca2+,Mg2+,Zn2+ etc.(except those of Na+, K+and NH4+)
(c)solubility	Soluble in water	Carbonates and sulphides of Na+, K+and NH4+),all halides, all nitrates, all sulphates,

TEST FOR ACID RADICAL

DRY TEST FOR ACID RADICALS

Experiment	Observation	Inference
Heat a small quantity of the supplied salt in a clean dry test tube fast slowly and then strongly for about three to four minutes.	A gas or vapour is evolved.	
	(i) A colorless and odourless gas(O2) which rekindles a glowing splinter	May be Nitrates of Na+ and K+
	(ii) A colorless and pungent smelling gas (NH3) which turns red litmus paper blue.	May be Certain ammonium salts
	(iii) A colorless and odourless gas which turns lime water milky	May be carbonates
	(iv)a colorless gas (SO2) with burning sulphur smell which turns acidified K2Cr2O7solution green	May be sulphate
	(v)a colorless gas (HCl)with irritating smell which fumes in moist air. it produces dense white fumes with a glass rod	May be hydrated chloride salt.

	dipped in conc.NH4OH (vi) a colorless gas(H2S) with rotten egg smell which turns lead acetate paper black.	May be hydrated sulphide salts.

WET TEST FOR ACID RADICALS

1.Test for carbonate (CO32-)

Experiment	Observation	Inference
(a)Take2ml of dilute HCl in a clean test tube. Warm it and add a little of the salt in to it.	Effervescence takes place with evolution of colorless &odourless gas.	May be CO2 gas from CO32-. [Na2CO3+2HCl → 2NaCl+H2O+CO2]
(b)Show a glowing splinter to the colorless & odorless gas.	The splinter extinguishes.	CO2 gas from CO32-.
(c)Pass the gas into lime water and then in excess.	Lime water turns milky on excess of gas milkyness disappears.	CO32- confirmed.

2. Test for Sulphide radical (S2-)

Experiment	Observation	Inference
(a)Take2ml of dilute HCl in a clean test tube. Warm it and add a little of the salt in to it.	Effervescence takes place with evolution of a gas having rotten egg smell.	May be **H_2S** gas from sulphide.
(b)Show lead acetate paper to the color less gas with rotten egg smell.	Lead acetate paper turns black	**PbS** is formed which is black in color. **S^{2-}** is confirmed
(c) Show a filter paper dipped in acidified KMnO4solution to the evolved gas.	KMnO4 solution get decolorized.	**S^{2-}** is confirmed.

3. Test for chloride radical

Experiment	Observation	Inference
(a)Take a pinch of the salt ina clean and dry test tube and add 2	A colorless fuming gas with pungent smell is evolved.	It may be HCl gas from Cl^{--}

drops of conc.$H2SO_4$ and warm it.		
(b)Show a glass rod dipped in conc.NH_4OH solution to the above gas.	Evolution of dense white fumes and white solid deposited on the tip of the glass rod.	It is due to the formation of NH_4Cl. Cl- may be present.
(c) Take a pinch of the salt in a clean and dry test tube. Add a little MnO_2 and 2-3 drops of conc. H2SO4and heat it.	A greenish yellow gas is evolved which turns starch io-dide paper blue.	Cl- may present.
(d)Take 1 ml of salt so-lution ina test tube. Acidified with 1 ml of dil.HNO_3 then add Ag-NO_3 solution. (e) wash the above pre-cipitated with distilled water and divide in to two parts	A curdy white ppt. is formed.	It is due to the formation of AgCl. Cl- confirmed.
(1) Add dil.HNO_3 and shake well.	Precipitate is insoluble in dil.HNO_3	AgCl is not soluble in HNO_3
(2) Add dil.NH_4OH and shake well.	Precipitate is insoluble in dil.NH_4OH.	AgCl is soluble in dil. NH_4OH due to formation of complex. Cl- is confirmed.

4. Test for Sulphate (SO_4^{2-})

Experiment	Observation	Inferences
(a) Take about 1-2 ml of salt solution. Acidify with 1-2 ml of dil. HCl. Add about 1ml of $BaCl_2$ solution. Add about 1 ml of Conc. HCl to the above solution and warm it.	A white precipitate is obtained. The precip-itate is not soluble in HCl.	SO42- is confirmed $Na_2SO_4+BaCl_2 \rightarrow$ $BaSO_4$ + 2NaCl

5. Test for Nitrate (NO^{3-})

Experiment	Observation	Inferences
(a) Take a pinch of salt in a clean and dry test tube. Add few pieces of copper turnings and 4-5 drops ofconc.H_2SO_4 and heat it.	Copious brown fumes are evolved and the solution turns green or bluish green.	Brown fume is due to NO_2 from nitrate NO_3-salt.
(b)Show a filter paper soaked in freshly prepared $FeSO_4$ solution to the above brown gas.	The paper turns black	[$Cu+4HNO_3 \rightarrow$ $Cu(NO_3)_2+2H_2O+$ $2NO_2$

(c) Brown ring test take 1-2 ml of salt solution. add equal volume of conc.H_2SO_4 slowly in to the test tube. Cool the test tube perfectly under tap. Then slowly add 2-3 ml of freshly prepared ferrous sulphate solution through the sides of the test tube.	A brown ring is formed at the junction of the two liquid layers.	May beNO_3- The brown ring is due to the formation of $[Fe(H2O)_5(NO)]SO_4$ NO_3-is confirmed

EXPERIMENT NO-6

Test for basic radical (known)

STUDY OF PHYSICAL PROPERTIES OF SALT

TEST FOR BASIC RADICAL

Dry test for basic radical

1. Dry test tube heating

Experiment	Observation	Inference
Heat a small quantity of the supplied salt in a clean dry test tube fast slowly and then strongly for about three to four minutes.	(a) water particles condense at the cooler part of the test tube (b) the salt volatilises out completely forming a white sublimate (c) the salt decrepitates (produces cracking sound) (d) The salt melts on heating and solidifies upon cooling. (e) The salt changes its color. Yellow when hot and white when cooled (f) the salt is swelled upon heating	Salt contains water of crystallization. Volatile salts. May be NH4+ salt Crystalline salts. Alkali and alkaline earth metal salts. May be zinc salt. May be Al3+.

2. Test for volatile salts(Soda lime Test)

Experiment	Observation	Inferences
(a) Take a pinch of salt in a watch glass add a little soda lime (NaOH + CaO) and few drops of water to it. Rub it with the thumb.	A colorless gas with ammonia smell is observe	Ammonium salt is present
(b) A glass rod dipped in conc. HCl is shown to the evolved gas.	Copious evolution of dense white fumes.	Ammonium salt is present

3. Charcoal cavity heating

Experiment	Observation	Inferences
Make a small cavity on a charcoal block. Take a little of the salt in the cavity and heat it strongly in oxidizing flame with a blow pipe.	(i)The salt produces cracking sound. (ii) The salt deflagrates (suddenly catches fire and burns vigorously). (iii) The salt melts and sink in to the charcoal cavity on heating and reappears on cooling (iv) the salt may or may not melt (a) A white infusible in candescent (giving light) residue is obtained. (b) Salt becomes yellow when hot, white when cooled.	May be crystalline salt. May be nitrates. May be alkali or alkaline earth metals(Ca2+,Mg2+,Na+,K+) (Flame test to be performed) May be aluminium. May be zinc salt. Performed cobalt nitrate test.

3. Cobalt Nitrate Test (for infusible salt)

Experiment	Observation	Inferences
Heat a small quantity of the salt in a charcoal cavity in the oxidizing flame with the help of a blow pipe till an infusible and in candescent residue is left. In Moisten the residue with a drop of cobalt nitrate solution and heat it in the oxidizing flame with the help of a blow pipe. Note the color of	(i) blue mass (ii) green mass (iii) Pink mass (iv) grey mass	May be Al3+salt. May be Zn2+salt. May be Mg2+salt. May be Ca2+ salt. (flame to be performed)

the residue.		

3. Flame Test (for fusible salts)

Experiment	Observation		Inferences
Clean a nichrome wire with sand paper. Dip it in conc. HCl kept in a watch glass. Show it to the flame. It should be done till no color is imparted to the flame. Moisten the nichrome wire with conc. HCl and touch it with a little of the salt. Now heat it in oxidizing flame and note the color of the flame through naked eye and through double blue glass.	Color through naked eye	Color through double blue glass	Name of the salt
	(a)Persistent golden yellow color	Colorless	Sodium (Na+) salt.
	(b)Violet crimson red	Crimson red	Potassium (K+) Salt.
	(c)Brick red	Light green	Calcium (Ca2+) salt.

WET TEST FOR BASIC RADICAL

For wet test for basic radicals, salt solution is to be prepared. The solubility of the salt should be examined in the following solvents. First in cold water and if failed then in (a) hot water (b) Dil. HCl (c) Conc. HCl.

1.Test for Al3+

Experiment	Observation	Inference
(a) To 3ml of salt solution, add solid NH4Cl till the solution is alkaline. Dil.NaOH is added drop wise and then in excess.	Gelatinous white precipitate is formed which dissolved in excess NaOH.	May be Al3+
(b)Take 1 of salt solution in a test tube. Add Disodium hydrogen phosphate solution in it.	Gelatinous white precipitate of AlPO4 is formed which is soluble in dilute HCl.	Al3+ is confirmed.
2.Test for Zn2+		
(a)To 3ml of salt solution, add solid NH4Cl saturation. Then add dil.NH4OH till alkaline. Pass H2S gas through it.	White precipitate is obtained due to formation of ZnS.	Zn2+ is present.
(b)To 2ml of salt solution, add potassium Ferro cyanide solution drop by drop.	White precipitate is formed.	Zn2+ is confirmed.
(c)To 2ml of salt solution, add dilNaOH solution drop by drop then in excess.	White Precipitate is obtained which is soluble in excess of dilute NaOH.	Zn2+ is confirmed.
3.Test For Ca2+		
(a)To 3ml of salt solution, add solid NH4Cl saturation. Then add dil.NH4OH till alkaline. Now add	White precipitate of CaCO3 is obtained.	Ca2+ present.

saturated solution of (NH4)2CO3 to it.		
(b)To 2ml of salt solution add 1ml. of ammonium oxalate solution. Make the solution alkaline with NH4OH.	White precipitate is formed	Ca2+ is confirmed.
Test for Mg2+		
(a)To 2ml of salt solution, add solid NH4Cl till saturation. Then add dil. NH4OH till alkaline. Add Disodium hydrogen phosphate solution.	White precipitate is obtained.	Mg2+ is present.
(b) To 2ml of salt solution, add 1ml of dil. HCl. Then add a few drops of Magneson reagent followed by addition of dil. NaOH in excess.	A blue precipitate is obtained.	Mg2+ is confirmed.
Test for(NH4+)		
(a)To 2ml of salt solution in a test tube, add dil. NaOH solution & boil.	Ammonia gas is evolved.	NH4+ is present.
(b) Show a glass rod dipped in conc. HCl to the above gas.	Dense white fumes obtained.	NH4+ is confirmed.
(c)To 2ml of salt solution add 1ml of Nessler's reagent.	A brown precipitate is obtained.	NH4+ is confirmed.

Nessler' reagent:Nessler' reagent is an alkaline solution of potassium mercuric iodide.

(a)Test for (Na+) Take 2ml of salt solution in a clean test tube. Add 1ml of potassium pyroantimonate solution.	White crystalline precipitate is obtained.	Na+ is confirmed.
(b)Test for (K+) Take 2ml of salt solution .Add 6drops of cobalt nitrate solution followed by sodium nitrite and dil. acetic acid.	Yellow precipitate is formed.	K+ is confirmed.

EXPERIMENT NO-7

Systematic procedure for detection of acid and basic radical in an unknown salt

AIM OF THE EXPERIMENT: To detect the acid and basic radical in an unknown salt.

PRELIMINARY TEST

(i) Salt number
(ii) Color of the salt
(iii) Structure of the salt
(iv)Solubility of the salt

1. DRY TEST TUBE HEATING

Experiment	Observation	Inference
Heat a small quantity of the supplied salt in a clean dry test tube fast slowly and then strongly for about three to four minutes.	(a) water particles condense at the cooler part of the test tube	Salt contains water of crystallization.
	(b) the salt volatilizes out completely forming a white sublimate	Volatile salts. May be NH_4^+ salt
	(c) the salt decrepitates (produces cracking sound)	Crystalline salts.
	(d) The salt melts on heating and solidifies upon cooling.	Alkali and alkaline earth metal salts.
	(e) The salt changes its color. Yellow when hot and white when cooled	May be zinc salt.
	(f) the salt is swelled upon heating	May be Al^{3+}.
	(g)A gas or vapour is evolved.	May be Nitrates of $Na^+$and K^+
	(i) A colourless and odourless gas(O_2) which rekindles a glowing splinter	May be Certain ammonium salts
	(ii) A colourless and pungent smelling gas (NH_3) which turns red litmus paper blue.	May be carbonates
	(iii) A colourless and odourless gas which turnslime water milky	
	(iv)acolourless gas (SO_2) with burning sulphur smell which turns acidified $K_2Cr_2O_7$solution green.	May be sulphate
	(v)acolourless gas (HCl)with irritating smell which fumes in moist air.it produces dense white fumes with a glass rod dipped in conc.NH_4OH.	May be hydrated chloride salt.
	(vi) a colorless gas(H_2S) with	May be hydrated sulphide salts.

	rotten egg smell which turns lead acetate paper black.	

2. TEST FOR VOLATILE SALTS (SODALIME TEST)

Experiment	Observation	Inferences
(a) Take a pinch of salt in a watch glass add a little sodalime (NaOH+CaO) and few drops of water to it. Rub it with the thumb. (b) A glass rod diped in conc. HCl is shown to the evolved gas.	A colorless gas with ammonia smell is observe Copious evolution of dense white fumes.	Ammonium salt is present Ammonium salt is present

3. CHARCOAL CAVITY HEATING

Experiment	Observation	Inferences
Make a small cavity on a charcoal block. Take a little of the salt in the cavity and heat it strongly in oxidizing flame with a blow pipe.	(i)The salt produces cracking sound. (ii) The salt deflagrates (suddenly catches fire and burns vigorously). (iii) The salt melts and sink in to the charcoal cavity on heating and reappears on cooling (iv) the salt may or may not melt (a) A white infusible in candescent (givinglight) residue is obtained. (b) Salt becomes yellow when hot, white when cooled.	May be crystalline salt. May be nitrates. May be alkali or alkaline earth metals(Ca2+,Mg2+,Na+,K+) (Flame test to be performed) May be aluminium. May be zinc salt. Performed cobalt nitrate test.

3. COBALT NITRATE TEST (FOR INFUSIBLE SALT)

Experiment	Observation	Inferences

Heat a small quantity of the salt ina charcoal cavity in the oxidizing flame with the help of a blow pipe till an infusible and in candescent residue is left. In Moisten the residue with a drop of cobalt nitrate solution and heat it in the oxi-dizing flame with the help of a blow pipe. Note the color of the residue.	(i) blue mass (ii) green mass (iii) Pink mass (iv) grey mass	May be Al3+salt. May be Zn2+salt. May be Mg2+salt. May be Ca2+ salt. (flame to be performed)

3.FLAME TEST (FOR FUSIBLE SALTS)

Experiment	Observation		Inferescence
Clean a nichrome wire with sand paper. Dip it in conc.HCl kept in a watch glass. Show it to the flame. It should be done till no color is imparted to the flame. Moisten the nichrome wire with conc.HCl and touch it with a little of the salt. Now heat it in oxidizing flame and note the color of the flame through nacked eye and through double blue glass.	Color through naked eye	Colour through double blue glass	Name of the salt
	(a)Persistent golden yellow color	Colorless	Sodium (Na+)salt.
	(b)Violet crim-son red	Crimson red	Potassium (K+) Salt.
	(c)Brick red	Light green	Calcium(Ca2+) salt.

WET TEST FOR ACID RADICALS

1. Test with dilute HCl.

Experiment	Observation	Inference

(a)Take2ml of dilute HCl in a clean test tube. Warm it and add a little of the salt in to it.	(i)Effervescence takes place with evolution of colorless and odour-less gas which extinguishes the burning splinter. (ii) Effervescence takes place with evolution of a gas having rotten egg smell.	May be CO2 gas from CO32-. [Na2CO3+2HCl 2NaCl+H2O+CO2] ↑ May be H2S gas from sulphide CO2 gas from CO32-.
(b)Test for CO32-. Pass the gas into lime water and then in excess.	Lime water turns milky on excess of gas milkyness disappears.	CO32- confirmed.
c)Test for(S2-) (i)Show lead acetate paper to the color less gas with rotten egg smell. (ii)Show a filter paper dipped in acidified KMnO4 solution to the evolved gas.	Lead acetate paper turns black KMnO4 solution get decolorized	PbS is formed which is black in color. S2- is confirmed. S2- is confirmed.

3. Test with conc. H_2SO_4

Experiment	Observation	Inference

(a)Take a pinch of the salt in a clean and dry test tube and add 2 drops of conc.H2SO4 and warm it.	A colorless fuming gas with pungent smell is evolved.	It may be HCl gas from Cl-. It is due to the formation of NH4Cl. Cl- may be present
(b)Show a glass rod dipped in conc.NH4OH solution to the above gas.	Evolution of dense white fumes and white solid deposited on the tip of the glass rod.	Cl- may present.
(c) Take a pinch of the salt in a clean and dry test tube. Add a little MnO2 and 2-3 drops of conc. H2SO4and heat it.	A greenish yellow gas is evolved which turns starch iodide paper blue.	
Confirmatory Test for Cl-		
(a)Take 1 ml of salt solution in a test tube. Acidified with 1 ml of dil.HNO3 then add AgNO3 solution.	A curdy white ppt. is formed.	It is due to the formation of AgCl. Cl- confirmed.
(b) Wash the above precipitate with distilled water and divide it into two parts.		
(1) Add dil.HNO3 and shake well.	Precipitate is insoluble in dil.HNO3.	AgCl is not soluble in HNO3.
(2) Add dil.NH4OH and shake well.	Precipitate is soluble in dil.NH4OH.	AgCl is soluble in dil. NH4OH due to formation of complex. Cl- is confirmed.

4.Action with conc. H_2SO_4 and copper turnings.

Experiment	Observation	Inferences
(a) Take a pinch of salt in a clean and dry test tube. Add few pieces of copper turnings and 4-5 drops ofconc.H2SO4 and heat it.	Copious brown fumes are evolved and the solution turns green or bluish green.	Brown fume is due to NO2 from nitrate NO3-salt. [Cu+4HNO3 ⟶ Cu(NO3)2+2H2O+2NO2 ↑
(b)Show a filter paper soaked in freshly prepared FeSO4 solution to the above brown gas. Confirmatory test for nitrate (NO3-).	The paper turns black	May beNO3-
(c)Brown ring test: Take 1-2 ml of salt solution. add equal volume of conc.H2SO4 slowly in to the test tube. Cool the test tube perfectly under tap. Then slowly add 2-3 ml of freshly prepared ferrous sulphate solution through the sides of the test tube.	A brown ring is formed at the junction of the two liquid layers.	The brown ring is due to the formation of [Fe(NO)]SO4. NO3-is confirmed

5. Action with dil. HCl and $BaCl_2$.

Experiment	Observation	Inferences
(a) Take about 1-2 ml of salt solution. Acidify with 1-2 ml of dil. HCl. Add about 1ml of BaCl2 solution. Add about 1 ml of Conc. HCl to the above solution and warm it.	A white precipitate is obtained which is insoluble in conc. HCl.	SO42- is confirmed Na2SO4+BaCl2 → BaSO4 + 2NaCl

WET TEST FOR BASIC RADICAL

For wet test for basic radicals, salt solution is to be prepared. The solubility of the salt should be examined in the following solvents. First in cold water and if failed then in (a) hot water (b) Dil. HCl (c) Conc. HCl.

<table>
<tr><td rowspan="6">Residue-1
(a)No residue</td><td colspan="5">Filtrate-1
Warm the filtrate and then passH_2S gas till complete precipitation then filter.</td></tr>
<tr><td>Residue-2</td><td colspan="4">Filtrate-2
Warm the filtrate slightly. Then saturate it by adding solid NH4Cl followed by dil.NH4OH solution then filter.</td></tr>
<tr><td rowspan="5"></td><td>Residue-3</td><td colspan="3">Filtrate-3
Warm the filtrate slightly and then pass H2S gas till complete precipitation and then filter.</td></tr>
<tr><td rowspan="4"></td><td>Residue - 4</td><td colspan="2">Filtrate – 4
Saturate the filtrate with (NH4)2CO3 solution followed by solid NH4Cl and NH4OH. Then filter.</td></tr>
<tr><td rowspan="3"></td><td>Residue-5</td><td>Filtrate – 5
Use this filtrate for the test of NH4+, Na+ , K+ and Mg2+.</td></tr>
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<tr><td colspan="2"></td></tr>
</table>

1. Test for Al^{3+}.

Experiment	Observation	Inference
(a) To 3ml of salt solution, add solid NH4Cl till the solution is alkaline. Dil. NaOH is added drop wise and then in excess.	Gelatinous white precipitate is formed which dissolved in excess NaOH.	May be Al3+
(b)Take 1 of salt solution in a test tube. Add Disodium hydrogen phosphate solution in it.	Gelatinous white precipitate of AlPO4 is formed which is soluble in dilute HCl.	Al3+ is confirmed.
2.Test for Zn2+		
(a)To 3ml of salt solution, add solid NH4Cl saturation. Then add dil.NH4OH till alkaline. Pass H2S gas through it.	White precipitate is obtained due to formation of ZnS.	Zn2+ is present.
(b)To 2ml of salt solution, add potassium Ferro cyanide solution drop by drop.		
(c)To 2ml of salt solution, add dil. NaOH solution drop by drop then in excess.	White precipitate is formed.	Zn2+ is confirmed.
3.Test For Ca2+	White Precipitate is obtained	
(a)To 3ml of salt solution, add solid NH4Cl saturation. Then add dil.NH4OH till alkaline. Now add saturated solution of (NH4)2CO3 to it.	which is soluble in excess of dilute NaOH.	Zn2+ is confirmed.
	White precipitate of CaCO3 is obtained.	
(b)To 2ml of salt solution add 1ml. of ammonium oxalate solution. Make the solution alkaline with NH4OH.		Ca2+ present.
Test for Mg2+		
(a)To 2ml of salt solution, add solid NH4Cl till saturation. Then add dil. NH4OH till alkaline. Add Disodium hydrogen phosphate solution.	White precipitate is formed	Ca2+ is confirmed.
(b) To 2ml of salt solution, add 1ml of dil. HCl. Then add a few drops of Magneson reagent followed by addition of dil. NaOH in excess.	White precipitate is obtained.	Mg2+ is present.
Test for(NH4+)		
(a)To 2ml of salt solution in a test tube, add dil. NaOH solution & boil.	A blue precipitate is obtained.	
(b) Show a glass rod dipped in conc. HCl to the above gas.		
(c)To 2ml of salt solution add 1ml of Nessler's reagent.	Ammonia gas is evolved.	Mg2+ is confirmed.
	Dense white fumes obtained.	

	A brown precipitate is obtained.	NH4+ is present. NH4+ is confirmed. NH4+ is confirmed.

N**essler' reagent:** Nessler's reagent is an alkaline solution of potassium mercuric iodide.

Test for (Na+) (a)Take 2ml of salt solution in a clean test tube. Add 1ml of potassium pyroantimonate solution	White crystalline precipitate is obtained.	Na+ is confirmed.
Test for (K+) Take 2ml of salt solution .Add 6drops of cobalt nitrate solution followed by sodium nitrite and dil. acetic acid.	Yellow precipitate is formed.	K+ is confirmed.