LABORATORY MANUAL
FOR
ENGINEERING CHEMISTRY

PREPARED AND DEVELOPED BY

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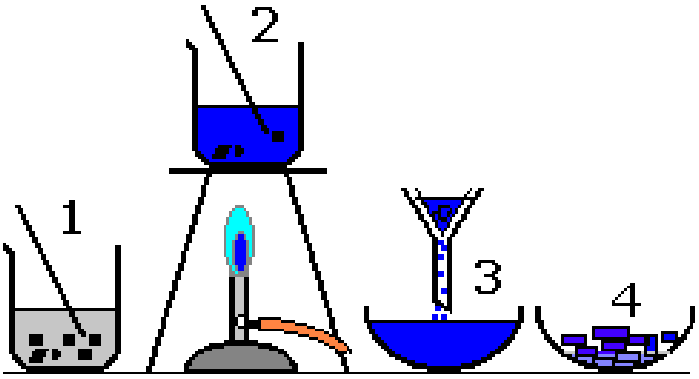
EXPERIMENT No.-1: Preparation and Crystallization of Copper sulphate from Copper carbonate

Aim of the Experiment:

To prepare the crystals of Copper sulphate from Copper carbonate.

Apparatus Required:

1. Beaker
2. Glass rod
3. Tripod stand
4. Wire gauze
5. Bunsen Burner
6. Funnel
7. Filter Stand
8. Filter paper
9. Porcelain Basin



Chemicals Required:

1. Copper Carbonate
2. Dilute Sulphuric acid

Theory:

Copper carbonate when react with dilute sulphuric acid is converted into copper sulphate. The resulting copper sulphate solution is evaporated till the crystallization point is reached. On cooling the resulting solution, the crystals of copper sulphate separates out.

Chemical Equation:

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

Procedure:

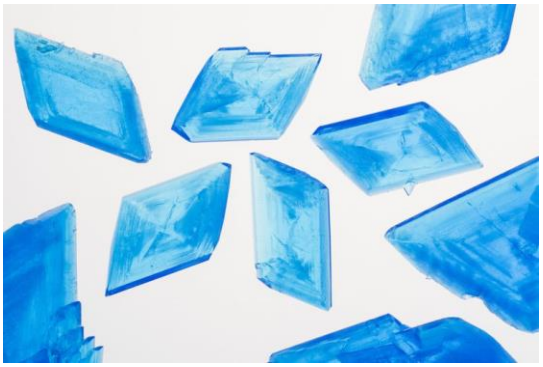
1. Take about 50-60ml of dil. H_2SO_4 solution in a beaker and add the supplied $CuCO_3$ salt pinch by pinch with stirring till a small quantity of $CuCO_3$ solid is left behind.
2. Heat the resulting solution to boil in the beaker for 2-3 minutes while stirring by glass rod to drive out CO_2 gas liberated in the process.
3. Then cool the solution slightly and filter into a china dish.
4. Then add a few drops of dil. H_2SO_4 to the filtrate to check the hydrolysis of salt.
5. Concentrate the filtrate in the basin by evaporation through heating with constant stirring till a drop of solution forms a crystal on the glass rod when it was cooled by blowing air from the mouth.
6. Remove the china dish from the flame and cool inside the water bath till the blue crystals of $CuSO_4$ separate out.

7. Then separate the mother liquor from the crystals and dry by gently pressing between the pads of filter papers.

Result:

Colour of crystal: Blue

Shape of the crystal- Triclinic



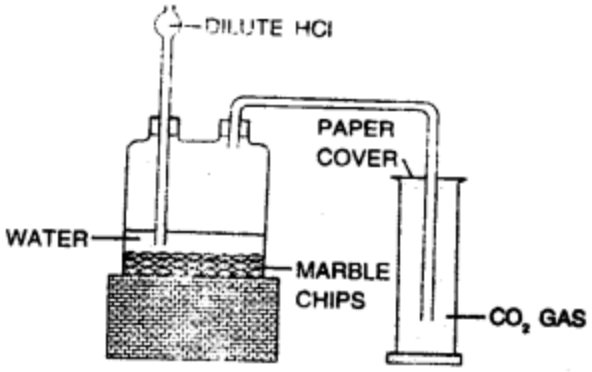
EXPERIMENT No.-2: Preparation of Carbon dioxide gas in Laboratory and study its physical & chemical properties

Aim of the experiment:

To Prepare of Carbon dioxide gas in Laboratory and study its physical & chemical properties.

Apparatus Required:

1. Woulf's Bottle
2. Thistle funnel
3. Delivery tube
4. Rubber cork
5. Gas Jar



Chemicals Required:

1. Marble Chips($CaCO_3$)
2. Dilute HCl
3. Litmus Paper
4. Lime water
5. NaOH solution
6. Phenolphthalein solution

Theory:

In the laboratory, CO_2 gas is prepared by the action of dilute HCl upon marble chips($CaCO_3$)in a Woulf's bottle.

Chemical Equation:

$$\underset{\text{marble chips}}{CaCO_{3(s)}} + 2HCl_{(aq)} \longrightarrow CaCl_2 + H_2O + CO_2 \uparrow$$

Procedure:

1. Fit a Woulf's bottle with rubber cork, thistle funnel and delivery tube in air tight condition.
2. Introduce a few small size marble chips($CaCO_3$) into the Woulf's bottle by opening one of its mouth.
3. Then pour a little amount of water into the Woulf's bottle through the thistle funnel so as to cover the marble chips($CaCO_3$). Insert the thistle funnel more into the Woulf's bottle such that its extreme end remains inside the water.
4. Then add a little quantity of the dil. HCl through the thistle funnel and collect the CO_2 gas is in the gas jar by upward displacement of air.
5. Test the collected gas in the gas jar by showing a burning matchstick at the mouth of the gas jar.
6. Study different physical and chemical properties of CO_2 gas.

Observations:

Physical Properties:

EXPERIMENT	OBSERVATION	INFERENCE
1. Observe the colour of the gas.	1. Colourless	1. CO_2 is a Colourless gas
2. Observe the odour of the gas.	2. Odourless	2. CO_2 is a Odourless gas
3. Show a glowing splinter/ burning matchstick into a test tube full of CO_2 gas.	3. The match stick will extinguish.	3. CO_2 gas neither combustible nor supporter of combustion.
4. Collect the gas in a test tube half filled with water & shake vigorously by putting the thumb at its mouth. Then remove the thumb and observe the level/volume of water inside the test tube.	4.The level of water inside the test tube is found to be increased.	4. CO_2 gas is highly soluble in water.

Chemical Properties:

EXPERIMENT	OBSERVATION	INFERENCE
1. Show a piece of moist blue litmus paper to the gas.	1. The blue litmus change to red.	1. CO_2 gas is acidic in nature.
2. Pass the gas through lime water solution.	2. Lime water turns milky.	2. CO_2 gas is present which produces $CaCO_3$ solution with lime water.
$Ca(OH)_2 + CO_2 \longrightarrow \underset{\text{milky white ppt}}{CaCO_3\downarrow} + H_2O$		
3. Continue to pass the gas through lime water solution in excess. Then boil the solution.	3. Milkiness disappear on excess passing of gas and reappears on boiling.	3. Excess CO_2 gas produces a soluble salt of Calcium bicarbonate and reappears on gently heating due to formation of $CaCO_3$ again.

$CaCO_3 + H_2O + CO_2 \longrightarrow \underset{\text{Soluble}}{Ca(HCO_3)_2}$ $\underset{\text{Soluble}}{Ca(HCO_3)_2} \xrightarrow{\Delta} CaCO_3 + H_2O + CO_2$		
4. Pass the gas through 2-3 ml of very dilute solution of NaOH containing one drop of phenolphthalein indicator solution.	4. The solution turns pink to colourless.	4. CO_2 gas is acidic in nature.

EXPERIMENT No.-3: Volumetric analysis(TITRATION)-Acidimetry-Determination of the strength of an acid solution by using a standard alkali solution.

Aim of the experiment:

To find out the strength of the supplied acid solution by using a standard alkali solution in the laboratory.

Apparatus Required:

1. Burette-50ml
2. Pipette-10ml
3. Conical Flask
4. Beaker
5. Wash bottle
6. Burette stand with clamp
7. Dropper

Chemicals Required:

1. Unknown strength of acid solution
2. A standard base solution
3. Phenolphthalein indicator solution

Theory:

The strength of an unknown acid or basic solution can be calculated by using the Normality Equation:

$$N_1.V_1 = N_2.V_2$$

Where; N_1 = Strength of acid(unknown)

V_1 = Volume of used acid(Burette reading)

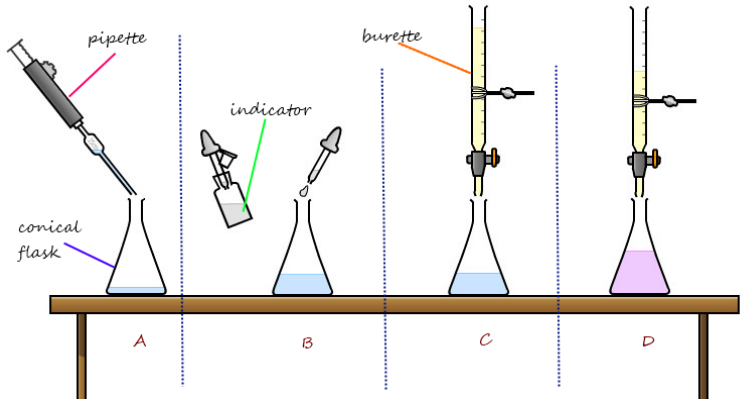
N_2 = Strength of standard base(known)

V_2 = Volume of the alkali(pipette reading)

Procedure:

1. Wash the burette, pipette and conical flask with tap water and then rinse with distilled water.
2. Then rinse the burette thrice with a few ml. of the given acid solution and reject the washings. Then fill the burette with the given acid solution to a convenient level without air bubbles and clamp to the burette stand in a vertical position.
3. Next, rinse the pipette with the supplied alkali/base solution thrice and the washings are reacted.

4. Then, pipette 10ml of the supplied alkali into the conical flask. After transferring the alkali solution, touch the tip of the pipette to the inner side of the conical flask thrice. Wash the sides of the conical flask with a little distilled water.



5. Now, add one drop of phenolphthalein indicator to the solution. The solution turns pink in the flask.
6. Then keep the conical flask under the burette.
7. Note the initial burette reading avoiding parallax error.
8. Now, run down the acid from the burette slowly into the conical flask and shake the flask well. Continue the slow addition of the acid solution till the solution in the conical flask becomes colourless. Now, stop the addition of acid and note the final burette reading.
9. Note down the difference between final burette reading and initial burette reading. This gives the volume of acid consumed.
10. Repeat the titration process till three concordant readings are obtained.

Tabulation:

No. of observ-ations	Volume of alkali (V_2) (In ml)	Burette Reading		Difference F.B.R-I.B.R	Volumes of acid consumed (V_1) (In ml)
		I.B.R	F.B.R		
1					
2					
3					
4					

Calculation:

We know that, from Normality equation;

$N_1.V_1 = N_2.V_2$

Where;

N_1 = Strength of acid(unknown)

V_1 = Volume of used acid(Burette reading)-Avg. value from calculation table

N_2 = Strength of standard base(given)

V_2 = Volume of the alkali(pipette reading) = 10ml

$$N1 = \frac{N2.V2}{N1}$$

Conclusion:

From the above experiment, we conclude that; the normality of acid is _____

EXPERIMENT No.-4: Volumetric analysis(TITRATION)-Alkalimetry-Determination of the strength of an alkali solution by using a standard acid solution.

Aim of the experiment:

To find out the strength of the supplied alkali solution by using a standard acid solution in the laboratory.

Apparatus Required:

1. Burette-50ml
2. Pipette-10ml
3. Conical Flask
4. Beaker
5. Wash bottle
6. Burette stand with clamp
7. Dropper

Chemicals Required:

1. Unknown strength of alkali solution
2. A standard acid solution
3. Phenolphthalein indicator solution

Theory:

The strength of an unknown acid or basic solution can be calculated by using the Normality Equation:

$$N_1.V_1 = N_2.V_2$$

Where; N_1 = Strength of alkali(unknown)

V_1 = Volume of used alkali(Burette reading)

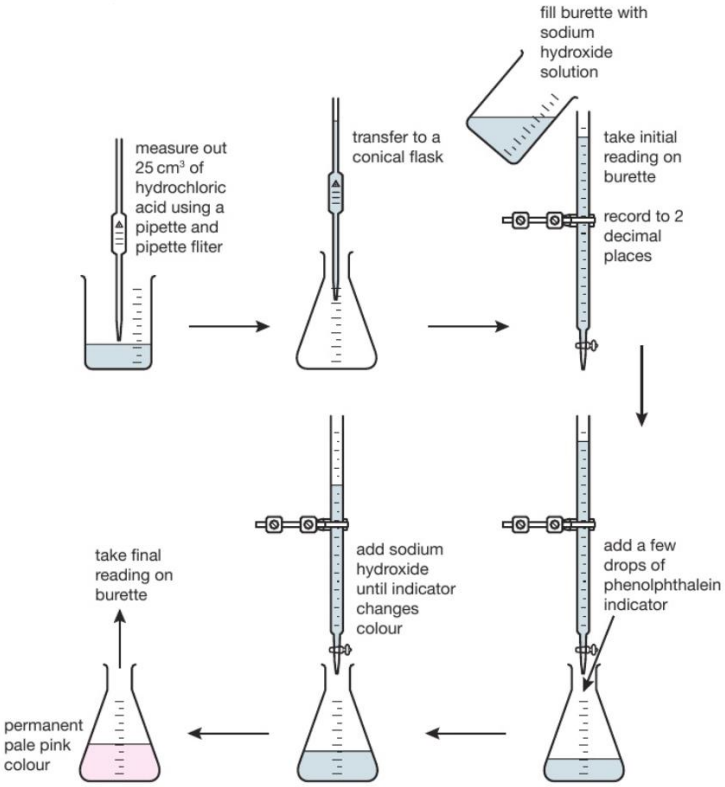
N_2 = Strength of standard acid(known)

V_2 = Volume of the acid(pipette reading)

Procedure:

1. Wash the burette, pipette and conical flask with tap water and then rinse with distilled water.
2. Then rinse the burette thrice with a few ml. of the given base solution and reject the washings. Then fill the burette with the given alkali solution to a convenient level without air bubbles and clamp to the burette stand in a vertical position.
3. Next, rinse the pipette with the supplied acid solution thrice and the washings are reacted.
4. Then, pipette 10ml of the supplied acid into the conical flask. After transferring the alkali solution, touch the tip of the pipette to the inner side of the conical flask thrice. Wash the sides of the conical flask with a little distilled water.

5. Now, add one drop of phenolphthalein indicator to the solution. The solution remains colourless in the flask.
6. Then keep the conical flask under the burette.
7. Note the initial burette reading avoiding parallax error.
8. Now, run down the alkali solution from the burette slowly into the conical flask and shake the flask well. Continue the slow addition of the acid solution till the solution in the conical flask turns pink. Now, stop the addition of alkali and note the final burette reading.
9. Note down the difference between final burette reading and initial burette reading. This gives the volume of alkali consumed.
10. Repeat the titration process till three concordant readings are obtained.



Tabulation:

No. of observ-ations	Volume of acid (V_2) (In ml)	Burette Reading		Difference F.B.R-I.B.R	Volumes of alkali consumed (V_1) (In ml)
		I.B.R	F.B.R		
1					
2					
3					
4					

Calculation:

We know that, from Normality equation;

$N_1.V_1 = N_2.V_2$

Where;

N_1 = Strength of alkali(unknown)

V_1 = Volume of used alkali(Burette reading)-Avg. value from calculation table

N_2 = Strength of standard acid(given)

V_2 = Volume of the acid(pipette reading) = 10ml

$$N1 = \frac{N2.V2}{N1}$$

Conclusion:

From the above experiment, we conclude that; the normality of given alkali is _____

EXPERIMENT No.-5: Preparation of Ammonia gas in Laboratory and study its physical & chemical properties

Aim of the experiment:

To Prepare of ammonia gas in Laboratory and study its physical & chemical properties.

Apparatus required:

1. Round bottom flask
2. Delivery tube
3. Gas jar
4. Clamp stand
5. Cork
6. Test tube

Chemicals required:

1. Solid ammonium chloride(NH_4Cl)
2. Quick Lime(CaO)

Theory:

Ammonia gas is prepared in the laboratory by heating an intimate mixture of solid NH_4Cl and powdered quick lime (CaO) or dry slaked lime $Ca(OH)_2$ in the ratio 1:3 the gas is collected by downward displacement of air as it is lighter than air.

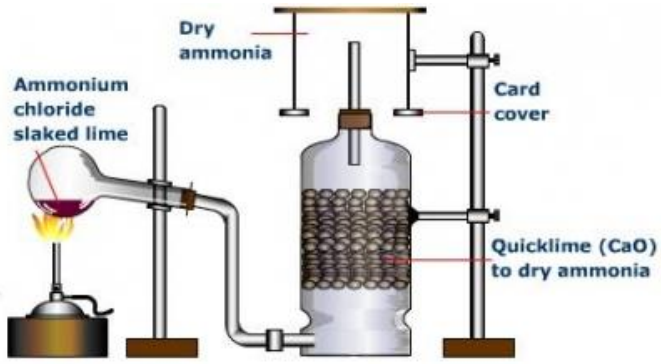
Chemical reaction:

$$\underset{\text{Ammonium Chloride}}{2NH_4Cl} + \underset{\text{Quick Lime}}{CaO} \xrightarrow{\Delta} CaCl_2 + H_2O + \underset{\text{Ammonia}}{2NH_3\uparrow}$$

$$\underset{\text{Ammonium Chloride}}{2NH_4Cl} + \underset{\text{Slaked Lime}}{Ca(OH)_2} \xrightarrow{\Delta} CaCl_2 + H_2O + \underset{\text{Ammonia}}{2NH_3\uparrow}$$

Procedure:

1. Take a mixture of ammonium chloride and quick lime in 1:3 ratio in a mortar and then mix thoroughly and take this mixture in a round bottom flask.
2. Fit the cork along with the delivery tube into the mouth of the round bottom flask.
3. Clamp the RB flask in the clamp stand and heat it continuously.
4. Collect the gas by downward displacement of air.
5. Study its physical & chemical properties.



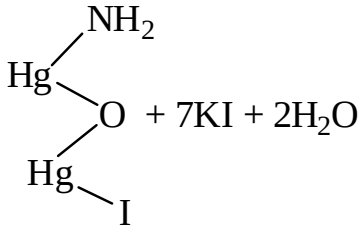
* Better results can be obtained by passing the gas through a CaO tube to dry ammonia.

Observation:

Physical properties:

EXPERIMENT	OBSERVATION	INFERENCE
1. Observe the colour of the gas.	1. Colourless	1. NH_3 is a Colourless gas
2. Observe the odour of the gas.	2. Pungent suffocating odour	2. NH_3 is a pungent suffocating gas.
3. Introduce a burning matchstick into a test tube full of NH_3 gas.	3. The match stick will extinguish.	3. NH_3 gas neither combustible nor supporter of combustion.
4. Invert the gas jar containing NH_3 gas into a trough of water.	4. The level of water is found to be increased.	4. NH_3 gas is highly soluble in water.

Chemical properties:

EXPERIMENT	OBSERVATION	INFERENCE
1. Show a piece of moist red litmus paper to the gas.	1. The red litmus change to blue.	1. NH_3 gas is basic in nature.
2. Show a glass rod dipped in concentrated HCl to the gas.	2. White dense fumes are observed.	2. White dense fumes are observed due to formation of NH_4Cl.
$NH_3 + HCl \longrightarrow NH_4Cl$ (White fumes)		
3. Pass the gas through about 2cc of Nessler's reagent in a clean dry test tube.	3. The colour of the solution changes to reddish brown.	3. The reddish brown colour is due to a complex.
$NH_3 + 2K_2[HgI_4] + 3KOH \longrightarrow$ (Nessler's Reagent)  $+ 7KI + 2H_2O$ Iodide of million's base Reddish brown ppt.		

4. Pass the gas through 2cc of ferric chloride solution.	4. The colour of the solution changes to reddish brown.	4. The reddish brown colour is due to formation of $Fe(OH)_3$ precipitated.
$FeCl_3 + 3NH_3 + 3H_2O \longrightarrow \underset{\text{Reddish brown ppt.}}{Fe(OH)_3\downarrow} + 3NH_4Cl$		
5. First slowly pass the gas through 2cc of aqueous copper sulphate solution in a clean dry test tube and then in excess.	5. First, the colour changes to bluish white and on excess the colour changes to dark blue.	5. NH_3 gas is confirmed.
$CuSO_4 + NH_3 + 2H_2O \longrightarrow \underset{\text{Bluish white gelatinousppt.}}{Cu(OH)_2\downarrow} \xrightarrow{NH_3} \underset{\text{Deep Blue ppt.}}{[Cu(NH_3)_4](OH)_2\downarrow}$		

EXPERIMENT No.-6: SALT ANALYSIS-Test for Known Acid radicals in the given salts

AIM OF THE EXPERIMENT:

To detect known acid radicals in a given salt.

WORKING PROCEDURE FOR INDIVIDUAL ACID RADICALS:

a) TEST FOR CARBONATE(CO_3^{2-}):

EXPERIMENT	OBSERVATION	INFEERENCE
i) **Test with dilute HCL**: Take 1 ml of dilute HCl in a clean dry test tube and heat it upto boiling. Then remove it from boiling and add a pinch of salt to it.	Effervescence takes place with evolution of colourless and odourless gas.	CO_2 gas comes out of carbonate salt.
$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2\uparrow$		
ii) Pass the gas through limewater in small quantity and then in excess.	Limewater turns milky. Milkiness disappears with excess gas.	CO_3^{2-} is confirmed.
$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3\downarrow + H_2O$ $CaCO_3 + H_2O + CO_2 \longrightarrow Ca(HCO_3)_2$ (Soluble)		

b) TEST FOR SULPHIDE(S^{2-}):

EXPERIMENT	OBSERVATION	INFEERENCE
i) **Test with dilute HCL**: Take 1 ml of dilute HCl in a clean dry test tube and heat it upto boiling. Then remove it from boiling and add a pinch of salt to it.	Effervescence takes place with evolution of colourless gas with rotten egg smell.	H_2S gas comes out of sulphide salt.
$Na_2S + 2HCl \longrightarrow 2NaCl + H_2S\uparrow$		

ii) Pass the gas through the filter paper dipped in lead acetate solution.	The filter paper turns black	Lead sulphide is formed. S^{2-} is confirmed.
$(CH_3COO)_2Pb + H_2S \longrightarrow 2CH_3COOH + PbS\downarrow$ (Black ppt.)		

c) TEST FOR CHLORIDE(Cl^-):

EXPERIMENT	OBSERVATION	INFEERENCE
i) **Test with conc. H_2SO_4**: Take a pinch of given salt in a clean dry test tube and add 1 to 2 ml of conc. H_2SO_4 to it.	Effervescence takes place with the evolution of a colourless fuming gas with pungent smell	HCl gas comes out of chloride salt.
$2NaCl + H_2SO_4 \longrightarrow Na_2SO_4 + 2HCl$ $BaCl_2 + H_2SO_4 \longrightarrow BaSO_4 + 2HCl$		
ii) Show a glass rod dipped in conc. NH_4OH to the mouth of the test tube containing salt and conc. H_2SO_4	Dense white fumes are evolved	Volatile NH_4Cl is formed
$NH_4OH + HCl \longrightarrow NH_4Cl + H_2O$ (dense white fumes)		
iii) Action of $AgNO_3$: Take 1 to 2ml of salt solution with distilled water in a clean test tube & acidify it with dilute HNO_3 acid & add a few ml of $AgNO_3$ solution	A curdy white precipitate is formed	The precipitate is due to formation of AgCl.
$NaCl + AgNO_3 \longrightarrow AgCl\downarrow + NaNO_3$		

To the above solution add NH_4OH solution.	The precipitate dissolves and reappears on addition of dilute HCl	The precipitate dissolves in dil. NH_4OH due to formation of complex.
$AgCl + 2NH_4OH \longrightarrow [Ag(NH_3)_2]Cl + 2H_2O$ Diamino silver chloride Soluble in water $[Ag(NH_3)_2]Cl + HNO_3 \longrightarrow AgCl\downarrow + NH_4NO_3 + H_2O$ ppt. reappears		

d) TEST FOR NITRATE(NO_3^-):

EXPERIMENT	OBSERVATION	INFEERENCE
i) **Test with conc. H_2SO_4**: Take a pinch of given salt in a clean dry test tube and add 1 to 2 ml of conc. H_2SO_4 to it and warm gently	Effervescence takes place with the evolution of light brown fumes	Vapours of HNO_3 comes out from a nitrate salt.
$2NaNO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + 2HNO_3$		
ii) **Test with Cu-turnings:** To the above mixture add a few copper turnings and warm gently.	Evolution of copius brown fumes	NO_2 gas is formed from a nitrate salt.
$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2\uparrow$		
iii) **Brown Ring Test:** Take 1 to 2 cc of salt solution in a cleandry test tube and add equal volume of conc. H_2SO_4 to it. Cool it under tap water and add freshly prepared $FeSO_4$ solution through the side of the tube slowly.	A brown ring is formed at the junction of 2 liquid layers.	Brown ring is formed due to formation of nitroso ferrous sulphate.

$$KNO_3 + H_2SO_4 \longrightarrow KHSO_4 + HNO_3$$

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

$$FeSO_4 + NO \longrightarrow [Fe(NO)]SO_4$$

Nitroso ferrous sulphate
Brown ring

e) TEST FOR SULPHATE(SO_4^{2-}):

EXPERIMENT	OBSERVATION	INFEERENCE
Take a little of the given salt in a clean and dry test tube and add a few cc of distilled water and shake well. Acidify it with dil. HCl. Then add barium chloride solution to it	A white precipitate is obtained which is insoluble in conc. HCl even on boiling	Barium sulphate is formed which is insoluble in conc. HCl

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

white ppt.

EXPERIMENT No.-7: SALT ANALYSIS-Test for Known Basic radicals in the given salts

AIM OF THE EXPERIMENT:

To detect known basic radicals in a given salt.

WORKING PROCEDURE FOR INDIVIDUAL ACID RADICALS:

a) TEST FOR Al^{+3}:

EXPERIMENT	OBSERVATION	INFEERENCE
i) Take about 2ml of the supplied salt solution. Add solid NH_4Cl till saturation. Then add dil. NH_4OH solution first drop wise & then in excess till alkaline.	Gelatinous white precipitate is obtained.	Al^{+3} may be present.
$AlCl_3 + 3NH_4OH \longrightarrow Al(OH)_3 \downarrow + 3NH_4Cl$ (Gelatinous white ppt.)		
ii) Take about 2ml of the supplied salt solution. Add dil. NaOH drop by drop to it.	Gelatinous white precipitate is first obtained which is soluble in excess NaOH	Al^{+3} is confirmed
$AlCl_3 + 3NaOH \longrightarrow Al(OH)_3 + 3NaCl$ With excess NaOH solution: $Al(OH)_3 + NaOH \longrightarrow NaAlO_2 + 2H_2O$ (Sodium meta-aluminate)		

b) TEST FOR Zn^{+2}:

EXPERIMENT	OBSERVATION	INFEERENCE
i) Add solid NH_4Cl followed by dil. NH_4OH to the salt solution. Warm it & pass H_2S gas through it.	White precipitate is obtained.	Zn^{+2} may be present

$ZnCl_2 + H_2S \longrightarrow ZnS\downarrow + 2HCl$		
ii) Add dilute NaOH to the solution drop wise & then in excess.	white precipitate is first obtained which is soluble in excess NaOH	Zn^{+2} is confirmed
$ZnCl_2 + 2NaOH \longrightarrow Zn(OH)_2\downarrow + 2NaCl$ $Zn(OH)_2 + 2NaOH \longrightarrow Na_2ZnO_2 + 2H_2O$ Sodium Zincate Water soluble		

c) TEST FOR Ca^{+2}:

EXPERIMENT	OBSERVATION	INFEERENCE
i) Take about 2ml of the supplied salt solution. Add solid NH_4Cl till saturation. Then add dilute NaOH till alkaline. Add saturated solution of ammonium carbonate.	White precipitate is obtained.	Ca^{+2} may be present
$CaCl_2 + (NH)_4CO_3 \longrightarrow CaCO_3\downarrow + 2NH_4Cl$		
ii) The white ppt. obtained is dissolved in dil. CH_3COOH. Add ammonium oxalate solution to it followed by dil. NH_4OH	White precipitate of calcium oxalate is obtained.	Ca^{+2} is confirmed
$CaCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ca + H_2O + CO_2$ $(CH_3COO)_2Ca + (NH_4)_2C_2O_4 \longrightarrow CaC_2O_4 + 2CH_3COONH_4$		

d) TEST FOR Na^+:

EXPERIMENT	OBSERVATION	INFEERENCE
Add 1ml of potassium pyroantimonate solution to 1ml of the salt solution	White precipitate of sodium pyroantimonate is obtained.	Na^+ is confirmed

$$2NaCl + K_2H_2Sb_2O_7 \longrightarrow Na_2H_2Sb_2O_7\downarrow + 2KCl$$

e) TEST FOR K^+:

EXPERIMENT	OBSERVATION	INFEERENCE
Add a few drops of cobalt nitrate solution followed by sodium nitrite and dilute CH_3COOH to 1 ml of salt solution. Allow it to stand for 5 min.	Yellow precipitate of potassium cobalti nitrite if formed	K^+ is confirmed

$$KCl + NaNO_2 \longrightarrow KNO_2 + NaCl$$

$$Co(NO_3)_2 + 2NaNO_2 \longrightarrow Co(NO_2)_2 + 2NaNO_3$$

$$2CH_3COOH + Co(NO_2)_2 + 2KNO_2 \longrightarrow 2CH_3COOK + H_2O + NO + Co(NO_2)_3$$

$$3KNO_2 + Co(NO_2)_3 \longrightarrow K_3[Co(NO_2)_6]\downarrow$$

Potassium cobalti nitrate
yellow ppt

f) TEST FOR NH_4^+:

EXPERIMENT	OBSERVATION	INFEERENCE
a) Add dilute NaOH to 1cc of original salt solution and boil it	Brown precipitate is obtained.	NH_4^+ is confirmed
$NH_4Cl + NaOH \longrightarrow NH_3 + NaCl + H_2O$		
b) Add Nessler's reagent to 1cc original salt solution	Reddish brown precipitate is obtained.	NH_4^+ is confirmed

g) TEST FOR Mg^{+2}:

EXPERIMENT	OBSERVATION	INFEERENCE
a) To 1 ml of salt solution add solid NH_4Cl till saturation & dil. NH_4OH till alkaline. Then add disodium hydrogen phosphate solution to it.	White precipitate is obtained.	Mg^{+2} is confirmed.
$MgSO_4 + NH_4OH + Na_2HPO_4 \longrightarrow Mg(NH_4)PO_4 + H_2O + Na_2SO_4$		
b) To 1 ml of salt solution, add dil. HCl with a few drops of magneson reagent followed by addition of excess dil. NaOH	A blue precipitate is obtained.	Mg^{+2} is confirmed.

EXPERIMENT No.-8: SALT ANALYSIS-Test for unknown acid and Basic radicals in a given salt.

Aim Of The Experiment:

To identify the acid and basic radicals in a given unknown salt.

Preliminary Test:

Salt No.-
Colour- white/yellow/blue/grey/colourless
Structure- Crystalline/amorphous
Odour- Ammoniacal/rotten egg/odourless
Solubility- In Cold H_2O/Hot H_2O
In cold dil HCl/hot dil HCl

Litmus Test:

EXPERIMENT	OBSERVATION	INFEERENCE
Prepare a salt solution in water. Then add a drop of solution to blue litmus paper and red litmus paper.	a) Blue litmus paper changed to red b) Red litmus paper changed to blue c) No change observed	a) Salt is acidic in nature b) Salt is basic in nature c) Salt is neutral

Dry Test for Basic Radicals:

1. **Dry Test tube Heating:**

EXPERIMENT	OBSERVATION	INFEERENCE
Take a pinch of salt in a clean and dry test tube and heat in strongly.	i) White sublimate is formed. ii) Decripitation takes place. iii) Deflagration takes place. iv) The colour of the salt becomes yellow when hot and white when cold. v) The salt fuses(melts) on heating and solidifies on cooling. vi) The salt swells on heating. vii) No change observed	i) Ammonium salt may be present ii) Crystalline salt iii) Some nitrate salt iv) Zinc salt v) Alkali or alkaline earth metal salt vi) Some aluminium salt vii)Next test is to be performed.

2. Sodalime Test:

EXPERIMENT	OBSERVATION	INFEERENCE
Take a pinch of the salt in a mortar and add equal volume of soda lime to it & add few drops of distilled water to it. Then rub it with the help of a pestle.	i. Ammonia gas is evolved ii. No change observed	i. Ammonium salt may be present. ii. Next test is to be performed.

3. Charcoal cavity test:

EXPERIMENT	OBSERVATION	INFEERENCE
Take a little of the salt in a charcoal cavity and heat it in oxidizing flame strongly.	i. The salt decrepitates. ii. The salt deflagrates. iii. The salt fuses and is absorbed by the charcoal cavity which reappears on cooling. iv. Gives white infusible incandescent residue. v. No change observed	i. Crystalline salt ii. Nitrate salt iii. Alkali or alkaline earth metal salts iv. Perform cobalt nitrate test v. Next test is to be performed.

4. Cobalt nitrate test:

EXPERIMENT	OBSERVATION	INFEERENCE
In the above Charcoal cavity, take a pinch of the salt and add 1-2 drops of cobalt nitrate to it. Then heat it in a oxidizing flame of the bunsen burner strongly with the help of a blow pipe.	i. Blue mass ii. Green mass iii. Grey mass iv. No change observed	i. Aluminium salt ii. Zinc salt iii. Calcium salt iv. Next test is to be performed.

5. Flame Test:

EXPERIMENT	OBSERVATION		INFEERENCE
Take a few cc of concentrated HCl in a watch glass. Clean a piece of nichrome wire with the help of sand paper. Dip the nichrome wire in conc. HCl and show it to the oxidizing flame of burner. Repeat it thrice. Then first dip the nicrome wire in conc. HCl and allow it to touch the salt and show it to the outer layer of the non-luminous flame. Observe the colour of the flame through the naked eye and then with double blue glass.	Colour of the flame through naked eye	Colour of the flame through double blue glass	Sodium
	i. Persistent golden yellow	Colourless	
	ii. Violet	Red	Potassium
	iii. Brick red/orange red	Light green	Calcium
	iv. Green flame with blue center	-----	Copper

<u>Dry Test for Acid Radicals:</u>

1. Test for Gr-I acid Radicals (CO_3^{2--} and S^{2--}):

EXPERIMENT	OBSERVATION	INFEERENCE
Take a pinch of salt in a test tube with 2cc of dilute HCl and warm it.	1. Effervescence takes place with evolution of colourless and odourless gas or colourless gas with rotten egg smell.	1. CO_3^{2--} or $S^{2—}$ may be present. Confirmatory tests are to be performed.
	2. No change is observed	2. Next test is to be performed.

3. Test for Gr-II acid Radicals (Cl^{--} and NO_3^{--}):

EXPERIMENT	OBSERVATION	INFEERENCE
To a pinch of salt, add 3 to 4 drops of conc. H_2SO_4 in a clean and dry test tube.	1. A colourless fuming gas with pungent smell is evolved. 2. Brown fumes with pungent smell are evolved. 3. No change is observed	1. Cl^{--} may be present. Confirmatory test for chloride is to be performed 2. NO_3^{--} may be present. Confirmatory test for chloride is to be performed 3. Next test is to be performed.

4. Test for Gr-III acid Radicals(SO_4^{2--}):

EXPERIMENT	OBSERVATION	INFEERENCE
Take a little of the given salt in a clean and dry test tube and add a few cc of distilled water and shake well. Acidify it with dil. HCl. Then add barium chloride solution to it.	A white precipitate is obtained which is insoluble in conc. HCl even on boiling	Barium sulphate is formed which is insoluble in conc. HCl. Sulphate is confirmed.

<u>Wet test for acid radicals:</u>

1. Test for Carbonate

EXPERIMENT	OBSERVATION	INFEERENCE
Pass the gas through limewater in small quantity and then in excess.	Limewater turns milky. Milkiness disappears with excess gas.	CO_3^{2-} is confirmed.

2. Test for Sulphide

EXPERIMENT	OBSERVATION	INFEERENCE
Pass the gas through the filter paper dipped in lead acetate solution.	The filter paper turns black	Lead sulphide is formed. S^{2-} is confirmed.

3. Test for Chloride

EXPERIMENT	OBSERVATION	INFEERENCE
i) Show a glass rod dipped in conc. NH_4OH to the mouth of the test tube containing salt and conc. H_2SO_4	Dense white fumes are evolved	Volatile NH_4Cl is formed.
ii) Action of $AgNO_3$: Take 1 to 2ml of salt solution with distilled water in a clean test tube & acidify it with dilute HNO_3 acid & add a few ml of $AgNO_3$ solution iii) To the above solution add NH_4OH solution.	A curdy white precipitate is formed The precipitate dissolves and reappears on addition of dilute HCl	The precipitate is due to formation of AgCl. The precipitate dissolves in dil. NH_4OH due to formation of complex. Cl^- is confirmed.

4. Test for Nitrate

EXPERIMENT	OBSERVATION	INFEERENCE
i) Test with Cu-turnings: To the above mixture add a few copper turnings and warm gently.	Evolution of copius brown fumes	NO_2 gas is formed from a nitrate salt.
ii) Brown Ring Test: Take 1 to 2 cc of salt solution in a clean dry test tube and add equal volume of conc. H_2SO_4 to it. Cool it under tap water and add freshly prepared $FeSO_4$ solution through the side of the tube slowly.	A brown ring is formed at the junction of 2 liquid layers.	Brown ring is formed due to formation of nitroso ferrous sulphate. Nitrate is confirmed.

Wet test For Basic Radicals:

Separation into groups:

<table>
<tr><td colspan="6">Prepare a salt solution with dilute HCl and filter.</td></tr>
<tr><td rowspan="5"><u>Residue-1</u>

Gr-I radicals</td><td colspan="5">Filtrate-1: Warm the filtrate & then pass H_2S gas till complete precipitation and filter</td></tr>
<tr><td rowspan="4"><u>Residue-2</u>

Gr-IIA and Gr-IIB radicals</td><td colspan="4">Filtrate-2: Warm the filtrate slightly. Then saturate it by adding solid NH_4Cl followed by dilute NH_4OH solution and then filter.</td></tr>
<tr><td rowspan="3"><u>Residue-3</u>

Gr-IIIA radicals

Gelatinous white ppt(Al^{+3})

If no ppt. test for Filtrate-3</td><td colspan="3">Filtrate-3: Warm the filtrate slightly & then pass H_2Sgas till complete precipitation & then filter.</td></tr>
<tr><td rowspan="2"><u>Residue-4</u>

Gr-IIIB radicals

white ppt(Zn^{+2})

If no ppt. test for Filtrate-4</td><td colspan="2">Filtrate-4: Saturate the filtrate with $(NH_4)_2CO_3$ solution followed by solid NH_4Cl & NH_4OH and filter.</td></tr>
<tr><td><u>Residue-5</u>

Gr-IV radicals white ppt (Ca^{+2})

If no ppt. test for Filtrate-5 individually</td><td>Filtrate-5: Use this filtrate for the individual confirmatory test of Gr-V radicals.(Mg^{+2}, K^+, Na^+,NH_4^+)</td></tr>
</table>

Test for Gr-IIIA Radicals(Al^{+3}):

EXPERIMENT	OBSERVATION	INFEERENCE
Take about 2ml of the supplied salt solution. Add dil. NaOH drop by drop to it.	Gelatinous white precipitate is first obtained which is soluble in excess NaOH	Al^{+3} is confirmed

Test for Gr-IIIB Radicals(Zn^{+2}):

EXPERIMENT	OBSERVATION	INFEERENCE
Add dilute NaOH to the solution drop wise & then in excess.	white precipitate is first obtained which is soluble in excess NaOH	Zn^{+2} is confirmed

Test for Gr-IV Radicals(Ca^{+2}):

EXPERIMENT	OBSERVATION	INFEERENCE
The white ppt. obtained is dissolved in dil. CH_3COOH. Add ammonium oxalate solution to it followed by dil. NH_4OH	White precipitate of calcium oxalate is obtained.	Ca^{+2} is confirmed

Test for Gr-V Radicals(Na^+, K^+, Mg^+, NH_4^+):

EXPERIMENT	OBSERVATION	INFEERENCE
<u>Test for Na^+:</u> Add 1ml of potassium pyroantimonate solution to 1ml of the salt solution	White precipitate of sodium pyroantimonate is obtained.	Na^+ is confirmed
<u>Test for K^+:</u> Add a few drops of cobalt nitrate solution followed by sodium nitrite and dilute CH_3COOH to 1 ml of salt solution. Allow it to stand for 5 min.	Yellow precipitate of potassium cobalti nitrite if formed	K^+ is confirmed
<u>Test for NH_4^+:</u> i) Add dilute NaOH to 1cc of original salt solution and boil it	Brown precipitate is obtained.	NH_4^+ is confirmed
ii) Add Nessler's reagent to 1cc original salt solution	Reddish Brown precipitate is obtained.	NH_4^+ is confirmed

<u>Test for Mg^{+2}:</u> i)To 1 ml of salt solution add solid NH_4Cl till saturation & dil. NH_4OH till alkaline. Then add disodium hydrogen phosphate solution to it.	White precipitate is obtained.	Mg^{+2} is confirmed.
ii)To 1 ml of salt solution, add dil. HCl with a few drops of magneson reagent followed by addition of excess dil. NaOH	A blue precipitate is obtained.	Mg^{+2} is confirmed.

<u>Conclusion:</u>

The acid radical obtained:

The basic radical obtained:

So, the given salt is :