Chemistry Laboratory Manual

for

1st /2nd Semester

Diploma Engg. students

Govt. Polytechnic Sambalpur(Rengali)

Department of Maths & Science

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EXPERIMENT- 1

AIM OF THE EXPERIMENT

Preparation and study of properties of Carbon Dioxide

Objectives of the experiment:

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

- Specify the chemicals, which are used to prepare CO_2 gas in the laboratory.
- Know the physical and chemical properties of the gas.

APPARATUS REQUIRED

1. woulf's bottle
2. thistle funnel
3. delivery tube
4. Rubber cork
5. Gas jar with lid
6. Few test tubes

CHEMICALS REQUIRED

1. Marble chips
2. Dil. Hydrochloric Acid
3. Litmus paper
4. Magnesium ribbon
5. Lime water
6. Phenolphthalein solution

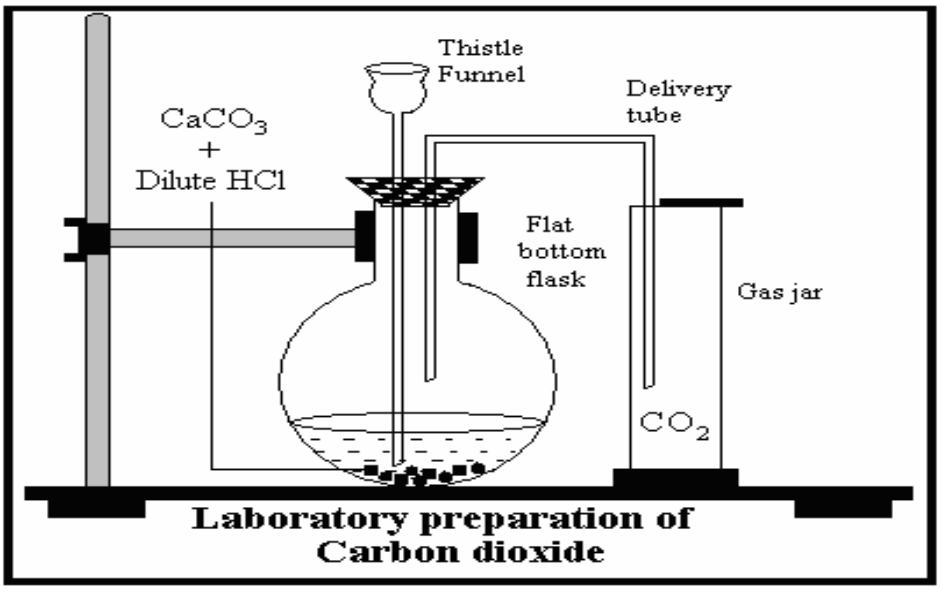
THEORY:

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

CHEMICAL EQUATIONS:

$$CaCO_3 + 2HCl \longrightarrow CaCl_2 + CO_2\uparrow + H_2O$$

LABORATORY DIAGRAM:



PROCEDURE:

1. Take a woulf's bottle fitted with rubber cork, thistle funnel and delivery tube. Examine that it is perfectly airtight. In case of air leakage, use melted paraffin wax or grease.
2. Introduce few small marble chips into the woulf's bottle by opening one of its mouths.
3. Now pour some water into the woulf's bottle through the thistle funnel so as to cover the marble chips.
4. Insert the thistle funnel more into the woulf's bottle such that its extreme end remains inside the water.
5. Now add little quantity of the dil. Hydrochloric acid through the thistle funnel. Do not add excess amount of acid at a time to exhaust the marble chips before the experiment is completed.
6. Then collect the carbon dioxide gas in the gas jar by upward displacement of air. Test the collected gas in the jar by showing a burning splinter at the mouth of gas jar.
7. Study the properties of carbon dioxide gas by collecting the gas in different test tubes.

OBSERVATION:

Physical properties

SL.	EXPERIMENT	OBSERVATION	INFERENCE
1.	Observe the colour of the gas		
2.	Observe the odour of the gas		
3.	Enter a glowing splinter into a test tube full of CO_2 gas.		
4.	Invert the 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.		
5.	Collect the gas in a test tube half-filled with water. Shake the test tube vigourously 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.	EXPERIMENT	OBSERVATION	INFERENCE
1.	A piece of moist blue litmus paper is shown to the gas.		
2.	Pass the CO2 gas through 2-3 ml of dilute solution of sodium hydroxide(NaOH) containing one drop of phenolphthalein solution.		
3.	a) Pass the gas through lime water. b) Pass the gas in excess. **c)** Boil the solution**.**		
4.	Introduce a burning magnesium ribbon into a test tube /gas jar containing carbon dioxide gas.		

SAFETY AND PRECAUTIONS

1. The fittings should be airtight.
2. The end of thistle funnel must be/remain deep inside the solution.
3. The shorter end of the delivery tube should remain above the surface of the solution in the woulf's bottle.
4. The longer end of the delivery tube must reach the bottom of the gas jar.
5. Addition of excess of dil.hydrochloric acid should be avoided.
6. The gas should be collected after removing air from the apparatus**.**

VIVA-VOCE

1. What is the nature(acidic/basic) of carbon dioxide gas?
2. Which chemicals are used for the preparation of carbon dioxide gas?
3. What are the observations when carbon dioxide gas is passed through the lime water? Name the compounds which leads to the following observations.
4. CO_2 is —— than air.(heavier/lighter)
5. CO2 gas is collected —— displacement of air.(downward/upward)
6. Lime water turns milky by passing CO_2 gas through it due to formation of ——— . Give equation
7. The milkiness of the limewater disappears by passage of excess CO_2 gas due to the formation of ——— . give equation
8. What happens when a burning magnesium ribbon is introduced in the gas jar containing carbon dioxide gas? Give equations?
9. State industrial uses of carbon dioxide gas.

Experiment- 2

Aim of the experiment

Preparation and study of properties of NH_3 gas

APPARATUS REQUIRED

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

CHEMICALS REQUIRED

1. Ammonium Chloride, NH_4Cl
2. Calcium Hydroxide, $Ca(OH)_2$

THEORY:

Ammonium salts when are treated to excess alkali gives off ammonia gas. Ammonia gas is prepared in laboratory by heating the mixture of Ammonium Chloride,NH_4Cl & Calcium Hydroxide,$Ca(OH)_2$ paste in 1:3 ratio by weight.
The reaction proceeds as,

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

The gas so formed is collected in the gas jar by downward displacement of air because ammonia gas is lighter than air.
The gas cannot be collected under water because it is highly soluble in water.
Ammonia gas is dried by passing it through the glass bottle contain CaO(lime tower).
Anhydrous $CaCl_2$ and conc.H_2SO_4 cannot be used as drying agent because they react with ammonia.

LABORATORY DIAGRAM:

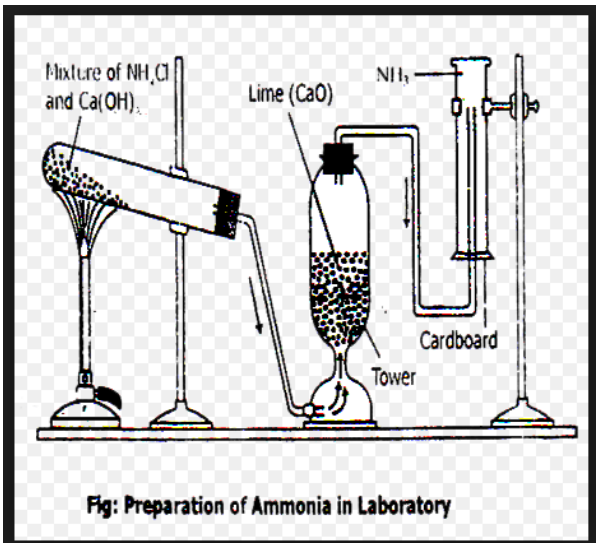


Fig: Preparation of Ammonia in Laboratory

PROCEDURE:

1. Take a hard glass test tube with rubber cork and delivery tube.
2. Mix 1:3 ratio of ammonium chloride and calcium hydroxide and place the mixture into the test tube.
3. Tilt the test tube at 30 degree angle and clamp it to the stand.
4. Attach the rubber cork to the test tube along with delivery tube connected at one end of the test tube and the other end connected to the glass jar containing CaO. The quicklime is present so as to absorb moisture present in the ammonia gas. Make sure that all the connections are airtight to prevent leakage of ammonia gas.
5. Now carefully and gently heat the mixture in the test tube.
6. Then collect the ammonia gas in the gas jar by downward displacement of air.

OBSERVATION:

Physical properties

SL.	Experiment	Observation	Inference
1	Color of the gas		
2	Odour of the gas		
3	Collect the gas in a test tube half-filled with water. Shake the test tube vigorously by putting the thumb at its mouth and remove the thumb andobserve the volume of water in the test tube.		

Chemical properties

Sl.	Experiment	Observation	Inference
1	A piece of moist red litmus paper is shown to the gas.		
2	Pass the gas into the test tube containing copper sulphate solution for short time.		
3	Pass the gas into the test tube containing copper sulphate solution for long time.		
4	Pass the ammonia gas into the test tube containing nessler's reagent.		

CONCLUSION

Ammonia gas is prepared at lab by using NH_4Cl & $Ca(OH)_2$. Ammonia gas is basic in nature. It is highly soluble in water.

SAFETY MEASURES

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

VIVA-VOCE QUESTIONS

1. What is the nature(acidic/basic) of ammonia gas?
2. Which reactants are used for preparation of ammonia gas?
3. Name the compound used as drying agent in preparation of ammonia gas.
4. What happens when ammonia gas is passed through the copper sulphate solution? Name the compounds which lead to the following observations.
5. What happens when ammonia gas is passed through nessler's reagent?
6. What is the shape of ammonia molecule?
7. Explain why ammonium nitrate is not used in the preparation of ammonia gas.

EXPERIMENT- 3

AIM OF THE EXPERIMENT

Crystallization of copper sulphate from copper carbonate

Objectives of the experiment:

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

- Know the formula of blue vitrol.
- Know application of and uses of copper sulphate.
- Know laboratory method of preparation of crystallization salt.

APPARATUS REQUIRED:

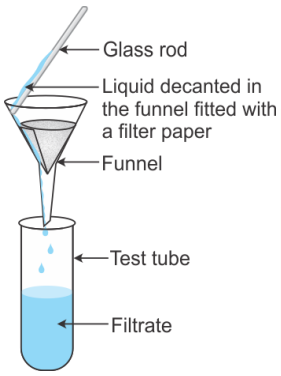
Beaker	funnel	glass rod
Porcelain basin	tripod stands	wire gauge
Bunsen burner	filter paper	filter stand

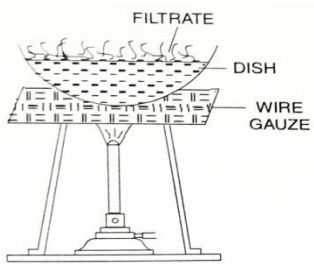
CHEMICALS REQUIRED:

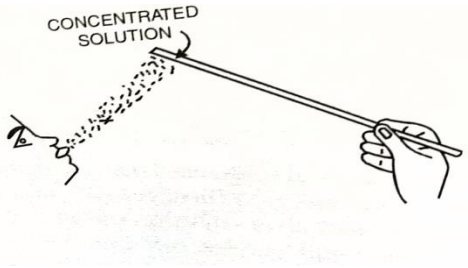
- Calcium carbonate ($CaCO_3$)
- Dilute sulphuric acid (H_2SO_4)

PROCEDURE:

1. Take about 60 ml of dilute sulphuric acid in a beaker.
2. Add the supplied copper carbonate pinch by pinch with continuos stirring till a small quantity of solid is lift undissolved.
3. Heat the resulting solution in a beaker for 2-3 minutes to escape CO_2 gas.
4. Cool slightly and filter into the porcelain basin.
5. Add a few drops of dilute H_2SO_4 to the filterate inorder to check hydrolysis of salt.
6. Concentrate the filterate in the basin by evaporation through heatingwith constant stirring till a drop of solution forms crystal on the the glass rod by blowing air into it from mouth.
7. Remove the basin from the flame and allow to cool slowly at room temperature for about happen an hour without disturbing the basin during cooling.
8. Decant the mother liquor to separate blue crystals of copper sulphate.
9. Dry the crystals in the folds of the filter paper.







Filtration of solution Concentrating the filtrate Testing the crystallization point

RESULT:

1. Colour: ……
2. Texture and shape: ……
3. Yield: ……. Gm

SAFETY AND PRECAUTIONS:

1. Minimum amount of dilute H_2SO_4 should be used to prepare $CuSO_4$ solution.
2. The filtrate should be made slightly acidic with a few drops of $H2SO_4$ to prevent hydrolysis of salt.
3. The solution should not be treated beyond the crystallization point unless the water of crystallization of the crystal will evaporate and colour of the crystal will not be blue.
4. The crystal should not be dried by heating as it results the evaporation of water of crystallization.
5. While adding the copper sulphate to H_2SO_4 as the saturation point approaches, it delays the solubility of the solute. So it should be given sufficient time for its solubility

VIVA-VOCE QUESTIONS

1. What is the formula of blue vitrol?
2. What is the shape of copper sulphate crystals?
3. Why dil. Sulphuric acid is added to the mother liquor after filtration?
4. What happens when blue vitrol is heated?
5. Write down the chemical reaction for preparation of copper sulphate crystals.
6. Define the term “cystallization”.
7. Why a substance is put to crystallization?
8. Why is the solution not much concentrated or heated to dryness to get crystals?
9. Is the aqueous solution of copper sulphate is acidic,neutral or basic ?

Experiment- 4

Aim of the experiment

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

Objectives of the experiment:

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

- Calculate the strength of the solution.
- Acquire knowledge about alkalimetry,standard solution,normal solution,different types of indicators.
- Know the difference between alkalimetry and acidimetry.

Apparatus Required

1. Burette(50 ml)
2. Burette stand with clamp
3. Pipette(10 ml)
4. Conical flask(100 ml)
5. Measuring flask(250 ml)
6. White glazed tile

Chemicals required

1. Acid solution of unknown strength
2. Alkali solution of known strength
3. Indicator: methyl orange

Theory

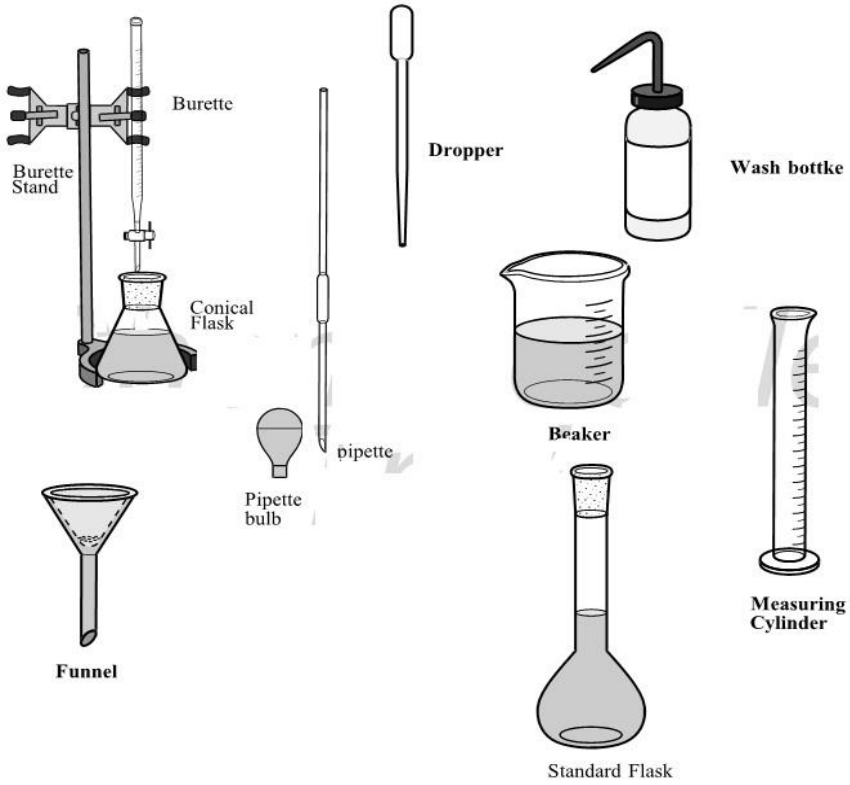
The strength of acid is determined by titrating it against a standard solution of alkali In this titration, methyl orange, a weak base (yellow in the unionised form) is used as an indicator. In this experiment also, the titration follows the usual course, i.e., the proton furnished by the addition of the acid first neutralises alkali solution. When the entire alkali solution is neutralised, the last drop of the acid added from the burette produces the pinkish red colour change, which is the end point. The concentration (strength) of the unknown solution is calculated in g/L. It is calculated from the molarity of the solution. Here, the molarity equation is written as

Base Acid

$$N_1V_1 = N_2V_2$$

where, N_1 and N_2 are the normalities, V_1 and V_2 are the volumes of the base and acid respectively used to neutralise each other.

Laboratory setup diagram



Procedure

(i) Clean the burette thoroughly, wash it with distilled water and finally rinse it with acid solution. (Always rinse the burette with the solution, which is to be taken in it. Clamp the burette vertically in a burette stand.

(ii) Fill the acid solution solution into the burette through a funnel above the zero mark.

(iii) Remove the air gap, if any, from the nozzle of the burette by running the solution forcefully from the burette nozzle.

(iv) Remove the funnel before noting initial reading of the burette. Also while noting the reading, see that no drop of the liquid is hanging at the nozzle of the burette.

(v) Note the initial reading by keeping the eye exactly at the same level as the lower meniscus of the solution.

(vi) Pipette out 10 mL of alkali solution in a washed and dried conical flask. Always wash the pipette with water and rinse with the liquid to be measured before pipetting out the liquid.

(vii) Add 1-2 drops of methyl orange indicator to the conical flask. The solution becomes straw yellow colour. Place the flask over the glazed tile. Then slowly add the alkali solution till it becomes faintly yellow colour is obtained.

(viii) Now add the alkali solution dropwise, until the colour of the solution just change from faint yellow to faint pink. This is the end point. Note the final burette reading. Repeat the process to get three concordant readings.

Observation

Sl. No.	Volume of alkali solution taken in ml(V_B)	Initial burette reading ml.	Final burette reading ml.	Difference ml.	Volume of acid consumed in ml.(V_A)
1.					
2.					
3.					
4.					

Calculation

We know that, $V_AS_A=V_BS_B$

Here VA= burette reading(volume of acid)

VB =pipette reading(volume of acid)

$$S_B=N/10 \text{ and } S_A = \frac{V_B \times S_B}{V_A}(\frac{N}{10})$$

Result

Strength of unknown acid solution = _____ (N/10)

Conclusion

From the above titration result, the strength of Unknown acid solution is found to be _____

Precautions

a. Care should be taken while handling the acid and base.
b. Always rinse the burette and the pipette with the solution which is to be taken in them.
c. Remove the air gap if any, from the burette before titration.
d. Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
e. Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
f. Never use burette and pipette with a broken nozzle.

g. Never suck a strong acid or an alkali with the pipette, use pipette bulb.
h. Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.
i. While transferring the solution to the flask, do not blow out the last drop of the solution from the jet of the pipette.

Experiment- 5

Aim of the experiment

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

Objectives of the experiment:

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

- Calculate the strength of the solution.
- Acquire knowledge about alkalimetry,standard solution,normal solution,different types of indicators.
- Know the difference between alkalimetry and acidimetry.

Apparatus Required

1. Burette(50 ml)
2. Burette stand with clamp
3. Pipette(10 ml)
4. Conical flask(100 ml)
5. Measuring flask(250 ml)
6. White glazed tile

Chemicals required

1. Alkali solution of unknown strength
2. Acid solution of known strength
3. Indicator: methyl orange

Theory

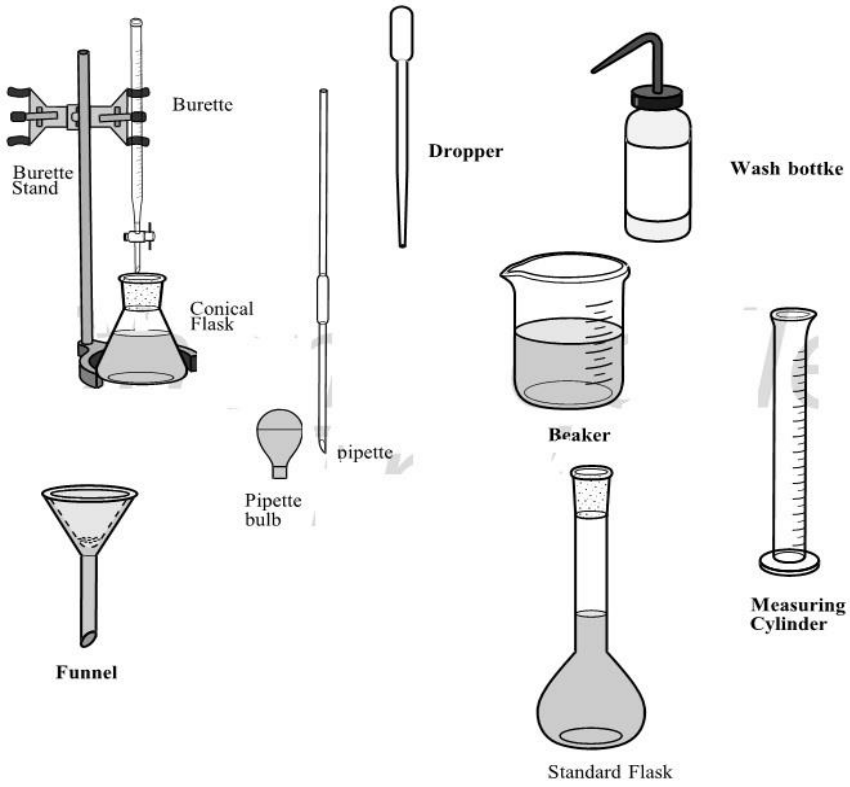
The strength of alkali is determined by titrating it against a standard solution of acid. In this titration, methyl orange, a weak base (yellow in the unionised form) is used as an indicator. In this experiment also, the titration follows the usual course, i.e., the proton furnished by the addition of the acid first neutralises alkali solution. When the entire alkali solution is neutralised, the last drop of the acid added from the burette produces the pinkish red colour change, which is the end point. The concentration (strength) of the unknown solution is calculated in g/L. It is calculated from the molarity of the solution. Here, the molarity equation is written as

Base Acid

$$N_1V_1 = N_2V_2$$

where. N_1 and N_2 are the normarities, V_1 and V_2 are the volumes of the base and acid respectively used to neutralise each other.

Laboratory setup diagram



Procedure

(i) Clean the burette thoroughly, wash it with distilled water and finally rinse it with acid solution. (Always rinse the burette with the solution, which is to be taken in it. Clamp the burette vertically in a burette stand.

(ii) Fill the acid solution solution into the burette through a funnel above the zero mark.

(iii) Remove the air gap, if any, from the nozzle of the burette by running the solution forcefully from the burette nozzle.

(iv) Remove the funnel before noting initial reading of the burette. Also while noting the reading, see that no drop of the liquid is hanging at the nozzle of the burette.

(v) Note the initial reading by keeping the eye exactly at the same level as the lower meniscus of the solution.

(vi) Pipette out 10 mL of alkali solution in a washed and dried conical flask. Always wash the pipette with water and rinse with the liquid to be measured before pipetting out the liquid.

(vii) Add 1-2 drops of methyl orange indicator to the conical flask. The solution becomes straw yellow colour. Place the flask over the glazed tile. Then slowly add the alkali solution till it becomes faintly yellow colour is obtained.

(viii) Now add the alkali solution dropwise, until the colour of the solution just change from faint yellow to faint pink. This is the end point. Note the final burette reading. Repeat the process to get three concordant readings.

Observation

Sl. No.	Volume of Alkali solution taken in ml(.V_B)	Initial burette reading ml.	Final burette reading ml.	Difference ml.	Volume of acid consumed in ml.(V_A)
1.					
2.					
3.					
4.					

Calculation

We know that, $V_AS_A=V_BS_B$

Here VA= burette reading(volume of acid)

VB =pipette reading(volume of acid)

$$S_A=N/10 \text{ and } S_B = \frac{V_A \times S_A}{V_B}\left(\frac{N}{10}\right)$$

Result

Strength of unknown alkali solution =_____ (N/10)

Conclusion

From the above titration result, the strength of Unknown alkali solution is found to be_____

Precautions

a. Care should be taken while handling the acid and base.
b. Always rinse the burette and the pipette with the solution which is to be taken in them.
c. Remove the air gap if any, from the burette before titration.
d. Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
e. Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
f. Never use burette and pipette with a broken nozzle.
g. Never suck a strong acid or an alkali with the pipette, use pipette bulb.
h. Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.
i. While transferring the solution to the flask, do not blow out the last drop of the solution from the jet of the pipette.

Viva voce questions

a. what is volumetric analysis?
b. What is meant by term titration?
c. Define titrant and titrate.
d. What is an indicator.
e. Name any two indicators mostly in use.
f. What is standard solution?
g. What is end point?
h. Tell the principle of volumetric analysis.
i. What is normality?
j. Define acidimetry?

EXPERIMENT-6

Aim of the Experiment :-

Identification of Unknown Acid Radicals.

Objective of the Experiment:-

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

a) To know the preliminary list done to identify the acid radicals.
b) Know the confirmatory tests of negative ions/ acid radicals.

Apparatus Required :-

i. Test tubes
ii. Test tube holder
iii. Test tube brushes
iv. Dropper
v. Gas burner
vi. Spatula

Chemical Required :-

a. Given Salt b. Various reagents c. Litmus paper

Theory :-

- Salt is defined as a crystalline compound which is formed by complete neutralization of aqueous strong acid with an aqueous strong base.

$$\underset{\text{(Strong base)}}{\text{NaOH}} + \underset{\text{(Strong acid)}}{HCl} \xrightarrow{neutralization} \underset{\text{(Salt)}}{NaCl} + \underset{\text{(Water)}}{H_2O}$$

In other words, Salt are crystalline compounds made up of negative & positive ions. The positive part/ cations comes from a base while the negative part/anion comes from an acid.

- **Radical** :- It is one or group of atoms having positive or negative charge over it. Radicals are of two types : Basic radical & Acid radical.

Metals form basic radicals & non-metals form acid radicals. Positively charged ions are called as “basic radicals” & negatively charged ions are called as “acid radicals”.
The acid radicals prescribed for the diploma engineering (1st /2nd semester) are given below:-

Acid Radicals :- CO_3^{2-} (Carbonate), S^{2-} (Sulphide)
Cl^- (Chloride), NO_3^{2-} (Nitrate)
SO_4^{2-} (Sulphate)

Preliminary Test:-

a) Colour of the salt _____. (White/Coloured)
b) Solubility of the Salt _____ . (Soluble in water/hot water/ dil. HCl/Conc. HCl)
c) Structure _____ . (crystalline/amorphous)
d) Odour of the salt _____ . (odour/odourless)

Identification of Acid Radicals

The acid radicals have been classified into three groups depending upon their reactions with certain reagents.

Classification of Acid radicals :-

Group I	Anions	Group reagents
I.	CO_3^{2-}, SO_3^{2-}, S^{2-}, NO_2^-	Dil. HCl
II.	Cl^-, Br^-, I^-, NO_3^-	Conc. H_2SO_4
III.	SO_4^{2-}	Dil. HCl + $BaCl_2$
	PO_4^{3-}	Conc. HNO_3 + ammonium molybdate

Test with dil. HCL

Experiment	Observation	Inference
About 1cc of dil. HCl is added in a clean & dry test tube. To this a pinch of salt is added	a. Effervescence tooks place with evolution of gas (colourless & odourless) which extinguishes a burning stick. b. Effervescence tooks place with the evolution of colourless gas having rotten egg smell. c. No effervescence.	a. CO_2 gas coming out from carbonate It may be CO_3^{2-}. Then proceed to lime water test. b. H_2S gas coming out from sulphide. It may be S^{2-}. Then proceed to lead acetate test. c. CO_3^{2-}, S^2 are absent

Test with conc. H_2SO_4

Experiment	Observation	Inference
A pinch of salt is taken in a clean & dry test tube. Then 2-3 drops of colc. H_2SO_4 is added to it. Then warm slightly.	a. Effervescence tooks place with evolution of colourless gas which fumes in moist-air is produces a dense white fumes when a glass rod dipped in conc. NH_4OH is shown to the above gas. b. Effervescence takes place with the evolution of brown fumes having pungent smell. Then a few pieces of Cu turning are added & heated. Deep brown vapours are produced & the solution turn green.	a. HCl gas is coming out. Cl^- may be present. Then proceed to test for Cl. b. It may be nitrate NO_3^- . Then proceed to test for nitrate (Brown ring test)

Test for Sulphate (SO_4^{2-})

Experiment	Observation	Inference
a. About 1ml of salt solution is taken in a clean test tube & it is acidify with dil. HCl. Then $BaCl_2$ is added to it. b. A little of the above ppt is taken in a test tube. To it conc. HCL is added & boiled	a. A white ppt is formed. b. Precipitate is soluble.	a. SO_4^{2-} may be present. b. SO_4^{2-} is confirmed.

Reaction of SO_4^{2-}:-

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

Test for Carbonate (CO_3^{2-})

Experiment	Observation	Inference
a. About 1cc of dil. HCl is taken in a test tube. It is gently warmed & little salt is added to it. b. A little more salt is added to the above test tube & evolved gas is passed through the lime water with the help of a delivery tube.	a. Effervescene takes place with evolution of colourless & odourless gas. b. At first white turbidity(milk color) appears which disappears with excess passing of the gas.	a. CO_3^{2-} may be present. b. CO_3^{2-} is confirmed.

Reaction of CO_3^{2-}:-

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + CO_2 + H_2O$$

$$Ca(OH)_2\ CO_2 \longrightarrow CaCO_3 + 2H_2O$$

$$CaCO_3 + H_2O + CO_2 \longrightarrow Ca(HCO_3)_2$$

Test for Sulphide (S^{2-})

Experiment	Observation	Inference
a. A pinch of salt is taken in a clean & dry test tube. About 2ml of dil.HCl/H_2SO_4 is added to it. Then it is warmed.	a. Effervescene takes place with evolution of colourless gas having rotten egg smell. $Na_2S + H_2SO_4 \downarrow Na_2SO_4 + H_2S$	a. H_2S gas is coming out from sulphide.
b. A filter paper is soaked with lead acetate soln. is shown to above gas.	$Na_2S + 2HCl \longrightarrow 2NaCl + H_2S$	b. S^{2-} is confirmed due to formation of PbS.
	b. The filter paper turned black due to formation of PbS. $(CH_3COO)_2Pb + H_2S \downarrow PbS + CH_3COOH$	

Test for nitrate (NO_3^-)

Experiment	Observation	Inference
a. A pinch of salt is taken in a clean & dry test tube & to it a piece of Cu turning are added. Then 4-5 drops of conc. H_2SO_4 is added to the mixture & heated.	a. Copious brown fumes are evolved.	a. This is due to formation of NO_2 gas.
b. Brown ring test About 2ml of salt solution is taken in a testube. To it equal volume of conc. H_2SO_4 is added. Then the test tube is cooled perfectly under tap water. Then slowly add freshly prepared ferrous sulphate solution through the side of the test tube. .	b. A brown ring is obtained at the junction of the two liquid. $FeSO4 + NO \longrightarrow FeSO4.NO$ $KNO_3 + H_2SO4 \longrightarrow KHSO_4 + HNO_3$ $6FeSO_4 + 2HNO3 + 3H_2SO_4 \longrightarrow 3Fe_2SO_4 + 2NO + 4H_2O$ $FeSO4 + NO + 5H2O \longrightarrow [Fe(H_2O)_5NO]SO_4$	b. Nitrate is confirmed.

Test for Chloride(Cl^-)

Experiment	Observation	Inference
a. A pinch of salt is taken in a clean & dry test tube. Then 3 drops of conc. H_2SO_4 is added by the side of the test tube.	a. Effervescene takes place with the evolution of the colourless gas which fumes in moist air.	a. Cl^- may be present.
b. A glass rod dipped in conc. NH_4OH is shown to the above test tube.	b. Dense white fumes are produced. $NH_4OH + HCl \longrightarrow NH_4Cl + H_2O$	b. Chloride may be present.
c. 1cc of salt solution is prepared in a clean and dry test tube & the solution is acidified by adding 0.5ml of dil. HNO3. To this test tube silver nitrate solution is added & shaken well.	c. Curdy white precipitate is formed.	c. Chloride may be present.
d. (a) The above ppt. Is allowed to stand & the liquid is decanted off. The ppt. Is washed 3 times with water & divided into 3 parts.		
Part-1: To the 1st part dil.HNO_3 is added & heated.	i. Precipitate doesn't dissolves.	
Part-2:To the 2nd part dil.NH_4OH is added & shaken well.	ii. The precipitate dissolves.	
Part-3:To the 3rd part conc.NH_4OH is added. Drop by drop with shaking.	iii. The precipitate dissolves.	
(b) To part-2 & part-3 test tube dil.HNO3 is added.	(b) The precipitate reappears.	d. Chloride is confirmed. $NaCl + AgNO3 \longrightarrow AgCl + NaNO3$ $AgCl + HNO_3 \longrightarrow$ no change $AgCl + NH_4OH$ $[Ag(NH_3)_2Cl]$(diammine silver choride)

EXPERIMENT-7

Aim of the Experiment :-

Identification of Unknown Basic Radicals.

Objective of the Experiment:-

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

c) To know the preliminary list done to identify the basic radicals.
d) Know the confirmatory tests of positive ions/ basic radicals.

Apparatus Required :-

i. Test tubes
ii. Test tube holder
iii. Test tube brushes
iv. Dropper
v. Gas burner
vi. Spatula

Chemical Required :-

b. Given Salt
b. Various reagents
c. Litmus paper

Theory :-

- Salt is defined as a crystalline compound which is formed by complete neutralization of aqueous strong acid with an aqueous strong base.

$$\underset{\text{(Strong base)}}{\mathrm{NaOH}} + \underset{\text{(Strong acid)}}{HCl} \xrightarrow{neutralization} \underset{\text{(Salt)}}{NaCl} + \underset{\text{(Water)}}{H_2O}$$

In other words, Salt are crystalline compounds made up of negative & positive ions. The positive part/ cations comes from a base while the negative part/anion comes from an acid.

- **Radical** :- It is one or group of atoms having positive or negative charge over it. Radicals are of two types : Basic radical & Acid radical.

Metals form basic radicals & non-metals form acid radicals. Positively charged ions are called as “basic radicals” & negatively charged ions are called as “acid radicals”.
The acid radicals prescribed for the diploma engineering (1^{st} /2^{nd} semester) are given below:-
Basic Radicals :- Zn^{2+}(zinc ion),Na^{+}(sodium ion), Mg^{2+}(magnesium ion),NH^{4+}(ammonium ion),Al^{3+}(aluminium ion),Ca^{2+}(calcium ion),K^{+}(potassium ion).

Preliminary Test:-

a) Colour of the salt _____. (White/Coloured)
b) Solubility of the Salt _____ . (Soluble in water/hot water/ dil. HCl/Conc. HCl)
c) Structure _____ . (crystalline/amorphous)
d) Odour of the salt _____ . (odour/odourless)

A. Dry test for Basic Radicals

1. Dry test tube heating

Experiment	Observation	Inference
A pinch of salt is taken in a clean & dry test tube. The test tube is held by a test tube holder. It was first heated gently & then strongly heated for 5 mins in non-luminuos flame.	a. The salt decrepitates(producing cracking sound). b. The salt melts on heating & solidifies on cooling. c. The salt volatizes out completely forming a white sublimate. d. The salt swells on heating. e. A white infusible incandescent mass is left. f. The salt changes its colour. (i) yellow when hot & white when cold. (ii) Yellow when hot & cold.	a. May be crystalline salts. b. May be alkaline or alkaline earth metals salts. c. It is volatile salt.(soda lime test to be performed). d. It may be aluminium salt. e. It may be Mg^{2+},Sn^{2+}/alkaline earth metals. f. (i) May be Zn salts. (ii) May be Pb salts.

2. Soda lime test(This test is performed for volatile salts)

Experiment	Observation	Inference
A pinch of salt is taken in a watch glass & a little amount of soda lime(CaO+NaOH) & one or two drop of water is added to it. Then it is rubbed with the fingertips.	A colorless gas having smell of ammonia evolves.	May be NH_4^{2+}ammonium salts.

3. Charcoal cavity test

Experiment	Observation	Inference
A little of the salt is taken in the charcoal cavity & it is moistened by one or two drops of water. Then it is heated by oxidising	a. The salt decrepitates(cracking sound). b. The salt deflagrates(catches	a. May be crystalline salt(perform flame test). b. May be NO_2^-,NO_3^-salt. c. May be alkaline

flame with the help of a blow pipe.	fire). c. The salt fuses(melts) on heating & sinks into the charcoal cavity. d. Leaves behind a infusible incandescent white residue. e. The color of the salt yellow when hot & white when cold.	salts.(perform flame test). d. May be Al^{3+},Mg^{2+} or alkaline earth.(perform cobalt nitrate test) e. It may be Zn^{2+} salts.

4. <u>Cobalt nitrate test</u>

Experiment	Observation	Inference
A drop of cobalt nitrate solution is added to the infusible incandescent residue obtained from the charcoal cavity test. Then it is heated in the non-luminous flame for 2 minutes.	a. Blue mass is obtained. b. Green mass is obtained. c. Pink mass is obtained. d. Grey mass is obtained.	a. May be Al^{3+}. b. May be Zn^{2+}. c. May be Mg^{2+}. d. May be Ca^{2+}. Flame test to be performed.

5. <u>Flame test(this test is performed for alkali [Na^+,K^+] or alkaline earth metal[Ca^{2+},Ba^{2+},Sr^{2+})</u>

<table>
<tr><th>Experiment</th><th>Observation</th><th>Inference</th></tr>
<tr><td>A nichrome wire is rubbed with sand paper. Then it is strongly heated in a non-luminous flame of the burner & at once dipping it in conc. HCl acid & touch it with a little of the powdered salt. Now heat it in non-luminous flame & the colour of the flame is noted.</td><td><table><tr><th>Colour observed in naked eye</th><th>Colour through double blue glass</th></tr><tr><td>a. Golden yellow
b. Brick red
c. violet</td><td>a. colourless
b. not to be viewed
c. crimson red.</td></tr></table></td><td>a. May be Na+.
b. May be Ca2+.
c. May be K+.</td></tr>
</table>

B. <u>Wet test for Basic Radicals</u>

For the identification of basic radicals by wet tests, they have been classified into six groups depending on their reactions in solution with different reagents. A list showing the groups, basic radicals & group reagents is given below.

<u>Classification of Basic radicals :-</u>

Group I	Anions	Group reagents
I.	• Pb^{2+},Ag^+,Hg_2^{2+}	• Dil. HCl
IIA.	• Hg^{2+},Bi^{2+},Cu^{2+},Cd^{2+} As^{3+},Sb^{3+},Sn^{2+} or Sn^{4+}	• H_2S in presence of dil.HCl.
IIB.	• Fe^{3+},Al^{3+},Cr^{3+}	• NH_4OH in presence of NH_4Cl.
IIIA.	• Co^{2+},Ni^{2+},Zn^{2+},Mn^{2+}	• $(NH_4)_2CO_3$ in presence of NH_4Cl & NH_4OH.
IIIB.	• Ca^{2+},Ba^{2+},Sr^{2+}	• No particular reagents.
IV.	• Mg^{2+},NH_4^+,Na^+,K^+	
V.		

1. **Wet test for Group-I radicalsl[Pb^{2+},Ag^{+},Hg_2^{2+})**

Experiment	Observation	Inference
About 1ml of salt solution is taken in a given test tube & dil.HCl is added.	a. White precipitate is formed. The above precipitate formed is then boiled, the ppt. is soluble & reappeared on cooling. b. No white ppt. is formed.	a. Pb2+ may be present. b. Grp-I radicals are absent.

2. **Wet test for Group-IIA radicals:[Pb^{2+},Cu^{2+},Hg^{2+}] & Group-IIB radicals:[As^{3+}, Sn^{2+}, Sb^{3+}]**

Experiment	Observation	Inference
About 2ml of salt solution is taken in a given test tube & dil.HCl is added. Then H_2S gas is allowed to pass through it.	Black ppt. is formed. No black ppt. is obtainted	Group II may be present. Group-II radicals are absent.

3. Wet test for Group-IIIA radicals:[Fe^{3+}, Al^{3+},Cr^{3+}]

Experiment	Observation	Inference
To 2ml of salt solution, solid NH_4Cl is added till solution is saturated then dil. NH_4OH is added till alkaline.	Gelatinous white ppt. is formed.	Group II may be present.
	No ppt. is obtainted	Group-II radicals are absent.
Test for Aluminium(Al^{3+})		
1. To 1ml of salt solution, solid NH_4Cl is added till solution is saturated then dil. NH_4OH is added till alkaline.	1. Gelatinous white ppt. is formed.	1. Al3+ may be present.
2. 1ml of salt solution is taken in a clean test tube & to it dil.NaOH is added drop by drop till excess.	2. Gelatinous white ppt. is first formed which is soluble inexcess of NaOH.	2. Al3+ is confirmed.

4.Wet test for Group-IIIB radicals:[Sn^{2+}, Mn^{2+},Co^{2+},Ni^{2+} ,Zn^{2+}]

Experiment	Observation	Inference
To about 2ml of salt solution, solid NH_4Cl is added till the solution is saturated then dil. NH_4OH is added till alkaline & H_2S gas is allowed to pass through it.	white ppt. is formed.	Zn^{2+} may be present.(Confirmatory test to be done)
	No ppt. is obtainted	Group-IIB radicals are absent.

Test for Zinc(Zn^{2+}) About 1ml of salt solution is taken in a clean test tube & to it dil.NaOH is added drop by drop till excess.	White ppt. first formed is soluble when excess of NaOH is added in excess dropwise.	Zn^{2+} is confirmed.

5.Wet test for Group-IV radicals

Experiment	Observation	Inference
To about 2ml of salt solution, solid NH_4Cl is added till solution is saturated then dil. NH_4OH is added till alkaline. Then saturated solution of($(NH_4)_2CO_3$ is added to it.	A white ppt. is formed.	Ca^{2+} may be present.
	No ppt. is obtainted	Group-IV radicals are absent.
Test for calcium(Ca^{2+}) 1. The above white ppt. is dissolved in minimum amount of dil.CH3COOH, boiled to remove CO_2 & then ammonium oxalate soln. is added to it.	White ppt. of CaC_2O_4 soluble in mineral acid but insoluble in CH_3COOH is obtained.	Ca^{2+} is confirmed.

6.Wet test for Group-V radicals:[Mg^{2+},Na^+,K^+,NH_4^+]

Experiment	Observation	Inference
Test for potassium(K^+) About 1ml of salt solution is treated with 2 drops of $Co(NO_3)_2$ soln. followed by the addition of solid $NaNO_2$ & dil.CH_3COOH.	Yellow ppt. is formed.	K^+ is confirmed.

	No yellow ppt. is obtainted	K^+ is absent.
Test for magnesium(Mg^{2+})		
1. To about 2ml of salt solution, solid NH4Cl is added till saturation. And then dil. NH4OH is added till alkaline & disodium hydrogen phosphate is also added to it.	1. White ppt. is formed.	1. Mg2+ may be present.
2. About 2ml of salt solution is taken in a test tube & dil. HCl is added. Then 2-3 drops of magneson reagent is added. Finally the soln. Is made alkaline with NaOH.	2. A blue ppt. is obtained.	2. Mg2+ is confirmed.
Test for Ammonium($NH4^+$)		
1. About 1ml of salt solution is taken in a test tube & to it 5 drops of NaOH soln. is added followed by addition of little Nessler's reagent.	A brown ppt. is formed.	NH_4^+ is confirmed.

Viva-voce question

1. What is analytical chemistry?
2. What is understand by qualitative & quantitative analysis?
3. What is salt? What do you mean by acid & basic radical?
4. What is an acid?
5. What is a base?
6. What is an alkali?
7. Name the burner which is used in laboratory?
8. Why is stoppered bottle used for lime water?
9. Is there any other gas like CO2which can turn lime water milky?
10. What is brown ring test?

11. What are the principles on which separation of basic radicals into groups is based?
12. What do you understand by solubility product & what importance it has got in qualitative analysis?
13. What is common ion effect?
14. Why salts give colour in flame test?