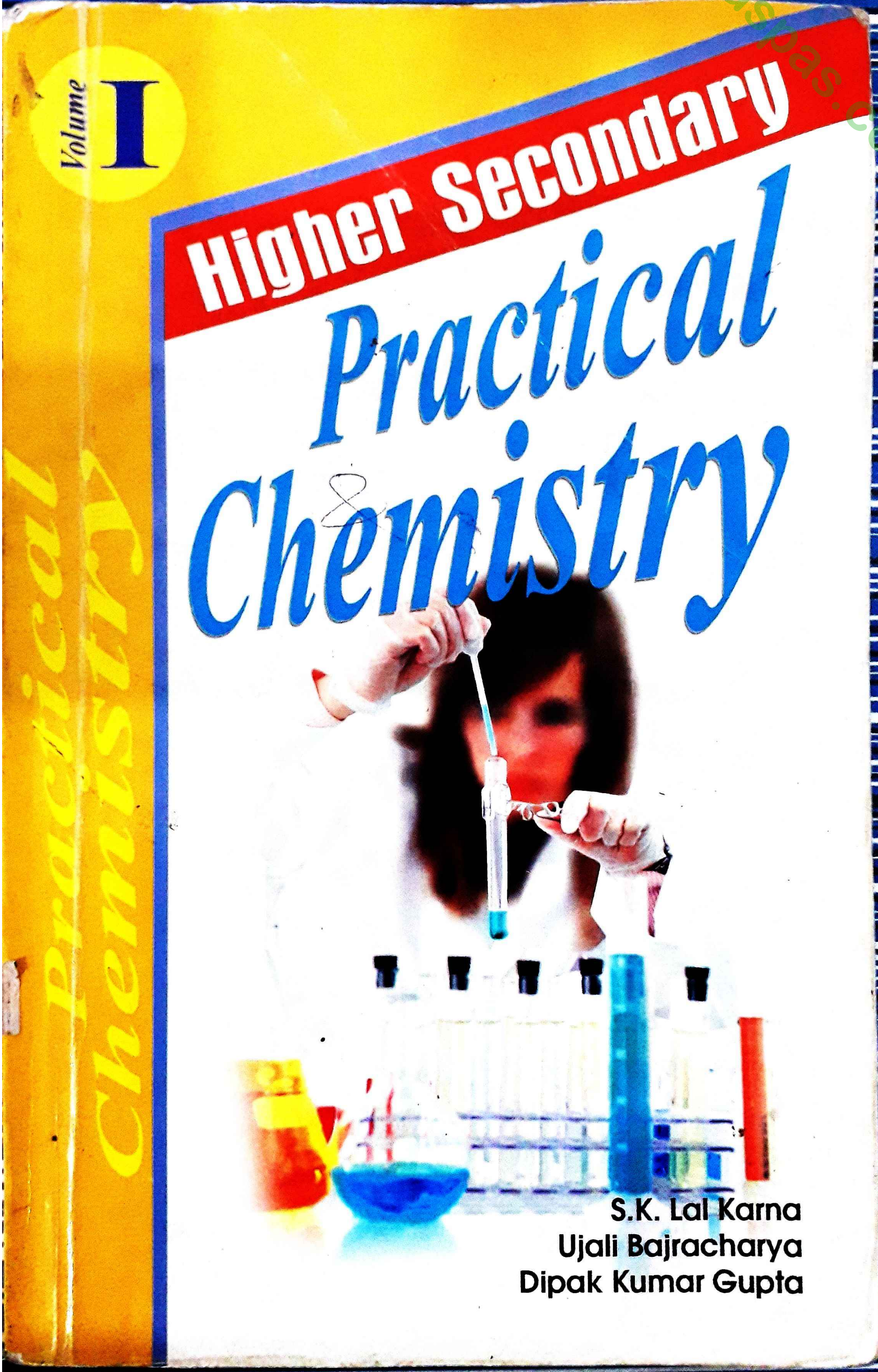
Volume I
Higher Secondary
Practical Chemistry
S.K. Lal Karna
Ujali Bajracharya
Dipak Kumar Gupta
Practical Chemistry

Higher Secondary

Practical Chemistry

Volume I

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Rabean Subedi

Dedication

Humbly dedicated to
The memory of my beloved
parents Ramapati Lal Karna and
Gaurídevi Karna who are no more
in this world

–S.K. Lal Karna

PREFACE TO THE THIRD EDITION

We are extremely happy to present the third edition of the Higher Secondary Practical Chemistry book for grade XI and XII. The main feature of this edition is the inclusion of experiments for class XI in volume I and experiments for class XII in volume II. The salt analysis is kept in volume I so that students feel comfortable while doing the experiments.

We are confident that if a student goes through the book thoroughly he/she will be able to carry experiments very smoothly and can answer almost all questions.

The sincere thanks go to Dr. Amar Prasad Yadav, Mr. Kanchan Sharma, Mr. Bijuli Rana, Mr. Binay Sah, Mr. Dipendra Singh Dhami, Mr. Sharad Sundar, Ms. Dipika Regmi, Ms. Shova Neupane, Mrs. Shusila Sanjel, Mr. Lalit Bahadur Chanda and all our colleagues for their valuable suggestions and criticism about the book.

We also express our appreciation to Mr. Shanta Gautam of Vidyarthi Pustak Bhandar, Mrs Sarita Thapa for computer works. We have tried our best to remove errors/misprints in the book. Finally any suggestion and criticism for the improvement of the book are highly appreciated.

Authors

PREFACE TO THE SECOND EDITION

It gives us immense pleasure to bring out the **Higher Secondary Practical Chemistry** book for grade XI and XII. The book is thoroughly revised and typographical errors are removed. It presents all experiments in simple form. It also includes viva voice.

We are very thankful to Mrs. Lila Gnawali, Mr. Sanjay Singh, Ganga Bir Rai, Sushil Pokharel, Netra Prasad Subedi, Anju Das, Maya Das, Bhoj Raj Paudel and all our colleagues for their valuable suggestions and criticism about the book.

We also express our appreciation to Mr. Shanta Gautam of Vidyarthi Pustak Bhandar, Mr. Nariswor Gautam and Mrs. Sarita Thapa for computer works. Finally, any suggestion and criticism for the improvement of the book are highly appreciated.

Thanks

Authors

PREFACE

It is a great pleasure for us to bring out this practical book on the laboratory and practical aspects of Grade XI and XII. This book covers the latest curriculum prescribed by the Higher Secondary Education Board of Nepal. The book is written in a simple language. The subject matter has been lucidly presented. In short the book has all the qualities to meet the expectations of the teaching and learning communities. Therefore, it is our belief that both teachers and students will be pleased to welcome and use this new book.

We have aimed here to provide a sound theoretical knowledge of subject matter to the students beforehand the experiment. At the same time, the theory and procedure of each experiment are given in such a clear systematic way with viva voice that students will find the book to be more informative and more effective to perform experiment with confidence on the laboratory room that students will be benefitted with this book in theory and practical both.

We further hope that this book will help my fellow teachers to instruct students more easily in practical classes, although, we request colleague, friends and students for the creative suggestions, so that we can make the book more perfect in coming editions.

We are grateful to Prof Dr. Mohan Bikaram Gewali, Rajaram Pradhanang, Tulshi Pathak, P. P. Wagley, Associate Professor Motikaji Sthapit, Nagendra Mani Khadka, Dr. Kedar Nath Ghimire, P.C. Lama, S.P. Singh, Ramesh Silwal, B. Tamrakar, Dr. M.P. Baral, Dr. R.N. Jha, Surendra Gautam, Rajendra Gautam, Kanchan Sharma, B. Rana, Shiva P. Subedi, Rajeeb Chaudhary, Sanjay Singh, Sharmila Bajracharya, Yubraj Adhikari, Dr Daman Gautam, Uday Jha and Rabindra Tuladhar for their active support and constant encouragement.

Authors

SYLLABUS

Chemistry Practical Paper for Grade XI

F.M. =25
P.M. =10

Students are required to secure the pass mark in the practical paper separately. The following is the list of experiments. The students are required to perform in the practical classes in Grade XI.

A. Experiments based on laboratory techniques

1. To separate the insoluble component in pure and dry state from given mixture of soluble and insoluble solids (NaCl and sand)
2. To separate volatile component from the given mixture of volatile and nonvolatile (camphor + sand) components.
3. To separate the mixture of two soluble solids by fractional crystallization ($KNO_3 + NaCl$)
4. To prepare a saturated solution of impure salt and obtain the pure crystals of the same salt by crystallization [$CuSO_4$ (impure)].
5. To separate the component of a mixture of two insoluble solids ($CaCO_3$ + sand)
6. To obtain pure water from given sample of water (distillation)

B. Experiment to study the different reactions: (Neutralisation, precipitation, redox reaction, electrolysis)

7. To perform precipitation reaction of $BaCl_2$ and H_2SO_4 and obtain solid $BaSO_4$ (pure and dry).

8. To neutralize sodium hydroxide with hydrochloric acid solution and recover the crystal of sodium chloride.

9. To test the ferrous ions in the given aqueous solution and oxidize it to ferric ion (ferrous → Ferric ion) by redox reaction.

10. To study the process of electrolysis and electroplating.

C. Experiments on quantitative analysis

11. To determine the equivalent weight or weight of metal by hydrogen displacement method.

12. To determine the solubility of the given soluble solid at laboratory temperature.

13. To determine the relative surface tension of unknown liquid by drop count method.

14. To study the rate of flow of liquid through Ostwald's viscometer and determine the relative viscosity of unknown liquid.

D. Experiments on preparation of gas and study of properties

15. To prepare and collect hydrogen gas and study the following properties
    a. solubility with water, its colour and odour
    b. litmus test
    c. burning match stick test
    d. reducing properties of nascent hydrogen

16. To prepare and collect ammonia gas and investigate the following properties.
    a. solubility with water, its colour and odour
    b. litmus test
    c. action with copper sulphate solution
    d. with mercurous nitrate paper

17. To prepare CO_2 gas and investigate the following properties:

a. solubility, colour and odour

b. litmus paper test

c. lime water test

d. action with burning magnesium ribbon

18. To study the properties of hydrogen sulphide. Study its physical, analytical and reducing properties.

19. To study the following properties of sulphuric acid

a. solubility with water

b. litmus paper

c. Precipitating reaction

d. dehydrating reaction

E. Experiments on qualitative analysis

20. To detect the basic radicals and acid radicals of given salt by dry way and wet ways.

Basic radicals: Zn^{++}, Al^{+++}, NH_4^+, Na^+, Mg^{++} etc.

Acid radicals: CO_3^{--}, SO_4^{--}, NO_3^-, Br^-, I^-, Cl^- etc.

Note: Experiment number from 1 to 19 requires one practical period for each experiment and experiment No. 20 requires four practical periods.

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Chapter-3

Chapter-4

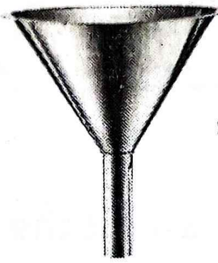
Chapter-5

Chapter-6

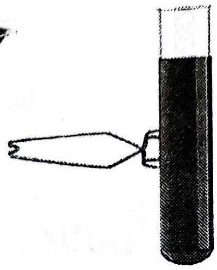
Some Common Laboratory Apparatus

Beaker

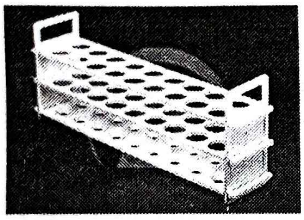
Test tube



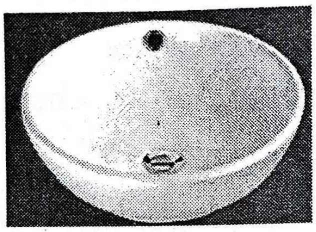
Funnel



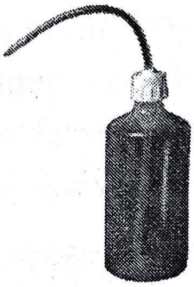
Test tube holder



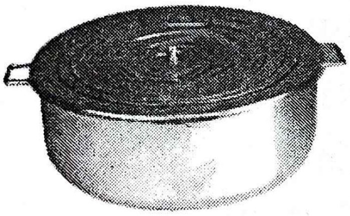
Test tube stand



Porcelain basin

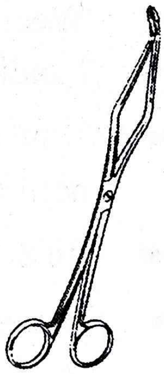


Wash bottle

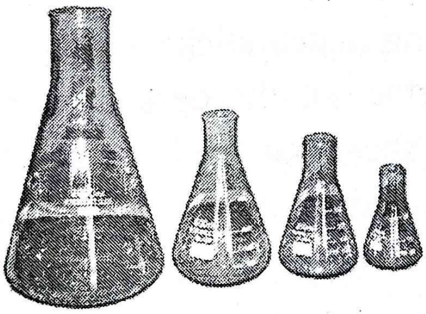


Water bath

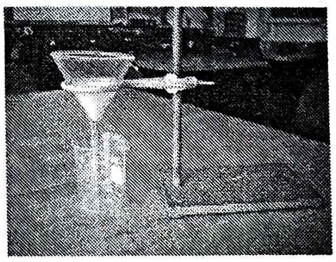
Round bottom flask



Tongs



Conical flasks



Filter stand

Chapter–one

A. Introduction about the Chemistry Laboratory

Essential precautions should be maintained while working in a chemical laboratory, which reduces chance of accidents. Every one (teachers, students, lab assistants) working in a chemical laboratory should follow instructions such as

- Always wear apron and shoes while working in the chemistry laboratory
- Wear protective eye goggles and gloves while handling dangerous chemicals
- Never taste and touch any chemicals without the permission of instructors (teachers)
- Be economical with reagents, water and fuel gas
- Never work alone in laboratory
- Never throw sodium or potassium metal in the sink (because it is highly reactive with water)
- Never keep the burning match sticks and hot fused glass tube on the surface of the desk, rather keep them on the asbestos sheet and finally throw match sticks, broken glasses and insoluble waste materials in waste basket.
- Keep the bench tops free from chemical and waste materials

- As soon as you have finished with a reagent bottle replace its stopper and put it back into its proper position
- Do not contaminate one chemical with the other
- If accident happens to get on to our skin, eyes or mouth, wash it out thoroughly with water.
- If an accident happens, report it immediately to the laboratory in-charge so that first aid for the safety can be done in time.
- Never add water into concentrate acids but add Conc. acids like H_2SO_4, HCl, HNO_3 etc. in water with constant stirring for dilution.
- Do not bring any inflammable liquids such as alcohols, ether, carbon disulphide, etc. near the flame.
- Before entering the laboratory find the position of safety measures and before leaving the laboratory keep all things in the proper order.
- Do not move about and make noise in the laboratory unnecessarily.
- Leave your apparatus clean and tidy.

B. Laboratory Safety and First Aid

Suitable safety measure should be taken while working in the laboratory because most of injuries (accidents) occur due to the ignorance and carelessness of the students (lab workers). The injuries may be caused by burns, acids, alkalies, metals and other chemicals, so they should have some idea of the preliminary treatment for some common laboratory injuries (accidents).

First Aid Kit

A first aid kit should be kept ready in the laboratory and should contain the following materials:

1% boric acid solution, antiseptic, burnol, 1% acetic acid, 1 to 5% sodium bicarbonate solution, tincture of iodine (antiseptic and analgesic), Vaseline, lime water, bandages, sterile cotton and gauze leucoplasts, scissors, delicate forceps, glycerine, etc.

1. **Cuts and wounds**

   Remove the glass piece with forceps. Apply some dettol or rectified spirit (95% ethyl alcohol, 5% water), and cover with a piece of leucoplast. For serious bleeding, send the patient to the hospital.

2. **Burns**

   a. **Dry heat burn:** If the burn is minor and involves only the superficial tissue, use ice or cold water for some time and wash with soap water. At last apply burnol and bandage with sterile cotton. If burn is severe or deep, send to the hospital.

   b. **Moist heat burn (due to boiling water, steam, etc.):**

   Treatment is similar to dry heat.

   c. **Chemical burns:**

   i. **From corrosive acids:** Wash the affected area with plenty of water for a five minutes, followed by washing with 5-8% sodium bicarbonate solution. Then apply burnol and bandage. If the burn is severe send the patient (student) to the hospital.

ii. **From corrosive alkali:** Wash the affected area with water, followed by 1% acetic acid solution. Then apply antiseptic, and bandage.

iii. **By sodium burn:** Remove any visible sodium piece with forceps and wash with water, followed with 1% acetic acid. Apply burnol and bandage.

iv. **For bromine burns:** Wash the affected portion with petrol or carbon disulphide. Apply glycerine and finally burnol and bandage.

v. **For phosphorus burn:** Wash with cold water and immerse in aqueous silver nitrate solution. Apply burnol and bandage.

3. **Eye accidents**

a. **Acid in eye:** Wash the eye with plenty of water followed with 1% sodium bicarbonate solution, finally wash with water again if severe, send to the hospital.

b. **Alkali in eye:** Wash the eye with plenty of water, followed by 1% boric acid solution, wash again with water.

c. **Glass piece in eye:** Do not rub the eye. Remove the glass piece with delicate forceps or clean handkerchief. Wash with water and put a drop of olive oil or castor oil in the eye.

4. **Poisoning**

a. **Poison in the mouth (oral) but not swallowed**
Spit and wash the mouth several times with water

b. **Poison swallowed (orally)**

i. **Mineral acids:** Give plenty of water and then little amount of magnesia [($Mg(OH)_2$] or lime water. Next give milk and white of egg. No emetic (vomiting drug) should be given.

ii. **Oxalic acids :** Give excess of magnesia or lime water and milk. Do not take emetics.

iii. **Caustic alkalies :** Drink excess of water followed by lemon juice, orange juice or vinegar. Do not take emetics.

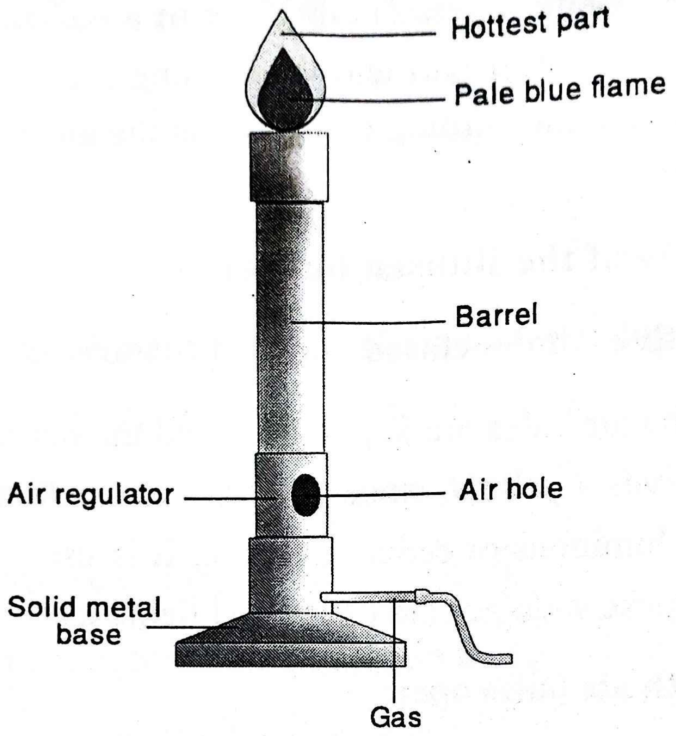
5. **Gas poisoning**

a. **Poisoning is due to CO, CO_2, H_2S etc.:** Keep the patient in fresh air and loosen the clothing at the neck. Give artificial respiration. In severe cases, send to hospital.

b. **Poisoning due to chlorine or bromine:** Give inhalation of dilute ammonia followed by sodium bicarbonate gargle.

C. Bunsen burner

The most commonly used heating device in the laboratory is the Bunsen burner, which was discovered by German Scientists Robert Bunsen in 1855. It mainly consists of three parts:



Bunsen Burner

1. The base having a solid tube, which communicates inside with a jet.
2. The barrel having a single or a couple of air holes near the lower end.
3. The air regulator fitted to the lower end of the barrel.

The burner burns a mixture of fuel gas and air. The supply of fuel gas is controlled by the gas tap and the supply of air by the air regulator.

Using of Bunsen burner

First gas tap is put on from the cylinder. Now the gas tap of the burner is put on and burner is lighted. When gas is escaped through jet, there is fall of pressure and air is sucked in through the air holes. The mixture of air and gas begins to burn from the lower part and the burning completes from the lower part and the burning completes at the upper most part of the flame.

The flame of the Bunsen burner

1. **With air holes closed**

   If the air holes are kept closed and the burner is lighted, it gives a yellow, smoky flame like candle flame called the luminous or reducing flame. It is used for reducing purpose such as borax bead and flame test.

2. **With air holes open**

   If the air holes are gradually opened in the fuel gas, while passing up the barrel, sucks in the air and burns with a nonluminous flame or oxidizing flame. The outer non luminous blue flame is the hottest zone (1540 - 1560°C). This zone is used for normal heating purpose. This normal Bunsen flame consists of two zones only.

   a. The inner blue zone containing the region of unburnt gas.
   b. The outer nonluminous zone which is the hottest part.

Note: While using the burner for heating purpose, do not close the air holes completely

Format for Writing in the Practical Notebook

Experiment No.: 1

Page No.1

Date: 069/1/7

Title of the experiment (object) should be written in block letters

Apparatus required : (i) (ii) (iii)

Chemical required : (i) (ii) (iii)

Theory: Written in present tense (main things)

Procedure: Past tense and passive voice

Experiments and observations are written in past tense and passive voice

Inference is written in present tense.

Diagram should be drawn with a pencil on the left hand page with properly well labelled.

Conclusion:

Precaution :

Experiment No. I

Object : TO STUDY THE FOLLOWING CHEMICAL REACTIONS

1. Copper sulphate solution + ammonia solution drop by drop till excess.
2. Copper sulphate solution + sodium hydroxide solution
3. Zinc sulphate solution + sodium hydroxide solution drop by drop till excess.
4. Dil. hydrochloric acid + silver nitrate solution + ammonia solution drop by drop till excess + nitric acid solution.
5. Sulphuric acid solution + barium chloride solution drop by drop.
6. Sodium carbonate solution + barium chloride solution drop by drop.
7. Potassium chromate solution + barium chloride solution
8. Potassium dichromate acidified with dilute sulphuric acid + sodium sulphite solution
9. Mercurous nitrate solution + ammonia solution
10. Mercurous nitrate solution + dil. hydrochloric acid solution + ammonia solution
11. Mercuric chloride solution + potassium iodide solution drop by drop till excess + ammonia solution

Apparatus required

i. Test tubes
ii. Test tube holders
iii. Test tube stand

Chemical required

i. $CuSO_4$ solution
ii. $ZnSO_4$ solution
iii. Dil.$H_2 SO_4$ solution
iv. NH_4OH solution
v. NaOH solution
vi. $Hg_2(NO_3)_2$ (mercurous nitrate solution)
vii. $AgNO_3$ solution
viii. Na_2CO_3 solution
ix. $BaCl_2$ solution
x. $K_2Cr_2O_7$ (potassium dichromate solution)
xi. K_2CrO_4 (Potassium chromate solution)

Theory

Transformation of one type of chemical substances (reactants) into another type of chemical substances (products) having different new properties is known as chemical reaction. Such type of transformation takes place by simple contact, heat, light, pressure, electricity, etc.

The completion of chemical reaction can be observed by different properties such as change in colour, formation of precipitate, evolution of gas, increasing or decreasing the temperature of the resulting solution. Thus chemical reactions are chemical change. They are of following types:

1. **Addition or synthesis reaction:** Such type of reaction, in which two or more simple molecules unite to each other to give a new molecule is known as addition reaction.

   For example: Hydrogen reacts with chlorine in presence of sunlight to give hydrogen chloride gas.

   $$H_2 + Cl_2 \xrightarrow{\text{sunlight}} 2HCl$$

2. **Decomposition or analysis reaction:** Chemical reaction in which large molecule is decomposed into two or more simple molecules is known as decomposition reaction.

For example: When calcium carbonate (limestone) is heated, we get calcium oxide and carbon dioxide gas

$$CaCO_3 \xrightarrow{\Delta} CaO + CO_2\uparrow$$

3. **Displacement or substitution reaction**

a. **Single displacement or substitution reaction:** Chemical reaction, in which more reactive element displaces less reactive element is known as single displacement reaction.

For example: When granulated zinc is reacted with dilute sulphuric acid, we get zinc sulphate and hydrogen gas.

$$Zn\ (s) + \text{dil. } H_2SO_4 \longrightarrow ZnSO_4 + H_2\uparrow$$

b. **Double displacement or substitution reaction:** Chemical reaction, in which there is mutual exchange of cations (Ag^+, Ca^{++}, Al^{+++}) and anions (Cl^-, Br^-, I^-) is known as double displacement reaction.

For example: When sodium chloride solution is reacted with silver nitrate solution, we get white precipitate of silver chloride and sodium nitrate.

$$Ag^+NO_3^-(aq.) + Na^+Cl^-(aq.) \longrightarrow \underset{\text{White precipitate}}{AgCl\downarrow} + NaNO_3$$

4. **Acid base or neutralization reaction**

It is complete reaction of H^+ ions (hydrogen ion) from acid side and OH^- (hydroxyl ion) from base side to give neutral water.

For example when sodium hydroxide solution is reacted with dilute hydrochloric acid, we get sodium chloride and water (neutral) is known as neutralization reaction.

$$\underset{\text{Base}}{NaOH\,(aq.)} + \underset{\text{acid}}{dil.\ HCl} \longrightarrow NaCl + H_2O\ (Neutral)$$

Or, $H^+ + OH^- \longrightarrow H_2O$

5. **Hydrolysis reaction:** When chemical reaction is carried out through water molecule is known as hydrolysis reaction. It is reverse of neutralization.

For example: When phosphorus trichioride is treated with water, we get phosphorus acid and hydrogen chloride.

$$PCl_3 + 3H_2O \longrightarrow \underset{\text{Phosphorus acid}}{H_3PO_3} + 3HC1$$

Observation Table:

Experiment	Observation	Interference
1. Take about 2-5 cc of $CuSO_4$ solution in test tube and add ammonia solution first dropwise and then excess.	1. At first bluish white precipitate appears, which dissolves in excess of ammonia solution.	1. Bluish white precipitate is due to formation of cupric hydroxide and deep blue colour (indigo) due to the formation of tetramine copper (II) sulphate
Reaction: $CuSO_4\,(aq.) + 2NH_4OH(aq.) \longrightarrow \underset{\text{Bluish white ppt}}{Cu(OH)_2\downarrow} + (NH_4)_2SO_4$ $CuSO_4 + (NH_4)_2SO_4 + NH_4OH \longrightarrow \underset{\text{Tetramine copper sulphate}}{[Cu(NH_3)_4]SO_4} + H_2O$		

Experiment	Observation	Interference
2. Add few drops of NaOH solution dropwise to about 2-5 mL (cc) of $CuSO_4$ solution.	2. Bluish white precipitate appears.	2. Due to formation of cupric hydroxide.

Reaction:

$$CuSO_4 + 2NaOH \longrightarrow \underset{\text{Bluish white ppt}}{Cu(OH)_2\downarrow} + Na_2SO_4$$

Experiment	Observation	Interference
3. Add NaOH solution dropwise in test-tube till excess to about 2-5 mL (cc) of $ZnSO_4$ solution.	3. At first gelatinous white precipitate appears which becomes colourless in excess of NaOH solution.	3. White gelatinous precipitate due to formation of zinc hydroxide, which becomes colourless due to formation of soluble sodium zincate.

Reaction:

$$ZnSO_4\,(aq.) + 2NaOH(aq.) \longrightarrow \underset{\text{Gelatinous white ppt}}{Zn(OH)_2\downarrow} + Na_2SO_4$$

$$Zn(OH)_2 + 2NaOH \longrightarrow \underset{\text{Sodium zincate}}{Na_2ZnO_2} + 2H_2O$$

Experiment	Observation	Inference
4. Take about 2-5 mL of dil. HCl solution in test tube		
a. Add few drops of $AgNO_3$	a. White precipitate appears	a. Due to formation of insoluble AgCl
b. Add NH_4OH (ammonia solution)	b. The white precipitate dissolves to give colourless solution	b. Due to formation of soluble diammine silver chloride
c. Add dil. HNO_3 solution	c. White precipitate reappears	c. Due to the formation of AgCl again

Reaction : $HCl\,(aq) + AgNO_3\,(aq.) \longrightarrow \underset{\text{white ppt}}{AgCl\downarrow} + HNO_3$

$$AgCl + 2NH_4OH \longrightarrow \underset{\text{Diammine silver (I) chloride}}{[Ag(NH_3)_2]Cl} + 2H_2O$$

$$[Ag(NH_3)_2]Cl + HNO_3 \longrightarrow \underset{\text{white ppt reappeared}}{AgCl\downarrow} + 2NH_4NO_3$$

Experiment	Observation	Inference
5. Take about 2-5 mL of H_2SO_4 solution in test tube and add $BaCl_2$ solution till the formation of precipitate and then add HCl acid solution.	5. White precipitate appears, which do not dissolve in dil. HCl acid	5. Due to formation of white precipitate of $BaSO_4$.

Reaction :

$H_2SO_4\,(aq) + BaCl_2\,(aq.) \longrightarrow \underset{\text{white ppt}}{BaSO_4\downarrow} + 2HCl$

$BaSO_4 + \text{dil. } HCl \longrightarrow$ No dissolution

Experiment	Observation	Inference
6. Take about 2-5 mL of Na_2CO_3 solution in test tube and add $BaCl_2$ solution drop wise till the formation of precipitate and then add dil. HCl acid.	6. White precipitate appears which dissolves in dil. HCl to give effervescence (coming out colourless CO_2 gas)	6. White precipitate is due to formation of insoluble $BaCO_3$, which gives soluble $BaCl_2$

Reaction :

$$Na_2CO_3\ (aq) + BaCl_2\ (aq) \longrightarrow \underset{\text{white ppt}}{BaCO_3\downarrow} + 2NaCl$$

$$BaCO_3 + 2HCl \longrightarrow BaCl_2 + H_2O + CO_2\uparrow$$

Experiment	Observation	Interference
7. Take about 2-5 mL of potassium chromate in test tube and add $BaCl_2$ solution dropwise	7. Yellow precipitate appears	7. Due to formation of insoluble barium chromate

Reaction :

$$\underset{\text{Pot. Chromate}}{K_2CrO_4\ (aq.)} + BaCl_2\ (aq.) \longrightarrow \underset{\substack{\text{Barium chromate}\\ \text{(yellow ppt.)}}}{BaCrO_4\downarrow} + 2KCl$$

Experiment	Observation	Inference
8. Take about 2-5 mL of potassium dichromate in test tube and add few drops of dil. H_2SO_4 and then add sodium sulphite solution.	8. Yellow colour changes into green coloured solution.	8. Due to formation of soluble green coloured chromium sulphate

Reaction:

$$\underset{\text{Pot. dichromate}}{K_2Cr_2O_7} + 4H_2SO_4 + 3Na_2SO_3 \longrightarrow K_2SO_4 + \underset{\text{Green colour}}{Cr_2(SO_4)_3} + 3Na_2SO_4 + 4H_2O$$

Experiment	Observation	Inference
9. Take 2-5 mL of mercurous nitrate solution in test tube and add few drops of ammonia solution.	9. The solution changes into black colour.	9. Due to formation of a mixture of mercury and mercuric amino nitrate.

Reaction:

$$\underset{\text{Mercurous nitrate}}{Hg_2(NO_3)_2(aq.)} + 2NH_4OH\,(aq.) \rightarrow \underset{\text{black precipitate}}{[Hg + Hg(NH_2)NO_3]} + NH_4NO_3 + 2H_2O$$

(Mixture of mercury and amino-mercuric nitrate)

Experiment	Observation	Interference
10. Take 2-5 mL of mercurous nitrate solution in test tube and add few drops of dilute HCl and then add few drops of ammonia solution.	10. White precipitate changes into black colour on adding ammonia solution.	10. White precipitate is due to formation of Hg_2Cl_2 and black precipitate is due to formation of mixture of mercury and basic amino mercuric chloride.

Reaction:

$$Hg_2(NO_3)_2\,(aq.) + 2HCl \longrightarrow \underset{\text{white precipitate}}{Hg_2Cl_2\downarrow} + 2HNO_3$$

$$Hg_2Cl_2 + 2NH_4OH \longrightarrow \underset{\text{Black precipitate}}{[Hg + Hg(NH_2)Cl]\downarrow} + NH_4Cl + 2H_2O$$

(Mixture of mercury and amino mercuric chloride)

Experiment	Observation	Inference
11. Take about 2-5 mL of $HgCl_2$ solution in test tube add KI solution dropwise till excess and then add few drops of NH_4OH solution.	11. At first scarlet red precipitate appears, which disappears on adding excess of KI solution.	11. Scarlet red precipitate is due to formation of HgI_2. Dissolution of HgI_2 due to formation of soluble K_2HgI_4. Reddish brown precipitate is due to formation of iodide of Million's base.

Reaction:

$$HgCl_2 + 2KI\ (aq.) \longrightarrow \underset{\text{Scarlet red precipitate}}{HgI_2\downarrow} + 2KCl$$

$$HgI_2 + 2KI \longrightarrow K_2HgI_4$$

$$K_2[HgI_4] + 2NH_4OH \longrightarrow 2KI + HgNH_2I + NH_4I + 2H_2O$$

$$2HgNH_2I + H_2O \longrightarrow NH_2HgO.HgI\downarrow + NH_4I$$

Iodide of millions base

Reddish brown precipitate

Conclusion: Thus, different chemical reactions can be studied by observing different types of chemical and physical changes.

Precautions

1. Chemical reagent should be taken in small amount.
2. All apparatus must be clean and tidy.

✦✦

Chapter–two

(A) Experiments Based on Laboratory Techniques

1. To separate the insoluble component in pure and dry state from the given mixture of soluble and insoluble solids (NaCl + sand)
2. To separate volatile component (camphor) from the given mixture of volatile and nonvolatile compounds (camphor + sand).
3. To separate a mixture of two soluble solids by fractional crystallization (KNO_3 + NaCl)
4. To prepare a saturated solution of impure salt and obtain the pure crystals of the same salt by crystallization (impure $CuSO_4$)
5. To separate the components of mixture of two insoluble solids ($CaCO_3$ + sand)
6. To obtain pure water from given sample of impure water (distillation)

Experiment No. 1

Object: **TO SEPARATE THE INSOLUBLE COMPONENT IN PURE AND DRY STATE FROM THE GIVEN MIXTURE OF SOLUBLE AND INSOLUBLE SOLIDS (NaCl + sand)**

Apparatus required:

i. Beaker

ii. Test tube and test tube stand

iii. Sand bath or asbestos sheet
iv. Filter stand
v. Filter paper
vi. Funnel
vii. Tripod stand and wire gauze
viii. Bunsen burner

Chemic required:

i. Mixture of sand and common salt
ii. $AgNO_3$ solution

Theory:

In the given mixture, sodium chloride is soluble in water and sand is insoluble in water. Sodium chloride and sand can be separated by preceding the following process.

Dissolution: The process of dissolving the solute in the solvent is known as dissolution.

Sedimentation: Setting down of insoluble chemical substance (e.g. sand) at the bottom of container is known as sedimentation.

Decantation: The process of transferring the clear supernatant liquid (water) from the top without disturbing the sediment layer is known as decantation.

Sediment: The heavy solid insoluble substance (sand) remaining at the bottom is known as sediment.

Supernatant liquid: The clear liquid above the sediment layer is known as supernatant liquid (water).

Filtration: An insoluble solid component can be separated from the liquid by filtration process or the process of removing the insoluble solid from a liquid by the use of a porous substance like a filter paper, Cotton, etc. is known as filtration.

Residue: The solid chemical substance (sand) remaining at filter paper is called residue.

Filtrate: The liquid substance obtained after filtration is known as filtrate or the substance which has been filtered is called filtrate.

Evaporation: The spontaneous escaping tendency of molecules from its liquid surface due to more kinetic energy than cohesive force is known as evaporation. It is an endothermic process, so on increasing the temperature, the rate of evaporation increases and soluble solid can be obtained from its solution by evaporating all the solvent (water).

Procedure:

1. Take about 4-5 gm the given mixture (say sand and common salt) in beaker and add a test tube-full of water and warm it for some time with constant stirring with glass rod.
2. Fold the circular filter paper into half and then into quarter and make a cone in funnel by taking three folds at one side and the remaining at the other side moistening with a little water as shown in figure 1(a).
3. Assemble the apparatus shown in figure 1(b) and transfer the clear (supernatant) liquid in funnel with a help of glass rod 1/2 inch below the brim of filter paper. Heat (evaporate) the filtrate (common salt and water) in a porcelain basin to get sodium chloride as residue.
4. Wash the sand 3-4 times in the beaker with some warm water till the freshly collected washing is free from chloride (test with $AgNO_3$ solution)
5. Finally take the filter paper out of the funnel and dry the sand over the asbestos sheet or sand bath.

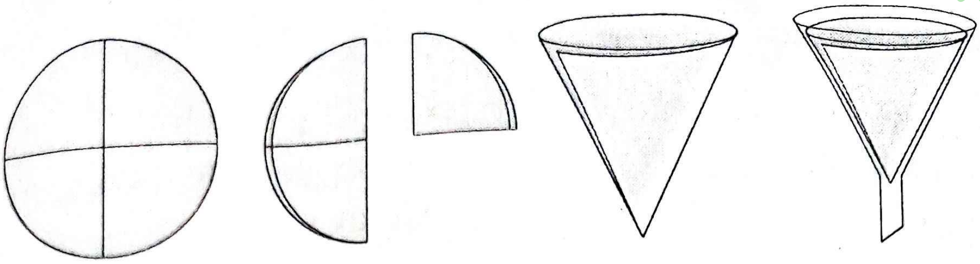


Fig. 1(a) : Folding of filter paper

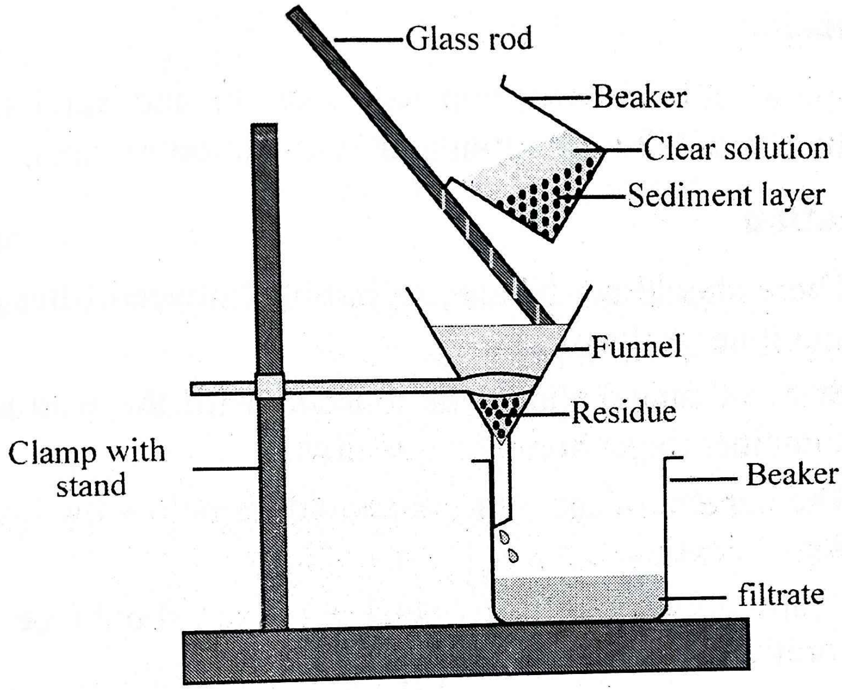


Figure 1(b) Filtration

Observation Table (Test for purity of sand)

Experiment	Observation	Inference
1. Take about 1-2 mL of fresh washing of sand and add few drops of $AgNO_3$ solution.	White precipitate appear	Sand is not free from salt
2. Repeat exp-1 again after further washing the sand	White precipitate does not appear	Sand is free from chloride

Test reaction:

Reaction: $Ag^{+}NO_3^{-}(aq.) + Cl^{-} \longrightarrow AgCl \downarrow + NO_3^{-}$

Silver nitrate — Silver chloride, Curdy white ppt

Result : Sand and crystals common salt are separated in pure and dry state from the mixture.

Conclusion:

Thus, pure and dry common salt (NaC1) and sand (SiO_2) could be obtained by filtration and evaporation process.

Precaution

1. There should not be any air bubble between filter paper and inner walls of funnel.
2. Stem of funnel should be touched with the wall of the container to get homogenous flow.
3. The content of the mixture should be below the brim of the filter cone.
4. Evaporation of filtrate (NaCl + water) should be done carefully.
5. Sand should be dried carefully.

Experiment no. 2

Object: TO SEPARATE VOLATILE COMPONENT (Camphor) FROM THE GIVEN MIXTURE OF VOLATILE & NON-VOLATILE COMPONENTS (Camphor and sand)

Apparatus required:

i. Porcelain basin
ii. Sand bath

iii. Tripod stand and wire gauze

iv. Funnel

Chemical required:

i. Mixture of sand and camphor

Theory:

In the given mixture of sand and camphor, sand is nonvolatile and camphor is volatile. So, camphor can be separated by sublimation method.

Volatile substance : The substance which can easily change into gaseous state is known as volatile substance e.g. camphor, iodine etc.

Non-volatile substance : The substance which cannot easily change into gaseous (vapour) state is known as non-volatile substance. e.g. sand, common salt etc.

Sublimation:

The process of direct conversion of solid into vapour state on heating and vapour state to solid state on cooling without passing through the liquid state is known as sublimation. For example camphor, iodine, NH_4Cl, Naphthalene, benzoic acid, mercuric chloride, etc.

$$\underset{\text{(Camphor)}}{\text{Solid}} \underset{\text{Cool}}{\overset{\text{Heat}}{\rightleftharpoons}} \text{vapour}$$

Sublimate

The vapour gives back the solid at inner side of the funnel which is known as sublimate or, the substance which sublimed off and turned into solid state on cooling is called sublimate.

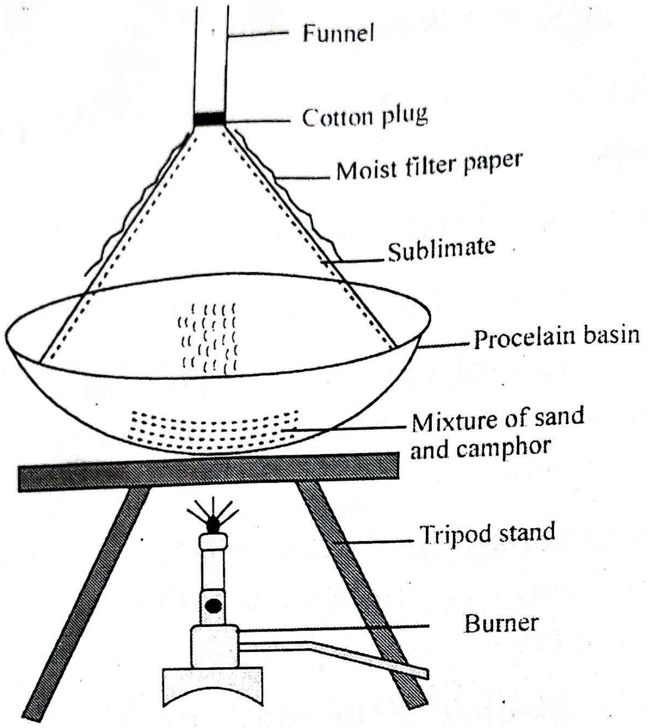


Fig. 2 : Sublimation

Procedure:

i. Take about 3-5 gm mixture of the volatile (camphor) and nonvolatile solid (sand) into a dry and clean porcelain basin.

ii. Cool the outside of the funnel by wrapping it with moist filter paper and plug up the inner open end of the funnel with cotton. Invert the funnel over the mixture containing porcelain basin and heat on sand bath gently as shown in figure 2.

iii. On heating, camphor changes into vapour, which condenses on inner wall of funnel on cooling as sublimate.

iv. After converting all camphor into vapour, put off burner and allow the basin to cool for some time. Now scrape out the camphor from funnel on a piece of filter paper and submit for inspection.

Test for purity of sand:

Experiment	Observation	Inference
1. Small amount of mixture was taken in the spatula and was smelt.	smell of camphor felt.	Presence of camphor in the mixture
2. Experiment no. 1 was repeated after further heating.	Smell of camphor was not observed	Sand is free from camphor

Result :

Camphor and sand are separated in pure form from the given mixture.

Conclusion:

Thus, camphor (volatile) and sand (non-volatile) can be separated from the given mixture by sublimation process.

Precaution:

i. Mixture should be heated in dry and clean porcelain basin gently.

ii. Inner opening of funnel should be closed with cotton or paper plug properly.

iii. Outer surface of funnel should be condensed with moist filter paper.

Experiment No. 3 (A)

Object: TO PREPARE A SATURATED SOLUTION OF IMPURE SALT (Bazaar $CuSO_4$) AND OBTAIN THE PURE CYRYSTALS OF THE SAME SALT (BLUE VITRIOL) BY CRYSTALLIZATION

Apparatus required:

i. Porcelain basin
ii. Beaker
iii. Funnel
iv. Tripod stand
V. Glass rod
vi. Burner
vii. Filter paper

Chemical required: Impure bazaar copper sulphate

Theory:

The pure crystal of copper sulphate (blue vitriol) from impure bazaar copper sulphate can be obtained by crystallization process.

Crystallization: The process of formation of crystals (blue vitriol) by cooling down the saturated solution at higher temperature is known crystallization.

Crystals: Homogeneous solid substance bounded by plane faces meeting in sharp edges having regular and fixed geometric shape with sharp melting point is known as crystal.

Crystallization point: The stage at which the heated solution starts forming crystals on cooling is known as crystallization point.

Mother Liquor: The solution left behind after the formation of crystal is known as mother liquor.

Water of crystallisation: The water molecules associated with crystal forming an integral part of its constitution, which maintain the shape, colour of the crystal is known as its water of crystallization.

For example:

Blue vitriol	:	$CuSO_4.5H_2O$
Green vitriol	:	$FeSO_4.7H_2O$
White vitriol	:	$ZnSO_4.7H_2O$
Epsom salt	:	$MgSO_4.7H_2O$

Unsatured solution : The solution which can dissolve more amount of solute at the given temperature is known as unsaturated solution.

Saturated solution: The solution, in which solvent has no more capacity to dissolve solute particles at certain temperature, is known as saturated solution.

For example: 25 gram of copper sulphate dissolves in 100 mL of water at 30°C to give saturated solution.

Supersaturated solution: It is the maximum saturation of given salt in water at higher temperature. A solution containing more solute dissolved than it is required to give a saturated solution at a given temperature for some time only, then the solution of this condition is called supersaturated solution.

For example: Saturated solution of copper sulphate is heated upto crystallization point to get supersaturated solution, which on cooling gives crystals of blue vitriol.

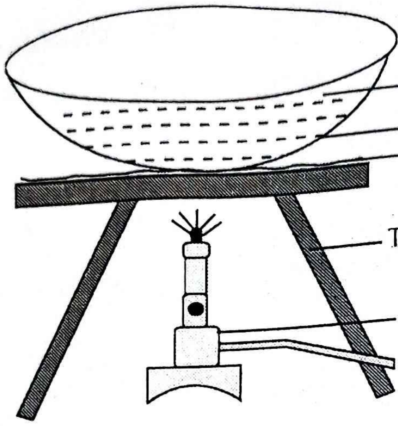


Fig 3(a) : Formation of supersaturated solution of copper sulphate

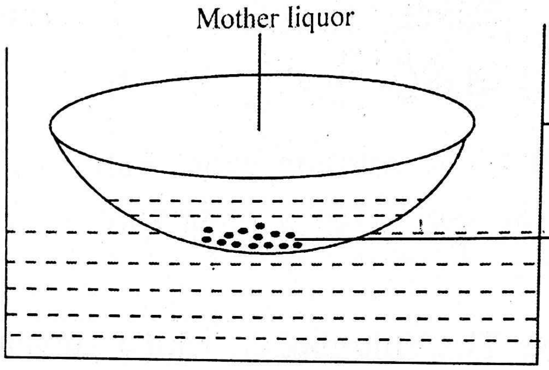


Fig 3(b) : Formation of copper sulphate crystals

Procedure:

i. Take one test tube of water in clean beaker and add impure copper sulphate in beaker to get saturated solution at room temperature and filtered with constant stirring with glass rod.

ii. Now take the saturated solution of copper sulphate in porcelain basin and evaporated upto crystallization point (In order to confirm the crystallization point, remove a drop of the solution with glass rod on dry outer surface of test tube filled with cold water appearance of milli-crystals shows crystallization point has reached).

iii. After reaching this point remove the basin from the flame and allow it cool slowly at room temperature, float basin in water trough containing cold water. After sometime there is formation of crystal.

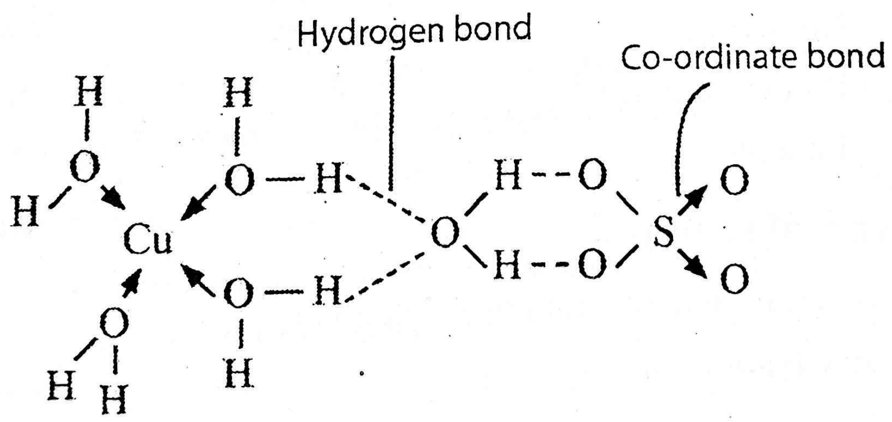
iv. Separate the crystals from liquid by decantation of mother liquor and soaked them between pieces of filter paper.

$$\text{Saturated copper sulphate solution} \xrightarrow[\text{up to C.P.}]{\text{heated}} CuSO_4.5H_2O$$

Observation table (test for crystallization point)

Experiment	Observation	Inference
1. Take drops of heated solution and keep over dry outer surface of test tube filled with cold water.	1. Millicrystals does not form	1. Crystallization point his not reached
2. Repeat experiment no. 1 after further heating of the solution.	2. Millicrystals observe	2. Crystallization point is reached.

Structure of copper sulphate pentahydrate crystal ($CuSO_4$. $5H_2O$):



Precaution:

i. Saturated solution of copper sulphate should be prepared at room temperature

ii. Solution should be evaporated upto crystallization point.

iii. Solution should not be evaporated completely

iv. Blue vitriol should never be dried in open flame.

Result

Pure and dry crystals of copper sulphate is obtained from its impure sample.

Conclusion

Thus, pure crystals of copper sulphate (blue vitriol) is obtained from impure copper sulphate by crystallization process. They are formed in triclinic shape.

Experiment No. 3(B)

Object: TO SEPARATE THE SAND AND COPPER SULPHATE IN PURE AND DRY STATE FROM THE GIVEN MIXTURE.

Apparatus required

i. Beaker

ii. Porcelain basin

iii. Funnel

Chemical required

i. Mixture of sand and copper sulphate

ii. $BaCl_2$ solution

Theory:

In the given mixture, sand is insoluble in water and copper sulphate is soluble in water. So, the mixture is treated with minimum quantity of water to dissolve copper sulphate completely. The mixture is filtered and washed till the sand is free from copper sulphate (Test with $BaCl_2$ solution). The filtrate collected in a basin as blue coloured ($CuSO_4$) is heated carefully up to crystallization point and cooled in water to get crystal of copper sulphate.

Procedure: Similar to experiment no. 3(a).

Purity test for sand

Experiment	Observation	Inference
a. Add few drops of $BaCl_2$ to the fresh filtrate	a. White precipitate appears	a. Sand is not free from SO_4^{--}
b. Filtrate obtain after further washing is taken in test tube and add few cc of $BaCl_2$ solution	b. White precipitate does not appear	b. Sand is free from SO_4^{--}

Reaction:

$$BaCl_2\,(aq.) + SO_4^{--}\,(aq.) \rightarrow \underset{\text{White precipitate}}{BaSO_4\downarrow} + 2Cl^-$$

$$BaSO_4 + \text{dil. } HCl \rightarrow \text{Not dissolved}$$

Result

Sand and crystals of copper sulphate are separated in pure and dry form from the given mixture.

Conclusion:

Thus, copper sulphate crystals and sand in pure and dry state are obtained from the mixture by filtration and crystallization processes.

Experiment No. 4(A)

Object: TO SEPARATE THE MIXTURE OF POTASSIUM CHLORIDE (KCl) AND POTASSIUM CHLORATE ($KClO_3$) BY FRACTIONAL CRYSTALLISATION

Apparatus required:

i. Test tube
ii. Porcelain basin
iii. Test tube

Chemicals required:

i. A mixture of $KClO_3$ and KC1 with ratio of 5:3.
ii. Distilled water.

Theory:

Two solutes having different solubility and comparable composition present in a solution can be separated by repeated fractional crystallisation (having different crystallisation point). The separation would be more effective, when the percent of more soluble component is less than the percent of less soluble component in the mixture and the nature of solubility curves provide useful information towards the feasibility of separation.

Standard data of solubilities of KCl and $KClO_3$ in water at different temperatures

°C	0	10	20	30	40	50	60	70	80
KCl	27.6	31.0	34.0	37.0	40	42	45	48.1	57
$KClO_3$	3.3	5.0	7.4	10.5	14.0	19.3	26.0	31.5	40

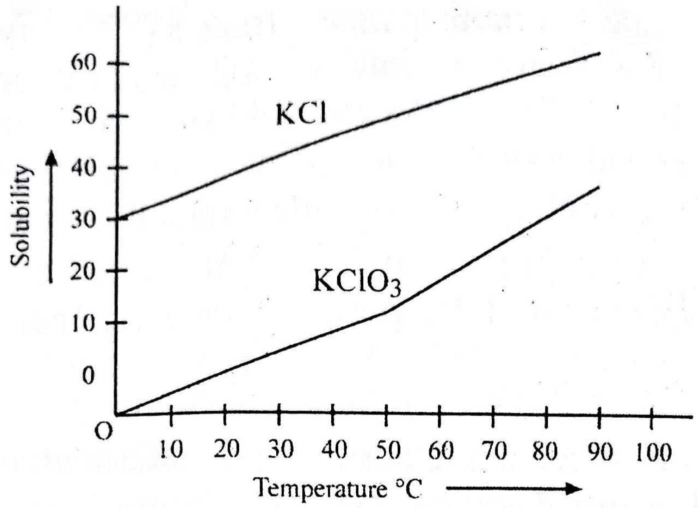


Figure 4 : Solubility curve

In the given composition of the mixture say $KClO_3$: KCl (5:3), when $KClO_3$ gets saturated around 70°C, KCl remains unsaturated and it gets its saturation, when it is cooled below 10°C in the given volume of the solution. Hence majority of $KClO_3$ crystallises out, when the solution saturated at 70°C is cooled to room temperature. After the separation of $KClO_3$ crystals, the mother liquor consists of majority of KCl with minor amount of $KClO_3$. When it is heated to crystallization point major amount of KCl crystallises with minor amount of $KClO_3$. Hence repeated operation enriches the purity of the component.

Procedure:

i. Take 8 grams of the given mixture (KCl + $KClO_3$) in a clean beaker and add about 60 mL of distilled water to dissolve mixture completely.

ii. Heat the solution gently in low flame either in the beaker or porcelain basin, so as to get the crystallization point of $KClO_3$ at about 70°C and then cool the mixture upto room temperature

Here major amount of $KClO_3$ crystallize out, it also contains small amount of KCl crystal.

iii. Separate the mother liquor from $KClO_3$ crystals, which contains major amount of KCl and minor amount of $KClO_3$ in dissolved state. Add small amount of distilled water and crystallization point of KCl, we get crystals of KCl on cooling. It also contains small amount of $KClO_3$.

iv. Repeat the process of fractional crystallization for each sample to enrich the purity of the component.

Result :

Crystals of potassium chloride and potassium chlorate are separated in pure dry state from the mixture.

Conclusion:

Thus, pure KCl and $KClO_3$ crystals are separated from the given mixture by fractional crystallization.

Precautions:

i. Crystallization point of KCl and $KClO_3$ crystals should be noted carefully.

ii. Solution should be heated gently.

Experiment No. 4(B)

Object: TO SEPARATE THE MIXTURE OF POTASSUM NITRATE (KNO_3) AND SODIUM CHLORIDE (NaCl) BY THE FACTIONAL CRYSTALLIZATION

Apparatus required:

i. Porcelain basin
ii. Beaker
iii. Test tube
iv. Stirrer
vi. Burner

Chemicals Required:

i. Mixture of KNO_3 and NaCl

Theory:

Here theory and process of separation are similar to that of experiment number 4(a).

Note: Here separation of KNO_3 and NaCl is somewhat difficult by fractional crystallization, because this mixture has chances of giving four different crystals. They are KNO_3, NaCl, KCl and $NaNO_3$ and the solubility curve of these salts give the idea of crystallization of one in preference to the other.

Experiment No. 5

Object: TO SEPARATE THE COMPONENTS OF A MIXTURE OF TWO INSOLUBLE SOLIDS ($CaCO_3$ and sand).

Apparatus required:

i. Beaker
ii. Funnel
iii. Wash bottle
iv. Filter paper
v. Burner, etc.

Chemicals required:

i. Mixture of $CaCO_3$+ sand
ii. Dil. HCl
iii. $BaCl_2$ solution
iv. $AgNO_3$ solution
v. Na_2CO_3 solution or $(NH_4)_2CO_3$ solution

Theory:

In the given mixture of sand and calcium carbonate (limestone) both are insoluble in water. When the mixture of sand and calcium carbonate is reacted with dilute hydrochloric acid, we get calcium chloride, water and carbon dioxide, while sand is unaffected.

$(Sand + CaCO_3) + dil.2HCl \rightarrow Sand + CaCl_2 + H_2O + CO_2$

On filtration, we get calcium chloride, as filtrate and sand as residue. Calcium chloride is treated with sodium or ammonium carbonate upto complete white precipitation of calcium, carbonate, which is filtered, washed with water (3-4 times) to make it free from CO_3^{--}, Cl^- and heated to get pure and dry calciumcarbonate.

$CaCl_2 + Na_2CO_3 \rightarrow CaCO_3 \downarrow + 2NaCl$

$CaCl_2 + (NH_4)_2CO_3 \rightarrow CaCO_3 \downarrow + 2NH_4Cl$

White ppt

The residue (sand) is washed with water till it is free from Cl^- ions.

Reaction for Purity Test:

$Cl^- + AgNO_3 \rightarrow AgCl\downarrow + NO_3^-$

White ppt.

$CO_3^{--} + BaCl_2 \rightarrow BaCO_3 \downarrow + 2Cl^-$

$BaCO_3 + 2HCl \rightarrow BaCl_2 + H_2O + CO_2$

Precipitation or Metathesis Reaction

It is a double decomposition reaction, in which two soluble or clear solutions are mixed together to get one of product in the insoluble state by the mutual exchange of constituent groups of two reacting substances.

Precipitate: The insoluble solid, which separates out is known as precipitate.

Precipitant: The reagent, which is added to obtain precipitate is known as precipitant.

Procedure:

i. Take about 2-5 gram of the mixture of sand and calcium carbonate in a beaker and add dilute hydrochloric acid (little) drop by drop till the effervescence (coming out of CO_2 gas) ceases.

ii. Warm the solution and filtered, we get sand as residue and $CaCl_2$ as filtrate. Wash the sand with warm water till the last drop of filtrate is free from Cl^- ions. (Test with $AgNO_3$ solution)

iii. Warm the filtrate ($CaCl_2$) and add Na_2CO_3 or $(NH_4)_2CO_3$ till complete precipitation takes place. Complete precipitation can be tested as;

    Take about 1 mL of supernatant clear solution in test tube and add 1- 2 drop of Na_2CO_3 solution. If it gives white precipitate there is no complete precipitation and add more Na_2CO_3. If it does not give white precipitate i.e. precipitation is completed.

iv. After complete precipitation, filter the content and residue ($CaCO_3$) is washed with warm water until the filtrate (last drop) does not give white precipitate with barium chloride solution.

v. At last dry the $CaCO_3$ precipitate along with filter paper over the sand bath or asbestos sheet or in air oven.

1. **Observation table for test of purity of sand:**

Experiment	Observation	Inference
1. Add few drops of $AgNO_3$ solution into the test tube containing few drops of fresh filtrate obtained after washing sand.	Formation of white precipitate	Presence of Cl^-
2. Repeat expt. No. 1. after further washing of sand.	No white precipitate	Sand is free from Cl^-.

2. **Observation Table for test of completeness in precipitation**

Experiment	Observation	Inference
1. Take small amount of supernatant liquid in the test tube and add few drops of Na_2CO_3 solution	1. Precipitate is observed	1. Precipitation is not complete
2. Repeat expt. no. 1 after adding more Na_2CO_3 solution	2. Precipitate is not seen	2. Precipitation is complete.

3. **Observation table for test of purity of $CaCO_3$**

Experiment	Observation	Inference
1. Take filtrate obtained after washing calcium carbonate in two test tubes 'A' and 'B'. a. In test tube 'A', add few drops of $AgNO_3$ solution. b. In test tube 'B' add few drops of $BaCl_2$ solution	a. Formation of white precipitate b. Formation of white precipitate	a. Presence of Cl^- b. Presence of soluble CO_3^{--}
2. Repeat exp. No. 1. after further washing $CaCO_3$ few more times.	No white precipitate	Calcium carbonate is free from soluble Cl^- and soluble CO_3^{--}

Test Reaction :

Reaction: $Ag^+NO_3^-$ (aq.) + $Cl^- \longrightarrow AgCl\downarrow + NO_3^-$

Silver nitrate　　　　　Silver chloride

Curdy white ppt

$CO_3^{--} + BaCl_2 \longrightarrow BaCO_3\downarrow + 2Cl^-$

white ppt

Result :

Sand and calcium carbonate are separated in pure and dry state from the mixture.

Conclusion:

Thus, pure and dry calcium carbonate and sand can be obtained by precipitation, filtration and on heating.

Precaution:

i. Little dilute HCl should be used otherwise, we need more Na_2CO_3 or $(NH_4)_2CO_3$ solution.

ii. $CaCO_3$ and sand should be dried carefully.

Experiment No. 6

Object: TO OBTAIN PURE WATER FROM GIVEN SAMPLE OF IMPURE WATER

Apparatus required:

i. Glass retort
ii. Conical flask
iii. Water trough
iv. Wire gauze
v. Tripod stand
vi. Burner

Chemical required:

i. Impure water containing soluble compound like $CuSO_4$, NaCl, etc.
ii. $AgNO_3$ solution
iii. $BaCl_2$ solution

Theory:

The pure water from impure water containing dissolved solids (say NaCl, $CuSO_4$ etc.) can be obtained by distillation process.

Distillation:

The process of converting liquid into vapour by heating and condensation into liquid without any change in its chemical composition is known as distillation.

$$\text{Impure water} \xrightarrow{\Delta} \text{vapour} \xrightarrow{\text{cool}} \text{pure water}$$

Distillate:

The liquid collected in a receiver (conical flask) after the condensation of vapour is known as distillate.

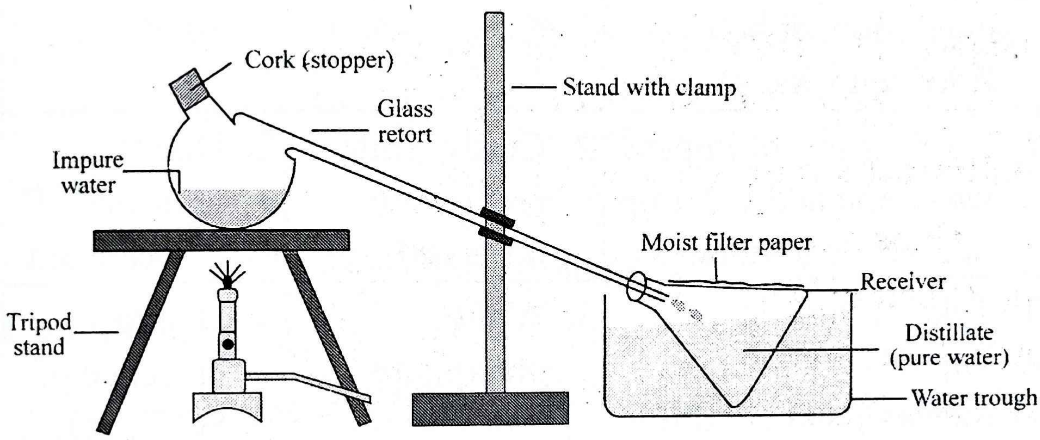


Fig 5 : Distillation using glass retort

Procedure:

i. Take one test tube impure water (given) in glass retort and heat it over the wire gauze by means of the Bunsen burner till it boils as shown in the figure 5.

ii. The solvent vapourised and is gradually condensed back into liquid in receiver wrapped with moist filter paper

Note: The first portion of the distillate is used to wash the receiver and reject into the sink

iii. Thus, after washing with distillate about one test tube distillate (pure water) is collected and test for dissolved (impure water) and pure water (distillate) is performed separately.

Test for impure water

Experiment	Observation	Inference
1. Evaporate a little impure water (1–2 mL) on a clock glass kept over a beaker half filled with water	1. Solid residue observed on the clock glass	1. Due to presence of dissolved solids.
2. Take 1-2 mL of impure water and add 1-2 drop of $AgNO_3$ solution.	2. Curdy white precipitate observed	2. Due to presence of chloride ion.
3. Take 1-2 mL of impure water in test tube and add 1-2 drops of $BaCl_2$ solution.	3. White precipitate	3. Due to presence of SO_4^{--}, SO_3^{--}, CO_3^{--}. etc.
4. Test with litmus paper (blue and red)	4. Moist blue litmus paper turns into red	4. It is acidic in nature.

Test reactions

$$AgNO_3 + Cl^- \longrightarrow AgCl\downarrow + NO_3^-$$

$$BaCl_2 + SO_4^{--} \longrightarrow BaSO_4\downarrow + 2Cl^-$$

$$BaCl_2 + SO_3^{--} \longrightarrow BaSO_3\downarrow + 2Cl^-$$

$$BaCl_2 + CO_3^{--} \longrightarrow \underset{\text{white ppt.}}{BaCO_3\downarrow} + 2Cl^-$$

Test for the purity of pure water (Distillate)

Experiment	Observation	Inference
1. Evaporate a little pure (1-2 mL) water on a clock glass as before.	1. No solid residue	1. Due to absence of dissolved solids.
2. Test pure water with blue and red litmus paper.	2. No any change	2. The pure water is neutral.
3. Take 1-2 mL of pure water and add 1-2 drop of $AgNO_3$ solution.	3. No white precipitate	3. Due to absence of Cl^- ion
4. Take 1-2 mL of pure water in test tube and add 1-2 drop of $BaCl_2$ solution.	4. No white precipitate.	4. Due to absence of CO_3^{--}, SO_4^{--} and SO_3^{--}

Result :

Pure water is obtained from impure sample of water.

Conclusion:

Thus, pure water (distillate) can be obtained from impure water by distillation process.

Precaution:

i. Glass retort should be heated carefully.

ii. Receiver and glass retort should be cooled with moist filter paper.

iii. First distillate should be rejected for washing receiver.

Questions

Q.1 What is fractional distillation? Give suitable examples.

Ans: When a mixture containing two or more liquids having different boiling point is evaporated by heating, the vapour phase get enriched in concentration of more volatile components (ethyl alcohol at 78.6°C) and the mixture left in the container get enriched in the concentration of less volatile liquid (eg water 100°C).

For example: Mixture of ethyl alcohol (b.p. 78°C) and water (b.p. 100°C) is separated by fractional distillation.

Q.2 What is molecular formula of silver nitrate solution? Give its one use with suitable reaction.

Ans: The molecular formula of silver nitrate is $AgNO_3$. It is used to detect halides $X^- \longrightarrow (Cl^-, Br^-, I^-)$ present in aqueous solution.

$$Cl^- + AgNO_3\,(aq.) \longrightarrow \underset{\text{white precipitate}}{AgCl\downarrow} + NO_3^-$$

$$Br^- + AgNO_3\,(aq.) \longrightarrow \underset{\text{Pale yellow precipitate}}{AgBr\downarrow} + NO_3^-$$

$$I^- + AgNO_3\,(aq.) \longrightarrow \underset{\text{Deep yellow precipitate}}{AgI\downarrow} + NO_3^-$$

Q.3 What is molecular formula of barium chloride? Give its use.

Ans: The molecular formula of barium chloride is $BaCl_2$. It is used to detect CO_3^{--} (carbonate), SO_4^{--} (sulphate) and SO_3^{--} (suiphite) present in given solution.

$$BaCl_2 + SO_4^{--} \longrightarrow \underset{\text{white ppt}}{BaSO_4\downarrow} + 2Cl^-$$

$$BaCl_2\,(aq.) + SO_3^{--} \longrightarrow \underset{\substack{\text{white ppt}\\\text{(barium sulphite)}}}{BaSO_3\downarrow} + 2Cl^-$$

$$BaCl_2\,(aq.) + CO_3^{--} \longrightarrow \underset{\text{white ppt}}{BaCO_3\downarrow} + 2Cl^-$$

Chapter–three

B. EXPERIMENT TO STUDY THE DIFFERENT REACTIONS: (Neutralization, precipitation, redox reaction, electrolysis)

Experiment No. 7

Object: TO PERFORM PRECIPITATION REACTION OF BARIUM CHLORIDE AND SULPHURIC ACID AND TO OBTAIN PURE AND DRY SOLID BARIUM SULPHATE

Apparatus required:

i. Beaker
ii. Test tube
iii. Filter paper
iv. Wash bottle
v. Funnel
vi. Asbestos sheet or sand bath

Chemicals required:

i. $BaCl_2$ solution
ii. Dil.H_2SO_4 solution
iii. $AgNO_3$ solution

Theory:

The solid barium sulphate can be obtained by the precipitation reaction of barium chloride with dilute sulphuric acid.

$BaCl_2$ (aq.) + dil. H_2SO_4 (aq.) → $BaSO_4$↓ + HCl (aq.)
white precipitate
(Barium sulphate)

Precipitation reaction or Metathesis

It is a double decomposition reaction between two clear solutions to give an insoluble chemical substance.

Precipitate

The insoluble solid which separates out is known as precipitate.

Procedure

i. Take about half test tube of barium chloride solution in beaker, warm it and add dilute sulphuric acid dropwise up to complete precipitation of barium sulphate.

ii. Test for the completeness of the precipitation by pouring carefully a little precipitant (dil. H_2SO_4) along the side of the beaker down to the upper clear solution without disturbing sediment layer ($BaSO_4$). If there is no precipitate formation, i.e. precipitation is complete, filter off the clear liquid. Wash the precipitate with 3-4 times with warm water by using the method 'washing by decantation'.

iii. Transfer the barium sulphate precipitate into the filter paper with the help of wash bottle and wash it with warm water till the washing is free from impurities like sulphate (SO_4^{--}) and chloride (Cl^-).

iv. Now dry the filter paper containing $BaSO_4$ precipitate on asbestos sheet or sand bath or inside the air oven.

Test for purity of $BaSO_4$

Experiment	Observation	Inference
1 Fresh washing (2-3 mL) is taken in test tube and add 1-2 drops of $AgNO_3$ solution.	1. White precipitate	1. Due to presence of Cl^- ions.
2. Fresh washing (2-3 mL) is taken in test tube and add 1-2 drops of $BaCl_2$ solution.	2. White precipitate	2. Due to presence of SO_4^{--} ions
3. Repeat this test with $AgNO_3$ and $BaCl_2$ solution separately	3. No precipitate	3. Due to absence of Cl^-, SO_4^{--} ions

Reaction:

$$Cl^- + AgNO_3\,(aq.) \rightarrow \underset{\text{white precipitate}}{AgCl\downarrow} + NO_3^-$$

$$SO_4^{--} + BaCl_2(aq) \rightarrow \underset{\text{white precipitate}}{BaSO_4\downarrow} + 2Cl^-$$

Result :

Pure and dry crystals of barium sulphate is prepared.

Conclusion:

Thus, white precipitate of barium sulphate can be obtained by precipitation.

Precaution

i. Solution of $BaCl_2$ should be warmed.
ii. There should be complete precipitation reaction.
iii. Barium sulphate precipitate should be dried carefully.

Questions

1. **Define precipitation and precipitate with suitable example**
2. **Give the another example of precipitation reaction.** (Hints $Ag^{+}NO_3^{-}(aq.) + Na^{+}Cl^{-}(aq.) \rightarrow \underset{\text{White precipitate}}{AgCl\downarrow} + NaNO_3$

Experiment No. 8 (A)

Object: TO NEUTRALIZE SODIUM HYDROXIDE WITH HYDROCHLORIC ACID SOLUTION AND RECOVER THE CRYSTAL OF SODIUM CHLORIDE

Apparatus required:

i. Porcelain basin
iii. Funnel Chemical required:
ii. Test tube
iv. Burner

Chemical required

i. Bench (dil.) HCl solution
ii. Bench (dil.) NaOH solution
iii. Phenolphthalein indicator

Theory:

Sodium chloride crystal can be obtained by the neutralization of dilute hydrochloric acid with dilute sodium hydroxide (base) solution.

Neutralization is the combination of hydrogen ion (H^+) from acid and hydroxyl ion (OH^-) from base to give neutral water.

Reaction

$$NaOH\ (aq.) + HCl\ (aq.) \rightarrow NaCl\ (aq.) + H_2O\ \ (neutral)$$

Net Ionic reaction

$$H^+\ (aq.) + OH^-\ (aq.) \rightarrow \underset{\text{Neutral}}{H_2O}$$

When the resulting solution of sodium chloride is evaporated upto crystallisation point, we get crystal of sodium chloride

Procedure

i. Take about half test tube of dilute sodium hydroxide solution in a porcelain basin and add one or two drop of phenolphthalein as indicator, the colour of the solution becomes pink.

ii. Add dil. HCl dropwise till the last drop of HCl just changes the pink colour of solution to colourless. This is tested with blue and red litmus paper separately at neutral point there is no effect of blue and red litmus paper.

Note: (a) If blue litmus paper turns into red i.e. solution is acidic add dil. NaOH solution dropwise upto neutral point.

(b) If red litmus paper turns into blue i.e. solution is basic and add dil. HC1

iii. After preparation of neutral solution, filter the solution and evaporate the solution upto crystallisation point, cool the solution, we get crystals of sodium chloride.

Result :

Pure and dry crystals of sodium chloride is prepared.

Conclusion:

Thus, pure and dry crystals of sodium chloride can be obtained from the given solution of dilute HCl and dilute sodium hydroxide by neutralization reaction.

Precaution:

i. Neutral solution should be prepared carefully.
ii. Resultant solution should be checked with blue and red litmus paper. If the solution is neutral, the colour of litmus paper remains unchanged.
iii. Solution should be evaporated carefully.

Experiment No. 8 (B)

Object: TO NEUTRALIZE SULPHURIC ACID WITH SODIUM HYDROXIDE SOLUTION AND TO RECOVER CRYSTALS OF GLAUBER'S SALT ($Na_2SO_4.10H_2O$).

Apparatus required:

i. Porcelain basin
ii. Test tube
iii. Burner
iv. Water trough

Chemical required:

i. Bench (about 2N) H_2SO_4 solution
ii. Bench (about 2N) NaOH solution
iii. Phenolphthalein indicator

Theory:

The crystal of Glauber's salt from the given solution of bench sodium hydroxide and sulphuric acid can be obtained by neutralisation reaction.

$$2NaOH\ (aq.) + H_2SO_4\ (aq.) \rightarrow Na_2SO_4 + H_2O$$

The net ionic equation of neutralization reaction is

$$H^+\ (aq.) + OH^-\ (aq.) \rightarrow H_2O\ (\ell)$$

After getting neutral solution of sodium sulphate, it is heated upto crystallisation point and on cooling, we get crystals of decahydrated of Glauber's salt.

$$Na_2SO_4\ (aq.) \xrightarrow[\text{Crystallisation point}]{\text{Heated upto}} Na_2SO_4.10H_2O$$

Procedure:

i. Take about 5 mL of bench sodium hydroxide in clean porcelain basin and add one or two drop of phenolphthalein indicator, it becomes pink colour.

ii. Now add bench sulphuric acid dropwise with glass tube or dropper in porcelain basin to make to colourless (neutral point). This colourless solution is tested with blue and red litmus paper, if there is no effect on both litmus paper, we get neutral solution, which is filtered.

iii. The resultant neutral solution of sodium sulphate is evaporated upto the crystallisation point (The crystallisation point is detected by adding a drop of the heated solution on the outer dry wall of the test tube containing cold water.) On reaching crystallisation point, the porcelain basin is floated in cold water containing in water trough.

iv. Remove the mother liquor and crystal of Glauber's salt is dried in filter paper.

Result :

Pure and dry crystals of Glauber's salt are prepared.

Conclusion:

Thus, crystals of Glauber's salt can be obtained by neutralisation of bench (dilute) sulphuric acid and bench sodium hydroxide solution.

Questions

1. What is hydrated salt? Give suitable example.'

Ans: Salt containing water molecule is known as hydrated salt. For example: Hydrated Glauber's salt contains 10 molecules of water crystallisation

2. What is anhydrous salt?

Ans: Salt containing no water molecule is known as anhydrous salt. For example: When Glauber's salt is heated more than 32.4°C, we get anhydrous salt.

Experiment no. 9

Object: TO TEST THE FERROUS IONS PRESENT IN THE GIVEN AQUEOUS SOLUTION AND OXIDISE IT TO FERRIC IONS BY REDOX REACTION ($Fe^{++} \rightarrow Fe^{+++}$ ions).

a. **TO TEST FOR FERROUS ION (Fe^{++}) IN THE GIVEN FERROUS SOLUTION**

b. **TO OXIDISE THE FERROUS INTO FERRIC (Fe^{3+}) IONS AND TO TEST FOR THE FERRIC ION IN THE OXIDIZED SOLUTION**

Apparatus required:

i. Beaker

ii. Test tube stand

iii. Test tube

iv. Burner

Theory:

When ferrous sulphate solution is heated with dilute sulphuric acid in presence of conc. nitric acid, ferric sulphate is obtained due to oxidation of ferrous into ferric.

Reduction

$$2\overset{+2}{Fe}SO_4 + H_2SO_4 + 2H\overset{+5}{N}O_3 \xrightarrow{\Delta} \overset{+3}{Fe_2}(SO_4)_3 + 2\overset{+4}{N}O_2 + 2H_2O$$

Ferrous sulphate   Ferric sulphate

Oxidation

Ionically:

$$2Fe^{++} + 4H^+ + 2NO_3^- \longrightarrow 2Fe^{+++} + 2NO_2 + 2H_2O$$

Oxidising agent (Oxidant): Those chemical substances, which bring about oxidation are known as oxidants. For example: Cl_2, Br_2, HNO_3, Conc. H_2SO_4, O_3, H_2O_2, etc

Reducing agent (Reductant): Those chemical substances which bring about reduction are known as reductants. For example HI, HBr, HNO_2, $SnCl_2$, CO, NH_3, H_2S, heated coke (carbon), etc.

Redox Reaction : Such type of reaction, in which oxidation and reduction takes place simultaneously is known as redox reaction.

Red = reduction, Ox = Oxidation

In the above reaction nitric acid oxidises ferrous sulphate into ferric sulphate, so it is oxidising agent but itself, gets reduced to nitrogen dioxide gas. Likewise ferrous sulphate reduces

Nitric acid into nitrogen dioxide, so it is reductant and itself gets oxidised to ferric sulphate i.e. oxidation and reduction takes place side by side or simultaneously.

a. Test for ferrous ion (Fe^{++})

Experiment	Observation	Inference
1. Take 1–2 mL original solution (O.S.) of $FeSO_4$ in a test tube and add few drops of potassium ferrocyanide solution	1. No ppt or blueish white ppt	1. Due to presence of ferrous ion (Fe^{++}).
2. Take 1–2 mL of O.S. in test tube and add potassium ferricyanide solution.	2. Deep blue precipitate (Turn bull's blue)	2. Due to presence of Fe^{++} ion
3. Take 1–2 mL of O.S. in test tube and add dil. H_2SO_4 and few drops of $KMnO_4$	3. Pink colour of $KMnO_4$ is discharged	3. Due to presence of Fe^{++} ion
4. Take original solution in test tube and add few drops of ammonium thiocyanate solution.	4. No precipitate or colouration	4. Due to presence of Fe^{++} ion
5. Take original solution in test tube and add few drops of ammonia solution	5. Green precipitate appears	5. Due to formation of ferrous hydroxide $[Fe(OH)_2]$

Reaction:

i. $\underset{\text{Pot. Ferrocyanide}}{FeSO_4 + K_4[Fe(CN)_6]} \longrightarrow K_2Fe[Fe(CN)_6] + K_2SO_4$

ii. $FeSO_4 + K_3[Fe(CN)_6] \longrightarrow KFe[Fe(CN)_6] + K_2SO_4$

Potassium ferro ferricyanide

Deep blue (Turn bull's) precipitate

Or,

$3Fe^{++} + 2K_3[Fe(CN)_6] \longrightarrow Fe_3[Fe(CN)_6]_2 + 6K^+$

Ferrous ferricyanide

iii. $2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$

$2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O] \times 5$

$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$

iv. $FeSO_4 + NH_4CNS \longrightarrow$ No reaction

Amm. thiocyanate

v. $FeSO_4 + 2NH_4OH \longrightarrow Fe(OH)_2\downarrow + (NH_4)_2SO_4$

green ppt

Formation of Ferric ion (Fe^{+++}) from ferrous (Fe^{++}) ions:

Take about 5-10 mL of original solution of ferrous sulphate in beaker. Add about an equal volume of bench sulphuric acid and few mL of Conc. Nitric acid (5-10 drops) and boil the solution upto yellow colour, cool and dilute to about 20 mL (one test tube) with water and perform the following tests for ferric ion (Fe^{+++}) in the oxidised solution.

b. Test for Ferric ion (Fe^{+++})

Experiment	Observation	Inference
1. Take 2–3 mL of oxidised solution (Fe^{+++}) in test tube and add few drops of potassium ferrocyanide solution	1. Deep blue or prussian blue precipitate appears.	1. Due to formation of ferric ferrocyanide

2. Take 1–2 mL of oxidised solution in test tube and add few drops of potassium ferricyanide solution	2. No precipitation or green colour	2. Due to presence of Fe^{+++} ions
3. Take 1–2 mL of oxidised solution (Fe^{+++}) and add dil. H_2SO_4 and a few drops of $KMnO_4$.	3. Pink colour of $KMnO_4$ is not discharged	3. Due to presence of Fe^{+++} ions.
4. Take 1-2 mL oxidised solution in test tube and add few drops of ammonia solution (NH_4OH)	4. Reddish brown precipitate	4. Due to formation of $Fe(OH)_3$.
5. Take 1–2 mL of oxidized solution in test tube and add potassium thiocyanate solution	5. Deep red coloured solution	5. Due to formation of Ferric thiocyanate

Reactions

i. $Fe_2(SO_4)_3 + \underset{\text{Pot. Ferrocyanide}}{2K_4[Fe(CN)_6]} \longrightarrow 2KFe[Fe(CN)_6]\downarrow + 3K_2SO_4$

Or,

$2Fe_2(SO_4)_3 + 3K_4[Fe(CN_6] \longrightarrow Fe_4[Fe(CN)_6]_3 + 6K_2SO_4$

Deep blue (Prussian blue)

Ferric ferrocyanide

ii. $Fe_2(SO_4)_3 + K_3[FeCN)_6] \longrightarrow$ No reaction

iii. $Fe_2(SO_4)_3 + \text{dil. } H_2SO_4 + KMnO_4 \longrightarrow$ No reaction

iv. $Fe_2(SO_4)_3 + 6NH_4OH \longrightarrow 2Fe(OH)_3\downarrow + (NH_4)_2SO_4$

Reddish brown ppt

v. $Fe_2(SO_4)_3 + 6NH_4CNS \longrightarrow Fe(CNS)_3 + (NH_4)_2SO_4$

Ammonium Thiocyanate — Blood red colour (ferric thiocyanate)

Results :

Presence of ferrous ion is confirmed in the given ferrous solution and ferric solution is prepared from the ferrous solution.

Conclusion

Thus, we can get ferric ion (Fe^{+++}) from ferrous ion by oxidation reaction and able to get clear concept of ferric and ferrous ions by studying with different chemical reagents.

Experiment No. 10

Object: TO STUDY THE PROCESS OF ELECTROLYSIS AND ELECTROPLATING

a. ELECTROLYSIS OF WATER

Apparatus required:

i. Voltameter
ii. Electrodes (platinum or graphite rod)
iii. Test tube
iv. Battery

Chemical required:

i. Water
ii. dil. H_2SO_4

Theory:

When electricity is passed through acidified solution of water having graphite or platinum as anode and cathode, the electrolysis takes place as follows:

$$2H_2O \rightleftharpoons 2H^+ + 2OH^-$$

The net ionic equation of neutralisation is

$$H^+ (aq.) + OH^- (aq.) \longrightarrow H_2O\ (l)$$

At cathode:

Hydrogen ion (H^+) moves towards cathode, where it gains electron by reduction method to give neutral hydrogen atom, which combines with another atom to give hydrogen gas, which is collected in cathode by downward displacement of water.

$$2H^+ + 2e^- \xrightarrow{\text{reduction}} 2H^0 \text{ (Neutral) or } H_2\uparrow$$

At anode:

Hydroxyl ion (OH^-) moves towards anode, it loses electron by oxidation process like

$$2OH^- \xrightarrow[-2e]{\text{Reduction}} 2OH^\circ$$

$$OH + OH \longrightarrow H_2O + O$$

Now the oxygen atom thus formed combines with another oxygen atom to give an oxygen molecule, which is collected in the test tube by downward displacement of water.

$$O + O \longrightarrow O_2\uparrow$$

$$2H_2O \longrightarrow 2H_2 + O_2$$

2 volume (1 volume)

Electrolytes: Those chemical substances (acid, base, salts, etc.), which conduct electricity in fused or aqueous condition are known as electrolytes. For example: $NaCl$, $CuSO_4$, $AgNO_3$, $HC1$, KOH, $NaOH$, etc.

Non–electrolytes: Those chemical substances, which do not conduct electricity even in fused or aqueous state are known as nonelectrolytes For example: sugar, petrol, etc.

Voltameter: The vessel, in which electrolysis is carried out is known as the voltameter.

Electrodes: The metal plates or wires that are dipped into the solution to make an electronic connection are known as electrodes Electricity enters or leaves the electrolyte through the electrodes. There are two types of electrodes.

Anode: The electrode connected to the positive terminal of a battery is known as anode.

Cathode: The electrode connected to the negative terminal of the battery is known as cathode.

Ionisation: The process of breaking up of an electrolytes into ions or radicals in solution is known as ionization. For example: When NaCl is dissolved into water, it dissociates as

$$NaCl \rightleftharpoons Na^+ + Cl^-$$

Fused or aqueous

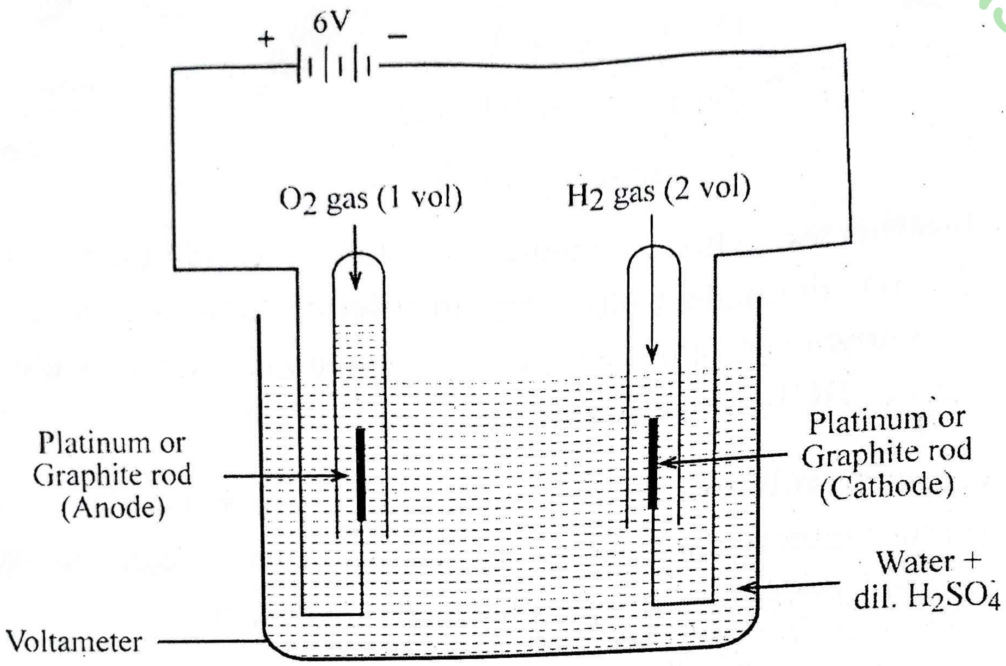


Fig 6 : Electrolysis of acidified water

Procedure: Take a beaker and test tube arranged as shown in figure 6.

- Put two graphite rods or platinum plates into the beaker such that the rods do not touch each other.
- Fill two-third of the beaker with water and acidified with dilute (5%) sulphuric acid.
- Fill the test tubes completely with water and invert over each plate (anode and cathode)
- Join the rods to battery (6V), so that current passes into the solution

As the current passes into the solution, evolution of gas starts to be collected inside by the downward displacement of water. The gas collected at the cathode tube is double than that collected at the anode tube. This gas is hydrogen because it gives pop sound on burning candle and the gas collected at anode is oxygen because it supports burning candle or matches sticks.

Results:

Process of electrolysis is studied.

Conclusion:

Thus, the experiment shows water is composed volumetrically 2 volumes of hydrogen and 1 volume of oxygen gas.

(B) ELECTROPLATING OF IRON NAIL OR SPOON WITH COPPER METAL

Apparatus required:

i. Voltameter
ii. Electric wire
iii. Electric resource or battery

Chemical required:

i. Copper sulphate solution
ii. Copper plate
iii. Iron nail or spoon

Theory

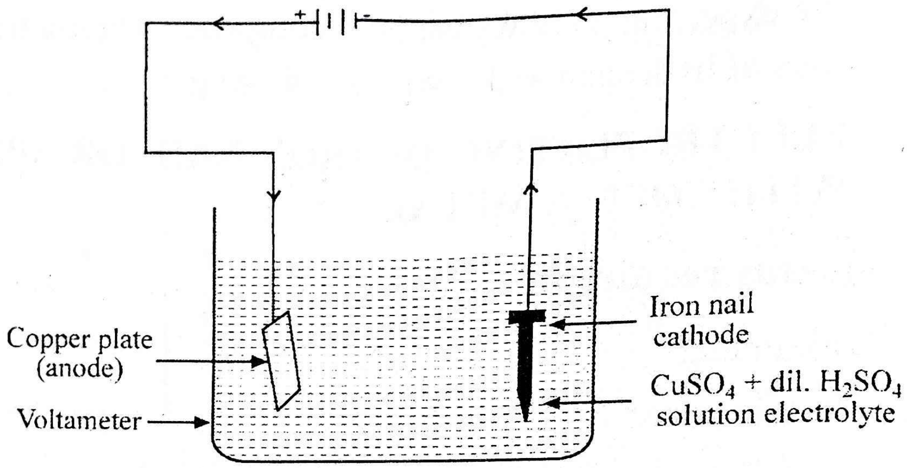
When electricity is passed through an aqueous solution of $CuSO_4$ (electrolyte) having copper plate as anode and iron nail or spoon as cathode, electrolysis takes place as follows.

$$CuSO_4 \rightleftharpoons Cu^{++} + SO_4^{--}$$

(i) **At Anode:** The SO_4^{--} ions move toward the anode plate, where the copper atom of anode loses two electrons (oxidation takes place) and become copper ion Cu^{++} (Cupric ion). These Cu^{++} ions combine with SO_4^{--} to give $CuSO_4$.

$$Cu^{++} + SO_4^{--} \longrightarrow CuSO_4$$

Because of the loss of this copper atom, the anode slowly becomes thin. The concentration of $CuSO_4$ remains same in the beaker because an equivalent amount of copper dissolves from anode into solution.



ii. **At Cathode (Reduction takes place):** The Cu^{++} ion (cupric) is attracted towards the cathode, where it gains two electrons and becomes copper atom (neutral).

$$Cu^{++} + 2e^- \xrightarrow{\text{Reduction}} Cu^0 \text{ (Neutral)}$$

The neutral or metallic Cu atom is deposited at the iron nail or spoon. Thus the copper that dissolved in the solution is deposited at the nail or spoon and the layer of copper at the spoon is increased slowly.

Electroplating: It is the process, in which a layer of a particular element is coated on the surface of a conductor by the method of electrolysis.

Conditions for Electroplating:

i. The conductor which is to be coated is made the cathode electrode.

ii. The material by which, the conductor to be coated is made the anode electrode.

iii. The electrolyte must contain one of the salt of the anode electrode.

Procedure:

i. Take a beaker containing copper sulphate solution as an electrolyte,

ii. Clean the iron nail or spoon and dip it into the solution and connect it to the negative terminal of the battery, so that it become the cathode.

iii. The copper plate is connected to the positive end (anode) of the battery and joined them to a battery by connecting wires, electrolysis takes place and neutral copper metal deposited at the surface of cathode (iron nail or spoon) and equivalent amount of copper dissolves from anode into solution to maintain its same concentration in beaker (voltameter).

Result:

Process of electroplating is studied.

Conclusion

Thus, the iron nail or spoon is coated with copper by electroplating process

Chapter–four

C. Experiments on quantitative analysis

Equivalent Mass (or weight)

Equivalent weight of an element is defined as the number of parts by weight of the element, which combines with or displaces from a compound 8 parts by weight of oxygen or 1.008 parts by weight of hydrogen or 35.5 parts by weight of chlorine. For example: The equivalent weight (or mass) of Mg is 12 i.e. 12 gram of Mg can displace 1.008 gram of hydrogen or can combine with 8 g of oxygen, we can illustrate with suitable example like

1. $Mg + \text{dil. } H_2SO_4 \longrightarrow MgSO_4 + H_2$

   2 g of hydrogen is displaced by 24 g of Mg

   1 g of hydrogen is displaced by $\frac{24}{2}$ g of Mg

   1.008 parts by mass of H_2 is displaced by $\frac{24}{2} \times 1.008 = 12$

   Equivalent weight of Mg is 12.

2. $2Mg + O_2 \xrightarrow[\text{air}]{\text{burnt in}} 2MgO$

   32 gram of oxygen combines with 48 gram of Mg

1 gm of oxygen combines with $\frac{48}{32}$ gram of Mg.

8 gram of oxygen combines with $\frac{48 \times 8}{32} = 12$

Equivalent weight of magnesium is 12

Relationship between equivalent mass, valency and atomic weight (mass)

$$\boxed{\text{Atomic mass} = \text{Equivalent mass} \times \text{Valency}}$$

Variable Equivalent Weight (Mass)

Equivalent weight of an element depends on its valency, we have

$$\text{Equivalent mass} = \frac{\text{atomic mass}}{\text{valency}}$$

Atomic mass of an element constant but the valency may vary. For example:

Compound	**Element**	**Valency**	**At. Mass**	**Eq. mass** $= \frac{\textbf{At. mass}}{\textbf{Valency}}$
Cu_2O	Cu	1	63.5	63.5/1 = 63.5
CuO	Cu	2	63.5	63.5/2 = 31.75
H_2O	H	1	1.008	1.008/1 = 1.008
CO_2	C	4	12	12/4 = 3
Na_2O	Na	1	23	23/1 = 23
MgO	Mg	2	24	24/2 = 12
ZnO	Zn	2	65.4	65.4/2 = 32.7

Gram Equivalent Mass

If the equivalent weight (mass) of an element is expressed in gram, it is known as gram equivalent weight of that element.

1 gram equivalent of hydrogen = 1.008 grams

1 gram equivalent of oxygen = 8 grams

1 gram equivalent of magnesium = 12 grams

Determination of equivalent mass of an acid, base, salt and ions (radicals)

1. $\text{Eq. mass of an acid} = \dfrac{\text{Molecular weight of acid}}{\text{Bascity of acid}}$

   $\text{Equivalent mass of HCl} = \dfrac{1 + 35.5}{1} = 36.5$

   $\text{Equivalent mass of } H_2SO_4 = \dfrac{2 + 32 + 64}{2} = \dfrac{98}{2} = 49$

2. $\text{Eq. mass of base} = \dfrac{\text{Molecular mass of base}}{\text{Acidity of base}}$

   For example

   $\text{Equivalent mass of NaOH} = \dfrac{23 + 16 + 1}{1} = 40$

   $\text{Equivalent mass of } Ca(OH)_2 = \dfrac{40 + 34}{2} = \dfrac{74}{2} = 37$

3. $\text{Eq. mass of salt} = \dfrac{\text{Molecular mass of salt}}{\text{charge on cation or anion}}$

For example:

$\text{Equivalent mass of NaCl} = \dfrac{23 + 35.5}{1} = \dfrac{58.5}{1} = 58.5$

$$\text{Equivalent mass of } CaCl_2 = \frac{40+71}{2} = \frac{111}{2} = 55.5$$

$$\text{Equivalent mass of } Na_2CO_3 = \frac{46+12+48}{2} = \frac{106}{2} = 53$$

4. $\text{Eq. mass of ions or radicals} = \dfrac{\text{Formula mass of ions}}{\text{Charge present in ions}}$

For example:

$$\text{Equivalent mass of } NH_4^{+} = \frac{14+4}{1} = 18$$

$$\text{Equivalent mass of } SO_4^{--} = \frac{32+64}{2} = \frac{96}{2} = 48$$

$$\text{Equivalent mass of } CO_3^{--} = \frac{12+48}{2} = \frac{60}{2} = 30$$

Electrochemical series (activity series)

When elements are arranged in series on the basis of their electron losing capacity with dilute acid, are known as electrochemical series.

$$\xrightarrow[\text{Electropositive character decreases}]{\text{Li K Ba Sr Ca Na Mg Al Zn Fe Sn Pb H Cu Hg Au Pt}}$$

$Li + dil. + HCl \longrightarrow LiCl + H_2$

$Zn + dil.\ H_2SO_4 \longrightarrow ZnSO_4 + H_2$

$Cu + dil.\ H_2SO_4 \longrightarrow$ do not produce hydrogen gas because it lies below the hydrogen in electrochemical series

Experiment No. 11(A)

Object: TO DETERMINE THE EQUIVALENT MASS OF THE GIVEN METAL BY HYDROGEN DISPLACEMENT METHOD

Apparatus required:

i. Eudiometric tube
ii. Beaker
iii. Short stem funnel
iv. Tall jar
v. Stand with clamp
vi. Chemical balance

Chemicals required:

i. Given metal (eg Mg, Zn, etc.)

ii. Dil. HCl or H_2SO_4 solution

Theory:

When a fixed mass (weight) of the given metal above the hydrogen in electrochemical series is allowed to react with an excess of an acid, the hydrogen gas liberated is collected in eudiometnic tube and measured under standard temperature and pressure.

By knowing the mass of the hydrogen displaced by the fixed mass (or weight) of the metal, the equivalent mass can be calculated from the following relation.

$$\text{Equivalent mass of the metal} = \frac{\text{Mass of metal}}{\text{Mass of hydrogen displaced}} \times 1.008$$

$M + \text{dil. } H_2SO_4 \longrightarrow MSO_4 + H_2$ (where M = any metal)

$Mg + \text{dil. } H_2SO_4 \longrightarrow MgSO_4 + H_2$

Note: Standard temperature and pressure (STP or NTP)

Normal temperature = 273K

Normal pressure = 1 atmospheric pressure = 760 mm of Hg

1 mL of hydrogen at NTP weighs 0.000089 gram.

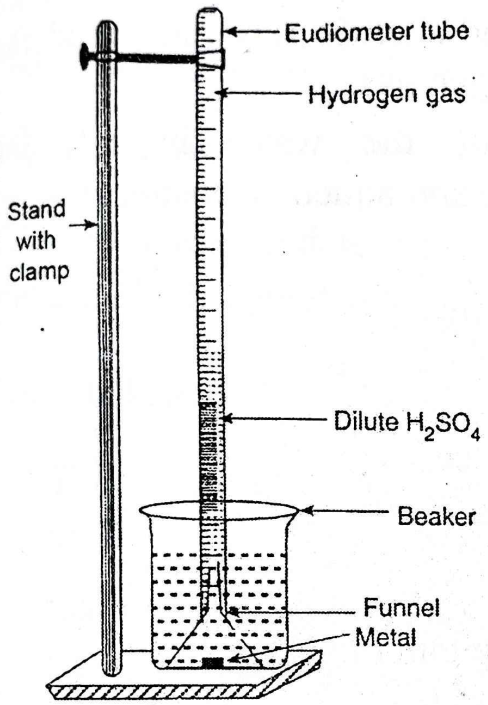


Figure 7(a): Hydrogen displacement method to determine equivalent mass

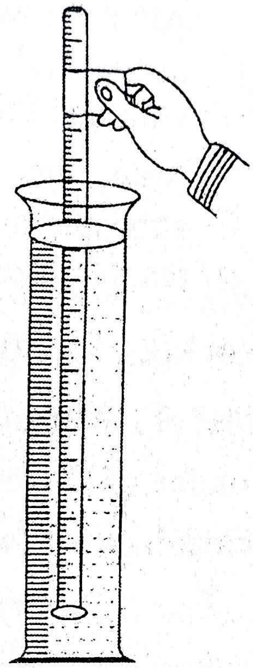


Figure 7(b) : Talljar measuring volume of H_2 gas

Procedure:

1. Take a piece of magnesium ribbon cleaned with sand paper and its mass is determined. Place the metal in a beaker and cover it by a short stem funnel tie with thread and pour down water slowly, so that the stem of the funnel is under water.

2. Half fill an eudiometer tube with dii. H_2SO_4 or HC1 (about 2N strength) and other half with water completely upto brim. Close it with thumb and invert it vertically over the stem of the funnel inside water (no air bubble inside the tube) and clamp the tube vertically as shown in figure 7(a).

3. Add some dilute acid in the beaker to increase the rate of reaction. When the reaction is complete, close the mouth of the eudiometric tube with thumb under water and immerse it under tall jar containing water in an inverted position.
4. After a few minutes, equalize the levels of water both inside and outside the eudiometric tube and read the volume of the moist hydrogen gas.
5. Note the temperature of the water in tall jar, atmospheric room pressure and aqueous tension at given temperature.

Working formula:

Mass of the metal taken = w g

Volume of H_2 gas in tall jar = V_1 cc.

Temperature of water in tall jar = t°C

$\therefore T_1 = (273 + t)K$

Atmospheric pressure (moist) = p mm of Hg

Aqueous tension at t°C = f mm of Hg

Pressure of dry H_2 gas $P_1 = (p - f)$ mm of Hg

Observation and calculation:

Initial condition	Final condition
Volume = V_1 mL	NTP or STP (Normal temp. & pressure)
Pressure of dry gas $P_1 = (p - f)$ mm of Hg	Volume of dry gas V_2 = ?
Room temp. = $T_1 = (273 + t)$ K	P_2 = 1 atm = 760 mm of Hg
	T_2 = 273K

We know from combined gas equation:

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2} \text{ or } V_2 = \frac{P_1V_1T_2}{T_1P_2} \text{ mL (or cc)}$$

Mass of x mL of hydrogen at NTP = ($x \times 0.000089$) g

$\therefore$ 1 mL hydrogen gas at NTP weights = 0.000089 g

$\therefore$ Equivalent mass of the given metal $= \dfrac{w}{x \times 0.000089}$

Result:

Equivalent mass of the given metal is determined.

Conclusion:

Thus, it is able to determine the equivalent mass of the given metal by hydrogen displacement method.

Precaution:

i. There should be no air bubbles inside the eudiometric tube.

ii. Eudiometric tube having hydrogen gas should not touch directly with thumb (expansion takes place due to temperature of thumb)

Questions

1. **Explain why the equivalent mass of an copper metal is not determined by hydrogen displacement method?**

Ans: Because it lies below the hydrogen in electrochemical series i.e. it does not displace hydrogen gas with dilute acid.

2. **The volume of moist hydrogen gas is determined in immersing inside the tall jar filled with water?**

Ans: To overcome excess of atmospheric pressure

Experiment 11(B)

TO DETERMINE THE MASS OF GIVEN SAMPLE OF METAL WITHOUT WEIGHING BY HYDROGEN DISPLACEMENT METHOD. GIVEN EQUIVALENT MASS OF THE METAL IS 12.

Apparatus, Chemical required, Theory and Procedure are same. (Not need to take the mass of the given metal) as 11 experiment.

$$\text{Equivalent mass of the metal} = \frac{\text{Mass of the metal}}{\text{Mass of hydrogen}} \times 1.008$$

Experiment No. 12

Object: TO DETERMINE THE SOLUBILITY OF THE GIVEN SOLUBLE SOLID (SODIUM CHLORIDE) AT LABORATORY TEMPERATURE

Apparatus required:

i. Beaker

ii. Porcelain basin or crucible with lid

iii. Desiccator

iv. Weighing balance

Chemicals required:

i. Sodium chloride

ii. Distilled water

Theory:

The solubility of a chemical substance at a certain temperature is the amount of soluble solid (solute) in gram required to saturate 100 grams of water at that temperature.

For example: 25 grams of copper sulphate dissolves in 100 grams water (solvent) at 30°C to give saturated solution, so solubility copper sulphate at 30°C = 25.

$$\text{Solubility} = \frac{\text{Weight of solute in gram}}{\text{Weight of solvent in gram}} \times 100$$

Procedure:

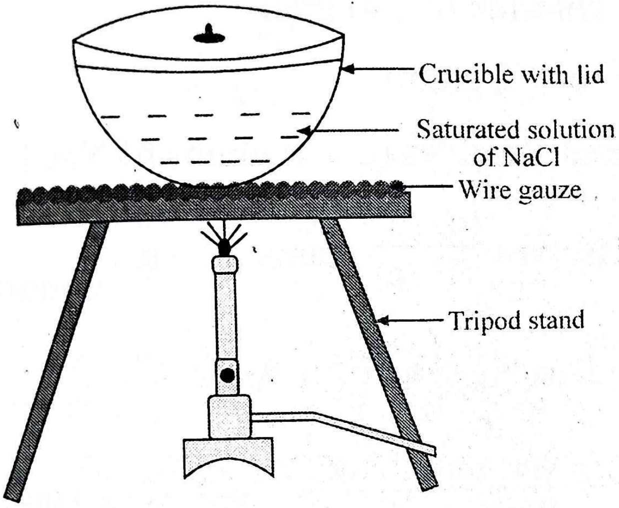


Fig 8(a) : Determination of solubility of NaCl

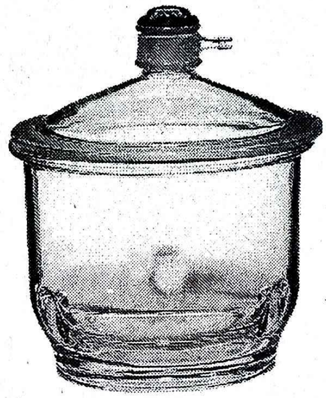


Fig 8(b) : Drying Apparatus dessicator

1. Take about 20-25 mL of distilled water in a clean small beaker and saturated solution of sodium chloride is prepared at room temperature and filtered.
2. Now weigh a small porcelain basin or crucible with lid.
3. Take filtered saturated solution in weighed porcelain basin and its weight is redetermined.
4. Evaporate the solution to dryness, cool the basin in desiccator and weight again.
5. Heating, cooling in desiccator and weighing of porcelain basin or crucible with lid is done upto constant weight (mass).
6. Note the lab room temperature.

Observation and calculation:

Weight of porcelain basin or crucible with lid = a gram.

Weight of crucible with lid and saturated solution NaCl = b gram.

Weight of crucible with lid and anhydrous sodium chloride = c gram (constant)

$\therefore$ Weight of sodium chloride (c – a) gram

Weight of water = (b – c) grams

$\therefore$ (b – c) gram of water dissolves (c – a) grams of NaCl

$\therefore$ 1 gram of water dissolves $\frac{(c-a)}{(b-c)}$ grams of NaCl

$\therefore$ 100 gram of water dissolves $\frac{(c-a)}{(b-c)} \times 100$

$\therefore$ Solubility of NaCl = $\frac{\text{Weight of NaCl in grams}}{\text{Weight of water in grams}} \times 100$

Result

Solubility of sodium chloride is determined.

Conclusion:

Thus, the solubility of sodium chloride at .... is ....

Precaution:

1. Saturated solution of NaCl should be prepared at room temperature.
2. Porcelain basin or crucible with lid should be heated carefully (gently).
3. Final weight of porcelain basin or crucible with lid and anhydrous sodium chloride must be constant.

Experiment No. 13

Object: **TO DETERMINE THE SURFACE TENSION OF GIVEN LIQUID BY DROP COUNT METHOD USING STALAGMOMETER**

Apparatus required:

i. Beaker

ii. Stalagmometer

iii. Pyknometer

iv. Balance

v. Thermometer

Theory

Surface tension of a liquid is the property related to the strength of interparticle forces. It is defined as the force in dyne required to increase the surface of the liquid by unit length. It is denoted by γ.

$$\text{Surface tension of liquid } (\gamma) = \frac{\text{Force}}{\text{Length}}$$

Its unit in CGS system is dyne/cm

In SI system, the unit is Newton per meter (Nm^{-1})

The surface tension of liquid depends upon the forces between the molecules of the liquid. Due to the surface tension, the drops of a liquid are spherical. Therefore, in laboratory, drop count method is used to determine the surface tension of liquids.

The surface tension of the given liquid can be determined by following expression.

$$\gamma_1 = (n_2 d_1)/(n_1 d_2) \times \gamma_2$$

Where γ_1 and γ_2 = surface tension of liquid and water respectively

n_1 and n_2 = the number of drops of liquid and distilled water respectively

d_1 and d_2 = density of liquid and water respectively

Instead of getting the absolute densities, the ratio of d_1/d_2 can be calculated by determining the weight of the same volume of liquid and water using pyknometer or specific gravity bottle.

$$\text{So, } d_1/d_2 = \frac{\text{Weight of certain volume of liquid}}{\text{Weight of same volume of water}}$$

$$\therefore \gamma_1 = (n_2 m_1)/(n_1 m_2) \times \gamma_2$$

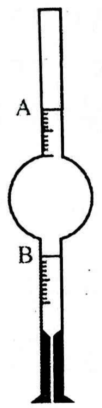


Fig. 9(a) : Traube's Stalag-mometer

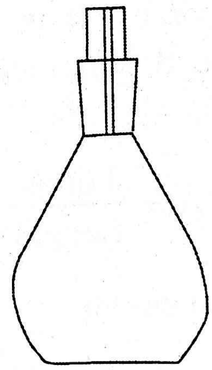


Fig. 9(b) : Specific gravity bottle

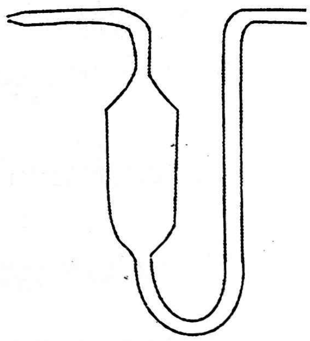


Fig. 9(c) : Pyknometer

1. Clean stalagmometer Pyknometer or specific gravity bottle first with chromic acid, then with dilstilled water and acetone thoroughly.
2. The empty, dry and clean pyknometer is weighed and filled with liquid and distilled water in turn and its weight is redetermined after wiping with a dry filter paper carefully.

3. The stalagmometer having no any obstruction in the capillary tube is rinsed with the given liquid, clamped vertically in a stand as shown in figure. The given liquid is taken in a clean beaker and the liquid is sucked up in the stalagmometer by fixing a rubber tube until the liquid crosses the upper marking.
4. The pinch cock of the rubber tube is adjusted in such a way that rate of fall of liquid does not exceed 18 - 20 drops per minute. The number of complete drops is counted as the liquid passes from upper mark to the lower mark of the stalagmometer This process is repeated three to four times. Similar process is carried out for distilled water.
5. Note the temperature of laboratory room.

Observation table

Liquid	No. of drops	Concurrent drops
1. Given liquid	1 = 2 = 3 =	n_l =
2. Distilled water	1 = 2 = 3 =	n_w =

Room temperature = (t°C)

Surface tension of distilled water at t°C, γ_2 = taken from standard table

The mass of empty dry pyknometer (w_1) =

The mass of pyknometer + liquid (w_2) =

The mass of pyknometer + water (w_3) =

The mass of the given volume of liquid = ($w_2 - w_1$) gm

The mass of the given volume of water ($w_3 - w_1$) gm

Thus, the surface tension of liquid = (γ_1) = ($n_2 n_1$) [($w_2 - w_1$) gm/($w_3 - w_1$)] × γ_2

Result:

The surface tension of the given liquid at ....°C is.............

Conclusion:

Surface tension of the given liquid can be determined by using stalagmometer.

Experiment No. 14

Object: TO STUDY THE RATE OF FLOW OF LIQUID THROUGH OSTWALD'S VISCOMETER AND DETERMINE THE RELATIVE VISCOSITY OF UNKNOWN LIQUID

OR

TO DETERMINE THE RELATIVE AND ABSOLUTE VISCOCITY OF ALCOHOL USING OSTWALD'S VISCOMETER

Apparatus required:

i. Ostwald's viscometer
ii. Pyknometer
iii. Stop watch
iv. Pipette
v. Clamp with stand

Chemicals required:

i. Distilled water
ii. Alcohol (or any organic solvent)

Theory

Viscosity:

It is the property of liquid which resists to the flow of liquids. The greater the intermolecular forces of attraction, greater will be the viscosity of the liquid.

For example: Glycerine has more viscosity than water due to hydrogen bond, higher molecular weight

Coefficient of Viscosity:

It is defined as the force required per unit area to maintain unit velocity difference of viscosity between two parallel layers of liquid as unit distance from each other.

The intermolecular forces of attraction thus produce a retarding force to flow of liquid. For this Newton deduced that this retarding force is proportional to the velocity gradient (dv/dx) normal to the direction of and area of contact 'A' between moving layers of liquid.

$$\text{i.e. } F \propto A\frac{dv}{dx} \text{ or } F = \eta A.\frac{dv}{dx} \quad \text{.............................(i)}$$

Where η is proportionality constant and is called coefficient of viscosity or simply viscosity of a liquid. Rearranging equation (i), the coefficient of viscosity, η is given by

$$\eta = \frac{Fdx}{A.dv} \quad \text{..........................(ii)} \qquad \eta \rightarrow \text{ita.}$$

A property of liquid which resists the flow of the liquids is known is viscosity. The relative viscosity of a liquid compared to water can be determined by using Ostwald's viscometer with the help of following formula.

$$\frac{\eta_l}{\eta_w} = \frac{d_l t_l}{d_w t_w} \text{ or } \eta_l = \eta_w \left(\frac{d_l t_l}{d_w t_w}\right)$$

Where, η_l = viscosity of liquid

η_w = viscosity of water

t_l = time of flow of liquid

t_w = time of flow of water

d_l = density of liquid

d_w = density of water

The ratio of densities (d_l/d_n) can be found out by determining the weight of the same volume of liquid and water using specific gravity bottle.

$$\therefore \frac{d_l}{d_w} = \frac{\text{wt. of certain volume of liquid}}{\text{wt. of certain volume of water}} = \frac{m_l}{m_w}$$

Procedure

1. Clean the Ostwald's viscometer and pyknometer first with chromic acid, washed with water, rinse with acetone and dry in air oven at 120°C. Attach a rubber tube in the limb containing capillary tube.

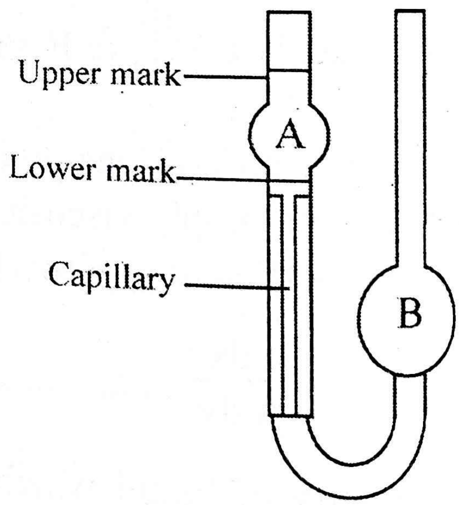


Fig 10 : Ostwald's viscometer

2. Introduce a definite volume (usually 10 mL) of distilled water in the other limb of viscometer by calibrated pipette.

3. Suck up the liquid by means of a rubber tubing little beyond the upper mark of the viscometer. Allow the liquid to flow through the capillary tube and start the stop watch, when the liquid reaches the upper mark and stop the stop watch as soon as water reaches the lower mark. This is the time taken by water to flow the volume of water from upper mark to lower mark. Repeat this process three or four times.
4. Drain out the distilled water, rinse with acetone and dry in air oven at 120°C.
5. Introduce the same volume of the liquid (10 mL) by calibrated pipette as in step (2), determine the time taken by the liquid to flow the volume of liquid from upper to lower mark. Repeat this process three or four times.
6. Determine the mass of dry and empty pyknometer and fill the pyknometer with distilled water and its mass is redetermined. Remove the water, dry the pyknometer and fill with alcohol (or any organic solvent) and determine its weight (mass).

Observation table

Liquid	Flow time (sec)	Mean flow time (sec)
Water	1 2 3	
Organic liquid	1 2 3	

Temperature (t°C) =

Viscosity of water at (t°C) = η_w = known standard value

Calculation

Mass of empty pyknometer = m_1 grams

Mass of pyknometer + water = m_2 grams

Mass of pyknometer + liquid (alcohol) = m_3 grams

Mass of water .... = $(m_2 - m_1)$ grams

Mass of alcohol mL = $(m_3 - m_1)$ grams

η_l = viscosity of liquid, η_w = viscosity of water

$$\eta_l = \eta_w \times \frac{(m_3 - m_1)t_l}{(m_2 - m_1)t_w}$$

Result:

The viscosity of the given organic solvent (alcohol) at t°C is found to be..............Poise.

Conclusion:

The relative viscosity of given liquid can be measured by using Ostwald's viscometer.

Questions

1. What is viscosity and coefficient of viscosity?
2. Give the unit of viscosity.
3. Why glycerine has higher viscosity than water?
4. What is the effect of temperature on
   (a) surface tension
   (b) viscosity of liquid
5. Define surface tension.
6. Write the unit of surface tension in CGS and SI unit.
7. What is stalagmometer and Ostwald's viscometer?
8. What is pyknometer? Give its use.

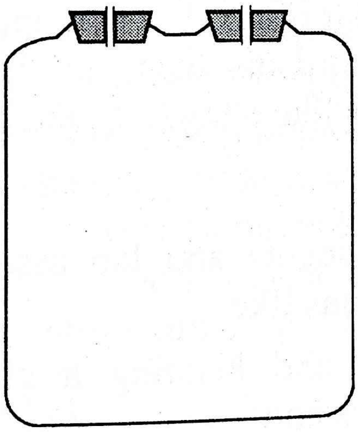
Chapter–five

D. Experiments on preparation of gas and study its properties.

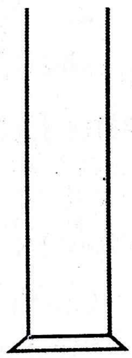
General ideas about the preparation and collection of gases

1. Preparation

The main apparatus used for the preparation of gases in the laboratory depends upon the nature of the reaction to be performed.



Woulfe's bottle



Gas jar

i. If the gases are to be prepared without the application of heat, we use generally Woulfe's bottle. For example: H_2, H_2S, CO_2, NO, etc.

ii. If the gases are to be prepared with the application of heat, we use generally rounded bottomed flask (RB flask), hard test tube, etc. For example, O_2, NH_3, HC1, SO_2, etc.

Thistle funnels are meant for introducing liquid reagents and dilute acid.

Dropping funnels: They are used for introducing liquid reagents and concentrated acid.

Delivery tubes: They are used for the outlet of the gases.

2. Collection of gases

The method of collecting a gas in jar depends upon the nature of the gas.

i. If the gas does not react with water and is insoluble in water, it is generally collected in gas jar by downward displacement of water. For example, H_2, O_2, N_2, etc.

ii. If the gas reacts with water or is moderately and highly soluble in water, it is collected in gas jar by downward displacement of air (For example, lighter gas NH_3) and by upward displacement of air (For example, heavier gases like CO_2, SO_2, etc.)

Apparatus fitting

It is important for teachers, students and lab assistants to know simple skill for the operations like

i. Cutting a glass tube and bending a glass tube through different angles and

ii. Boring a cork

Objective: TO CUT THE GLASS TUBE INTO THREE EQUAL PARTS, ROUND OFF ITS SHARP EDGES AND TO BEND THEM AT

a. Acute angle

b. Right angle

c. Obtuse angle

Apparatus required:

i. Glass tubing
ii. Asbestos sheet
iii. Triangular file
iv. Bunsen burner

Theory:

Glass is an amorphous, transparent, rigid, supercooled liquid having infinite viscosity and composed of a number of metallic silicates with general formula. xA_2O, yBO, $6SiO_2$, where

A = alkali metal (e.g. Na, K)
B = bivalent metal (e.g. Ca)
x and y are number of molecules

For example: sodalime glass: Na_2O, $CaO.6SiO_2$
potash lime glass: K_2O, $CaO.6SiO_2$

The main material of glass is silica (SiO_2) M.P. is 1600°C. The component added to silica during the manufacture of glass largely determines the properties of glass like strong, coloured or colourless nonattackable reagent, heat resistant or not, etc. The glass tube is soda lime glass or soft glass and a mixture of sodium and potassium silicates, which is easily fusible when glass is heated, it becomes gradually soft and can be transformed into desire shaped.

Procedure:

i. Put the glass tubing on the edge of the bench top, hold it strongly with left hand and make a single deep scratch with a sharp triangular file towards yourself

ii. Hold the glass tube with your hands horizontally in the air by keeping the scratch point outside and press the tube inward by placing your thumbs just at the black side of the scratch, so that it breaks with sharp cut edge as shown in figure (a).

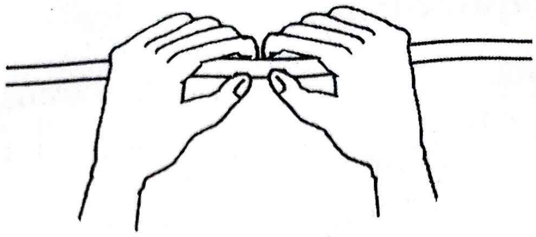


Fig. (a) : Breaking the tubing into two

iii. Round off the sharp cut edge of the tube one by one in the non-luminous flame by rotating till the tip becomes soft and then rest it on the asbestos sheet.

iv. Fit up the flame spreader on the top of the burner (to get fish tail flame) and heat the glass tube at the suitable position over the flame by holding it horizontally with your fingers at both end and rotate the glass tube constantly until it becomes soft enough for bending, then place it on the asbestos sheet and bend the tube quickly into desired angle by applying gently pressure. Keep it on the asbestos sheet for cooling. Bend other tubes one by one at different angles.

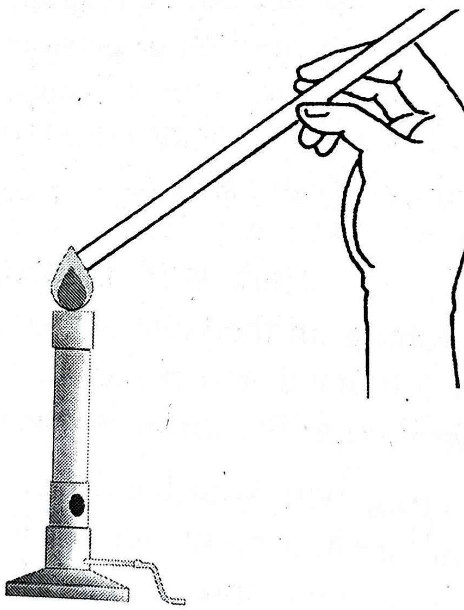


Fig. (b) : Rounding the edges

Precaution:

i. Glass tube should not heat for a long time.
ii. Triangular file should not draw toward and backwards like saw.
iii. If water drops or moisture is present within glass tube, we should not heat the tube otherwise it may crack.

Making of delivery glass tubes:

When you have the skill of cutting and bending of glass tubes, you are able to make the delivery tubes of different shapes like

i. Bent the glass tube twice right angles in the same direction for the gas preparation of CO_2, SO_2, HCl, HBr, etc.
ii. Bent the glass tube at acute angles in the opposite directions, for example: H_2 gas.

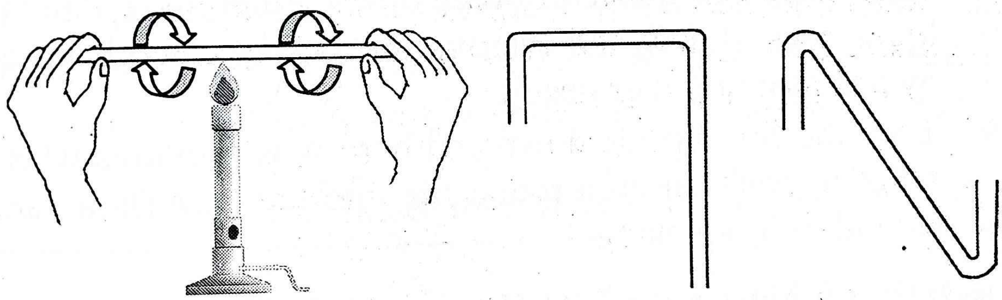


Fig (c) : Bending a glass tube at right angle, acute angles

Object: **TO BORE A HOLE IN A CORK AND INSERT A GLASS TUBE**

Apparatus required:

i. Cork
ii. Cork pressure
iii. Cork borer

After making a delivery tube of the desired shape, let us see how, we can bore holes in the cork.

i. Select a suitable cork (cork should be smooth and cracks free) whose narrow end just fit into the neck of flask, Woulfe bottle and wet the cork with water (if it is wooden), press it by rolling it in a cork pressure or under the shoe gently till it becomes soft and flexible.

ii. Select a cork borer in such a way that the diameter of cork borer should be slightly less than that of glass tube to be inserted and keep the cork on the top of the bench with its narrow end facing upward. Mark the position to be bored. If you have to bore only one hole, bore it at the centre.

If you have to bore two holes choose the appropriate portion and mark it.

iii. Screw the cork borer into the mark and push the borer vertically (perpendicular) downward gently with rotating. Continue boring till the borer is almost through to the other end (appearance of bulge on the other end). Pull the borer back slowly and remove the pieces of cork inside by means of a rod or needle.

iv. Then the cork upside down and bore it as previous where bulge appears. Insert a round file into the bore (hole) and smooth it by spinning.

Inserting a glass tube into the cork

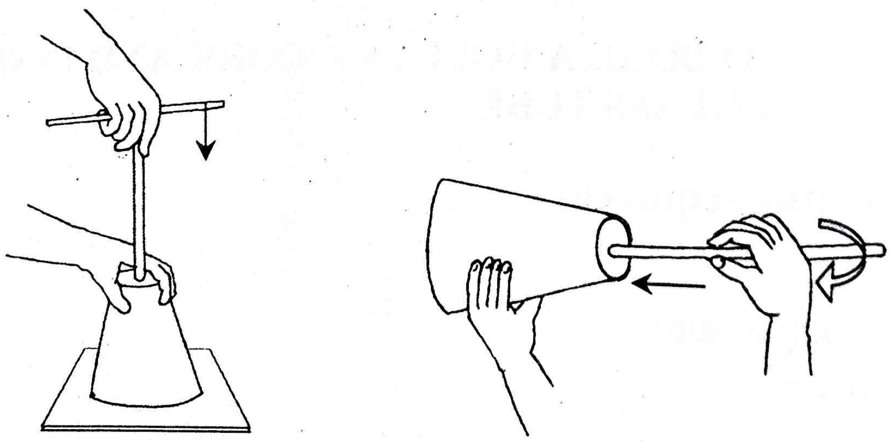


Fig (d): Boring and inserting a glass tube into a cork

v. Wet the end of glass tube (like delivery tube, thistle funnel, etc.) with few drops of water or glycerine. Hold the bored cork in one and glass tube in the other hand then insert the glass tube gently with spinning motion shown in the figure (d).

vi. At last, fit the cork with the glass tubing into the neck of the round bottom flask or Woulfe's bottle to get your apparatus ready.

To test the apparatus is air tight or not:

Pour down some water through the thistle funnel into Woulfe bottle or RB flask such that the lower end of the funnel is just under the water, Blow in through the other end of the delivery tube, water level will rise in the thistle funnel to a certain height, quickly close the end of the delivery tube with your finger (or tongue), if the water does not fall down, the apparatus is air tight, if water falls, the apparatus is not air tight (air tight is done with candle, wax, grease, etc.)

Experiment No. 15

Object : TO PREPARE AND COLLECT HYDROGEN GAS AND TO STUDY PROPERTIES

Apparatus required:

i. Woulfe's bottle
ii. Thistle funnel
iii. Delivery tube
iv. Water trough
v. Gas jar
vi. Test tube, etc.

Chemicals required:

i Granulated zinc
ii. Dil. H_2SO_4 or HCl
iii. Litmus paper
iv. $KMnO_4$ solution
V $K_2Cr_2O_7$ solution
vi. $FeCl_3$ solution

Theory:

Metals like Mg, Al, Ca, Zn. etc. which lie above the hydrogen in electrochemical series react with dilute sulphuric or hydrochloric acid to give hydrogen gas, which is collected in gas jar by downward displacement of water.

Reaction:

$$Zn\ (s) + \text{dil. } H_2SO_4 \longrightarrow ZnSO_4\ (aq.) + H_2$$

Ionic Reaction:

$$Zn^{\circ}\ (s) + 2H^+\ (aq) \longrightarrow Zn^{++}\ (aq.) + H_2(g)$$

Here zinc is oxidized into Zn^{++} by H^+ ions and H^+ ions are reduced by zinc into H_2 gas.

Nascent hydrogen

It is newly born hydrogen which is produced by the reaction of granulated zinc and dilute sulphuric acid. It is strong reducing agent (reductant).

Procedure

1. Add some granulated zinc pieces into the woulfe's bottle and pour down dilute sulphuric acid through the thistle funnel, such that the lower end of the funnel dips under the acid as shown in figure 11 and ensure that apparatus must be air tight.

2. Allow the gas bubbles to escape for 2-3 minutes from the delivery tube to remove out air.

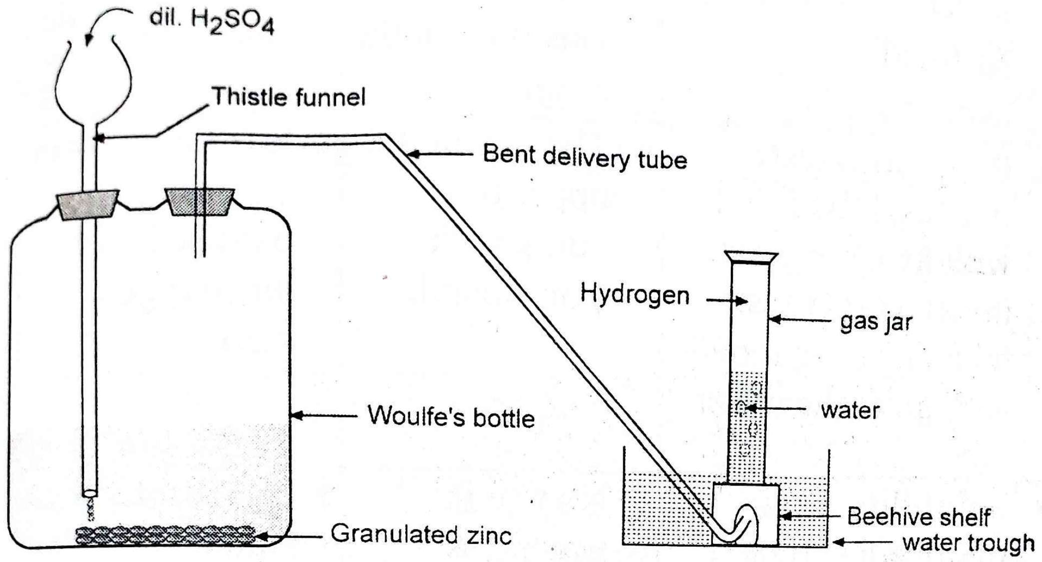


Figure 11 : Lab preparation of hydrogen gas

3. Collect the hydrogen gas in gas jar by downward displacement of water and perform the following experiments to study the properties of the gas.

Observation Table

Experiment	Observation	Inference
1. Colour and odour test: Note the colour and odour (smell) of gas	1. No colour and odour	1. The hydrogen gas is colourless and odourless gas.
2. Litmus paper test: Introduce moist red and blue litmus paper into the jar of hydrogen gas.	2. No any change in the colour of litmus paper.	2. The hydrogen gas is neutral to litmus paper.

3. Combustibility test: Introduce a burning match stick into the jar filled with hydrogen gas	3. The burning of match stick extinguishes. Gas burn at the mouth	3. It is combustible gas but not a supporter of combustion.
4. Pop sound test: In a test tube filled with hydrogen gas, invert a other test tube mouth to mouth and ignite the upper tube.	4. The has in the upper tube burns with 'pop' sound.	4. It is lighter than air. It is oxidised with air (oxygen gas).
5. Solubility Test: Invert a test tube filled with hydrogen gas over water in a trough and move up and down	5. No rise in water level inside the test tube	5. It is insoluble in water.
6. Reaction with acidified potassium permanganate ($KMnO_4$) solution. a. Pass hydrogen gas in test tube containing acidified $KMnO_4$ with dil. H_2SO_4 b. Add 1–2 pieces of granulated zinc in test tube containing acidfied solution of $KMnO_4$	a. No any change in colour b. Pink colour of $KMnO_4$ changes into colourless solution	a. Molecular hydrogen does not reduce acidified $KMnO_4$ solution b. Nascent hydrogen reduces $KMnO_4$ to colourless $MnSO_4$ solution.

7. Reaction with acidified solution of potassium dichromate ($K_2Cr_2O_7$) solution		
a. Pass H_2 gas into the test tube containing acidfied $K_2Cr_2O_7$	a. No any change in colour	a. Molecular hydrogen does not reduce acidfied $K_2Cr_2O_7$ solution.
b. Add 1–2 pieces of zinc inside the test tube containing acidfied solution of $K_2Cr_2O_7$.	b. Orange red coloured $K_2Cr_2O_7$ changes into green coloured solution.	b. Nascent hydrogen reduces potassium dichromate into green colour chromium sulphate.
8. Reaction with acidified solution of ferric chloride ($FeCl_3$) solution		
a. Pass H_2 gas into test tube containing acidified solution of $FeCl_3$ solution.	a. No any change in colour	a. Molecular hydrogen does not reduces ferric chloride solution.
b. Add 1–2 pieces of zinc in the test tube containing acidfied $FeCl_3$ solution	b. Ferric chloride changes from reddish yellow to light green.	b. Nascent hydrogen reduces ferric chloride (reddish) to light green coloured $FeCl_2$.

1. **Reaction with acidified potassium permaganate ($KMnO_4$) Solution**

a. $KMnO_4$ + dil. H_2SO_4 + H_2 (molecular) → No reaction

b. $\underset{\text{}}{Zn + \text{dil. } H_2SO_4 \rightarrow ZnSO_4 + \underset{\text{Nascent hydrogen}}{2[H]}}$

$\underset{\text{pink}}{2KMnO_4} + \text{dil. } 3H_2SO_4 + [H] \rightarrow K_2SO_4 + \underset{\text{colourless}}{2MnSO_4} + 8H_2O$

2. **Reaction with acidified potassium dichromate solution**

a. $K_2Cr_2O_7$ + dil. H_2SO_4 + H_2 → No reaction

b. $\underset{\text{orange red}}{K_2Cr_2O_7} + \text{dil. } H_2SO_4 + 6[H] \rightarrow K_2SO_4 + \underset{\text{green}}{Cr_2(SO_4)_3} + 7H_2O$

3. **Reaction with acidified ferric chloride solution ($FeCl_3$)**

a. $FeCl_3$ + dil. HCl + H_2 ⟶ No reaction

b. Zn + dil. HCl ⟶ 2[H]

$\underset{\text{reddish yellow}}{FeCl_3 + [H]} \xrightarrow{\text{reduction}} \underset{\text{Light green}}{FeCl_2} + HCl$

Result:

Hydrogen gas is prepared in the laboratory and its properties are tested.

Conclusion:

Thus, hydrogen gas is prepared by treating zinc pieces with dilute sulphuric acid or dilute hydrochloric acid.

Precaution

1. The apparatus must be air tight.
2. There should not be any flame near the hydrogen, otherwise explosion takes place.
3. Hydrogen gas must be free from air.

Questions

1. **Why nitric acid is not used for the lab preparation of hydrogen gas?**

Ans: Nitric acid oxidises hydrogen gas into water, so it is not used.

2. **Concentrated sulphuric acid is not used during the lab preparation of hydrogen gas. Why?**

Ans: When granulated zinc is heated with conc. H_2SO_4, we get sulphur dioxide gas instead of hydrogen gas.

$$Zn + \text{conc. } 2H_2SO_4 \xrightarrow{\Delta} ZnSO_4 + 2H_2O + SO_2$$

3. **How can you show nascent hydrogen is more powerful reductant than molecular hydrogen gas?**

Experiment No. 16

Object : TO PREPARE AND COLLECT AMMONIA GAS AND TO INVESTIGATE ITS PROPERTIES.

Apparatus required:

i. Hard glass test tube or round bottom flask
ii. Gas jar

iii. Delivery tube

iv. Burner

v. Test tube stands

Chemicals required:

i. Slaked lime [$Ca(OH)_2$]

ii. Ammonium chloride (sal-ammoniac)

iii. $CuSO_4$ solution

iv. $FeCl_3$ solution

v. Mercurous nitrate solution

Theory:

When paste of slaked lime and sal-ammoniac (NH_4Cl) is heated, we get ammonia gas, which is collected in gas jar by downward displacement of air (because it is lighter than air.)

$$\underset{\text{Sal-ammnoiac}}{2NH_4Cl} + \underset{\text{slaked lime}}{Ca(OH)_2} \xrightarrow{\Delta} CaCl_2 + 2NH_3 + 2H_2O$$

Ionic equation:

$$NH_4^+ (aq.) + OH^- \rightleftarrows H_2O\ (l) + NH_3\ (g)\uparrow$$

Procedure

Take paste of slaked lime (2 parts) and ammonium chloride (1 part) in round bottom flask and heated gently, we get ammonia gas, which is collected in gas jar by downward displacement of air. Apparatus is fitted as shown in figure 12.

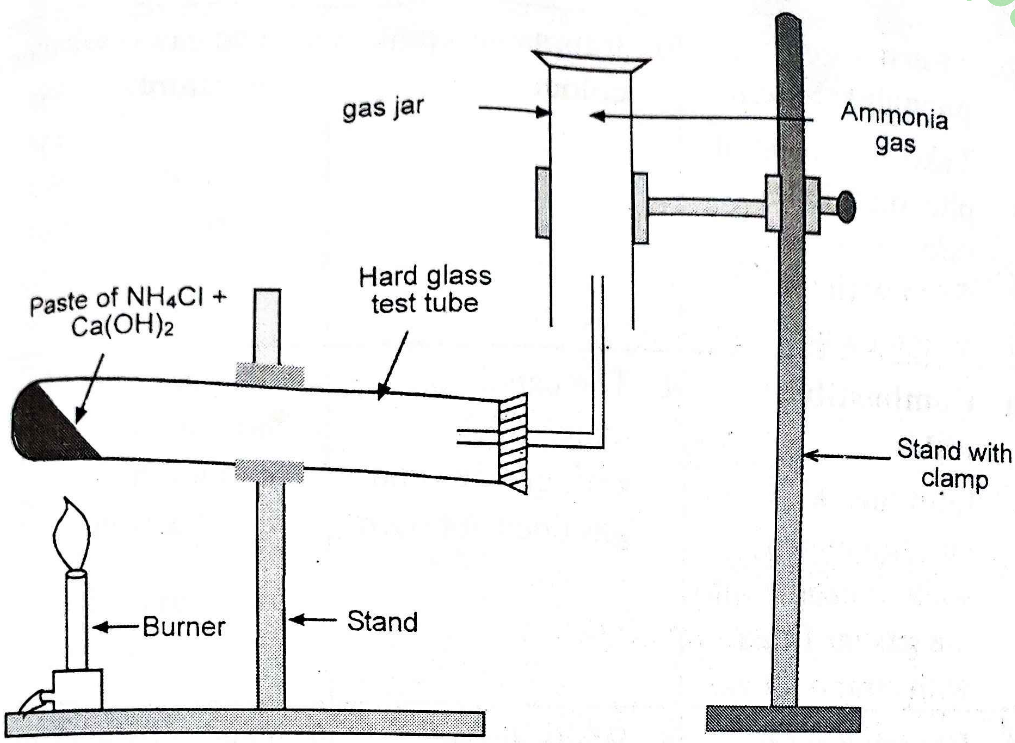


Figure 12 : Lab preparation of ammonia gas

Observation table for ammonia gas

Experiment	Observation	Inference
1. **Colour and odour test:** Note colour and odour of ammonia gas	1. No colour but pungent odour (irritating smell)	1. The ammonia gas is colourless gas with pungent smell.
2. **Solubility test:** Invert a test tube filled with ammonia gas over water in a trough	2. Water level rises up into the test tube.	2. The gas is highly soluble in water
3. a. **Test with moist litmus paper:** Introduce moist red and blue litmus paper into the gas gar filled with ammonia gas	a. The red litmus paper turns into blue but no any effect on blue litmus paper	a. The gas is alkaline in nature

b. **Test with phenolphthalein** Take 1-2 drops of phenolphthalein into a test tube filled with ammonia gas.	b. It turns into pink colour.	b. The gas is alkaline in nature.
4. **Combustibility test:** Introduce a burning match stick or candle into the gas jar filled with ammonia gas.	4. The candle or match stick extinguishes and gas does not burn.	4. The gas is neither combustible nor supporter of combustion.
5. **Reaction with Conc. HCl** Introduce a few drops of conc. HCl in a gas jar filled with ammonia gas	5. White dense fumes are produced	5. Due to formation of ammonium chloride.
6. **Reaction with $CuSO_4$ solution** Take about 1-2 mL of $CuSO_4$ solution with less amount of ammonia and then with excess of ammonia solution.	6. At first bluish white precipitate formed, which disolves in excess of ammonia giving deep blue (indigo) colour.	6. Bluish white precipitate due to formation of $Cu(OH)_2$ and deep blue colour due to formation of $[Cu(NH_3)_4]\ SO_4$
7. **Reaction with $FeCl_3$ solution:** Pass the ammonia gas through $FeCl_3$ solution containing in test tube.	7. A reddish brown precipitate forms.	7. Due to formation of ferric hydroxide.

8. **Reaction with mercurous nitrate solution:** Dip a piece of filter paper in mercurous nitrate solution and expose it near ammonia gas.	8. The filter paper becomes black.	8. Due to formation of mixture of black metallic mercury and mercuric amino nitrate
9. **Reaction with Nessler's reagent** Take 1-2 drops of Nessler's reagents (alkaline solution of K_2HgI_4) in test tube filled with ammonia gas.	9. Reddish brown precipitate appears.	9. Due to formation of iodide of Million's base.

Reactions:

1. **With water**

a. $NH_3 + H_2O \longrightarrow NH_4OH$

Ammonium hydroxide

2. **With conc. HCl**

a. $NH_3 + HCl \longrightarrow NH_4Cl$

Ammonium chloride

dense white fumes

3. **With $CuSO_4$ solution**

$CuSO_4 + NH_4OH \longrightarrow Cu(OH)_2\downarrow + (NH_4)_2SO_4$

Bluish white ppt

$CuSO_4 + 4NH_4OH \longrightarrow [Cu(NH_3)_4]^{++} SO_4^{--} + 4H_2O$

Tetramine copper II sulphate

deep blue (indigo colour)

4. **With ferric chloride solution:**

$$FeCl_3 + 3NH_4OH \longrightarrow \underset{\text{Brown ppt}}{\underset{\text{Ferric hydroxide}}{Fe(OH)_3\downarrow}} + 3NH_4Cl$$

5. **With mercurous nitrate solution**

$$Hg_2(NO_3)_2 + 2NH_3 \longrightarrow [Hg + Hg(NH_2)NO_3]\downarrow + NH_4NO_3$$

Mixture of mercury and meruric amino nitrate (black ppt)

6. **With Nessler's reagent**

$$\underset{\text{Nessler's reagent}}{K_2HgI_4} + 2NH_4OH \longrightarrow NH_4I + 2KI + NH_2HgI + H_2O$$

$$2NH_2HgI + H_2O \longrightarrow NH_2HgO.HgI\downarrow + NH_4I$$

Iodide of Millions base (Reddish brown ppt)

Result :

Ammonia gas is prepared in laboratory and its properties are investigated.

Conclusion:

Thus, ammonia gas can be prepared by heating paste of slaked lime and ammonium chloride and able to investigate its properties.

Precaution

1. Round bottom flask should be heated gently.
2. Different tests should perform carefully and fast (because formation of ammonia is fast)

Note:

1. **What is the suitable dehydrating reagent for ammonia gas?**

Ans: Quick lime (CaO) is the suitable dehydrating reagent for ammonia gas because it does not react.

2. **Why Conc. H_2SO_4,P_2O_5 and fused anhydrous $CaCl_2$ is not used for dehydrating ammonia gas?**

Ans: Because they react with ammonia like

Conc. $H_2SO_4 + 2NH_3 \longrightarrow (NH_4)_2SO_4$

$CaCl_2 + 8NH_3 \longrightarrow CaCl_2.8NH_3$

Fused anhydrous

$P_2O_5 + 3H_2O + 6NH_3. \longrightarrow 2(NH_4)_3PO_4$

Fused anhydrous ammonia phosphate

Experiment No. 17

Object : TO PREPARE CARBON DIOXIDE GAS AND TO INVESTIGATE ITS PROPERTIES

Apparatus required:

i. Woulfe's bottle
ii. Thistle funnel
iii. Delivery tube
iv. Gas jar
v. Test tube stand
vi. Cork

Chemicals required:

i. Marble chips or lime stones
ii. Dil. HCl
iii. Limewater (fresh)
iv. Magnesium ribbon
v. Caustic soda solution, etc.

Theory:

When marble chips or limestones are reacted with dil. hydrochloric acid, we get CO_2 gas, which is collected in gas jar by upward displacement of air (because it is heavier than air)

$$CaCO_3 + \text{dil. } 2HCl \longrightarrow CaCl_2 + H_2O + CO_2\uparrow$$

Ionic equation:

$$CaCO_3(s) + 2H^+ \longrightarrow Ca^{++}(aq.) + H_2O\ (l) + CO_2\ (g)$$

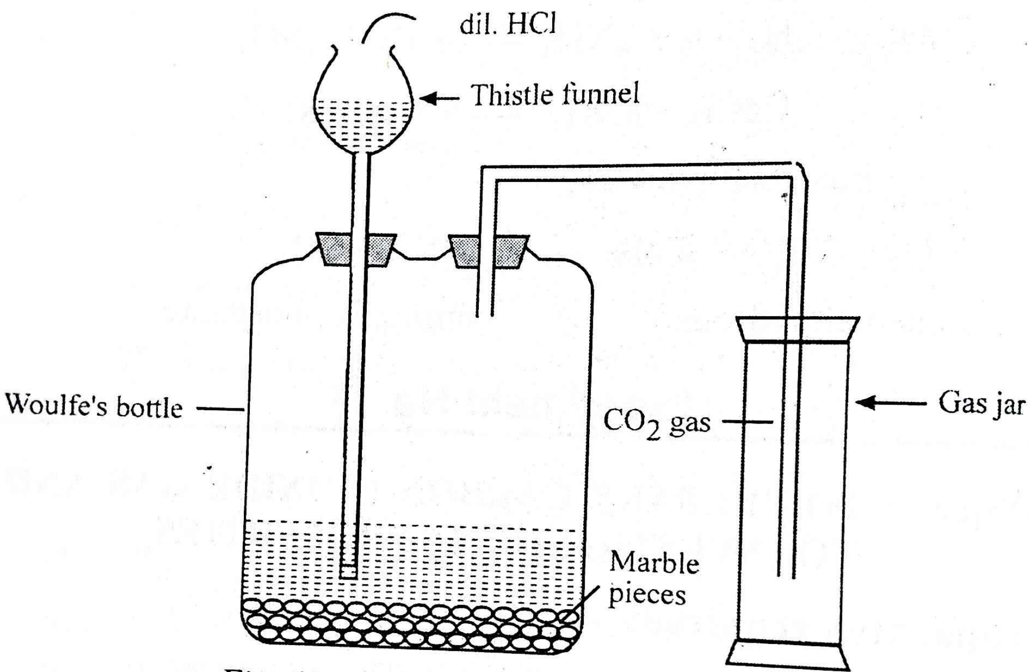


Fig. 13 : Lab preparation of CO_2 gas

Procedure:

1. Introduce some marble chips or limestones into the Woulfe's bottle. Check air tightness of the apparatus and pour down dilute hydrochloric acid through the thistle funnel such that the lower end of the funnel just dips under the dilute acid and apparatus fitted as shown in figure 13.
2. Reaction occurs with effervescence and the CO_2 gas is collected in gas jar by upward displacement of air.

3. Perform the following experiments to study the properties of the CO_2 gas.

Observation Table

Experiment	Observation	Inference
1. **Colour and odour test:** Note colour and odour of CO_2 gas	1. No colour and odour	CO_2 gas is colourless and odourless gas.
2. **Solubility test:** Invert a test tube filled with CO_2 gas over water in a trough and move up and down the water.	2. The water level slightly rises inside the tube.	2. This gas is slightly soluble in water.
3. **Litmus paper test:** Introduce moist blue and red litmus paper into the gas jar filled with CO_2 gas.	3. The blue litmus paper turns into faint red but not any change in red litmus paper.	3. The gas is weakly acidic in nature.
4. **Combustibility test:** Introduce a burning match stick or candle into the gas jar full of CO_2 gas.	4. The burning match stick or candle extinguishes and the gas does not burn.	The gas is neither combustible nor supporter of combustion.
5. **Test with burning magnesium ribbon:** Introduce a burning Mg ribbon with tongs into the gas jar filled with CO_2 gas.	5. Mg ribbon continues to burn inside forming black particles and white powder as resides	CO_2 supports the combustion of Mg forming white magnesium oxide and black carbon particles.

6. Test with lime water: a. Pass CO_2 gas (little) into a test tube containing fresh colourless lime water upto saturation.	6. a. Lime water turns milky, which on excess passing of CO_2 becomes colourless.	6. a. Due to formation of insoluble $CaCO_3$ and colourless solution due to formation of $Ca(HCO_3)_2$.
b. Heat the colourless solution.	b. White turbidity (ppt) reappears	b. Due to formation of insoluble $CaCO_3$.
7. Reaction with caustic soda (NaOH) solution: Fill a test tube with CO_2 gas, add few drops of NaOH solution, close its mouth with thumb, shake well and invert it over water in the trough.	7. The level of water rises up in the test tube.	The CO_2 gas is highly absorbed by caustic soda (NaOH) solution.

Important reactions involved

1. **Action with water**

a. $H_2O + CO_2 \longrightarrow H_2CO_3$

Carbonic acid (weak) ;

2. **CO_2 gas is decomposed by burning magnesium ribbon (because it evolves high heat energy), which decomposes CO_2 gas into carbon and oxygen gas.**

a. $CO_2 + Mg \longrightarrow 2MgO + C$

white powder black

3. **Reaction with lime water (colourless)**

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

Lime water (less) Milky coloured

Colourless

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

Excess Calcium bicarbonate

Colourless

c. $Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 \downarrow + H_2O + CO_2$

Calcium bicarbonate White turbidity

4. **Reaction with caustic soda (NaOH) solution**

a. $CO_2 + 2NaOH \longrightarrow Na_2CO_3 + H_2O$

Good absorbent

Result:

Carbondioxide gas is prepared in the laboratory and its properties are studied.

Conclusion:

Thus, CO_2 gas can be obtained from the reaction of marble chips and dil. hydrochloric acid and investigated its properties.

Precaution:

1. Apparatus must be air tight.
2. Thistle funnel should dip inside the dilute HCl.
3. We should only use dilute hydrochloric acid.

Note:

Q.1 Why dilute H_2SO_4 is not used for the lab preparation of CO_2 gas?

Ans: In starting, we get CO_2 gas with dilute H_2SO_4 but when there is formation of insoluble calcium sulphate, which forms a coat or covering around the marble chips and reaction stops.

$$CaCO_3\,(s) + \text{dil. } H_2SO_4 \longrightarrow CaSO_4(s) + H_2O + CO_2\uparrow$$

Experiment No. 18

Object: TO PREPARE AND COLLECT HYDROGEN SULPHIDE GAS AND TO INVESTIGATE ITS PROPERTIES

A. PHYSICAL
B. ANALYTICAL AND
C. REDUCING PROPERTIES

Apparatus required:

i. Woulfe's bottle
ii. Thistle funnel
iii. Delivery tube
iv. Gas jar
v. Cork

Chemicals required:

i. Pieces of ferrous sulphide (FeS)

ii. dilute H_2SO_4

Theory:

When ferrous sulphide is reacted with dilute sulphuric acid, we get hydrogen sulphidc gas, which is collected in gas jar by upward displacement of air.

$$FeS\ (s) + H_2SO_4 \longrightarrow FeSO_4 + H_2S\uparrow$$

$$FeS\ (s) + 2HCl \longrightarrow FeCl_2 + H_2S\uparrow$$

$$FeS\ (s) + H^+ \longrightarrow Fe^{++} + H_2S(g)$$

Nitric acid and Conc. Sulphuric acid are not used for the preparation of H_2S in laboratory.

They are strong oxidizing reagent (or oxidant)

$$3\ FeS + dil8\ HNO_3 \longrightarrow 3Fe(NO_3)_2 + 4H_2O + 2NO + 3S\downarrow$$

$$3\ FeS + conc.\ 4HNO_3 \longrightarrow Fe(NO_3)_2 + 2H_2O + 2NO_2 + S\downarrow$$

$$3\ FeS + conc\ 2H_2SO_4 \longrightarrow FeSO_4 + 2H_2O + SO_2 + S\downarrow$$

Procedure

1. Take a two necked Woulfe's bottle, add some pieces of ferrous sulphide in it, fit the delivery tube and thistle funnel as shown in figure 18(a) and check air tightness of the apparatus
2. Pour down dil. H_2SO_4 from the thistle funnel, such that the lower end of the funnel dips under the dilute acid, reaction occurs with evolution of H_2S gas, which is collected in gas jar by upward displacement of air.
3. Perform the following experiments to study the properties of the H_2S gas.

Observation Table

Experiment	Observation	Inference
1. **Colour and odour test:** Note colour and odour of the gas	No colour but smell similar to rotten eggs.	1. H_2S is colourless gas having smell similar to rotten eggs.
2. **Solubility test:** Invert a gas jar filled with H_2S over water taken in water trough and shake.	2. The water level rises up slightly inside the gas jar.	2. The gas is slightly soluble in water.
3. **Litmus paper test:** Introduce moist red and blue litmus paper into a gas jar filled with H_2S gas.	3. The blue litmus paper turns faintly red but no any change in red litmus paper.	3. The gas is slightly acidic in nature.
4. **Combustibility test:** Introduce a burning match stick or candle into the gas jar containing H_2S gas.	4. The match stick or candle extinguishes but the gas burns with yellow flame.	4. The gas is non-supporter of combustion but itself combustible in nature.
5. **Lead acetate paper test:** Introduce a moist lead acetate paper into the gas jar filled with H_2S.	5. The paper turns into black.	5. Due to formation of black precipitate of lead sulphide.

6. Pass H_2S gas into a test tube containing acidified solution of $KMnO_4$	6. Pink colour of $KMnO_4$ solution discharges and some yellowish white precipitate formed.	6. H_2S gas reduces $KMnO_4$ (pink) into colourless $MnSO_4$ and itself oxidises to free sulphur.
7. Pass H_2S gas into a test tube containing acidified solution of $K_2Cr_2O_7$.	7. Potassium dischromate changes from orange colour to green and pale yellow precipitate appears.	7. H_2S gas reduces $K_2Cr_2O_7$ (orange yellow) into green coloured $Cr_2(SO_4)_3$ and itself oxidises to free sulphur.
8. Pass H_2S gas into a test tube containing acidified solution of ferric chloride ($FeCl_3$+dil. HCl)	Pale yellow precipitate appears.	8. H_2S reduces $FeCl_3$ to $FeCl_2$.

Analytical use of H_2S gas

Experiment	Observation	Inference
9. Pass H_2S into test tube containing $CuSO_4$ solution acidified with dil. HCl	9. Black precipitate forms	9. Due to formation of black precipitate of cupric sulphide.

10. Pass H_2S gas into test tube containing $CuSO_4$ solution alkalined with NH_4OH	10. Black precipitate forms	10. Due to formation of cupric sulphide
11. Pass H_2S gas into test tube containing $NiSO_4$ acidified with dil. HCl	11. No precipitate	11. No reaction in acidic medium.
12. Pass H_2S gas into a test tube containing $NiSO_4$ alkalined with NH_4OH solution	12. Black precipitate forms	12. Due to formation of nickel sulphide.
13. Pass H_2S gas into a test tube containing $ZnSO_4$ acidified with dil. HCl	13. No precipitate	13. No reaction in acidic medium.
14. Pass H_2S gas into a test tube containing $ZnSO_4$ alkalined with NH_4OH solution	14. White precipitate forms	14. Due to formation of zinc sulphide

Reactions involved

1. Combustibility reaction of H_2S

$$\underset{\text{Limited}}{2H_2S + O_2} \xrightarrow{\text{burning}} 2H_2O + 2S$$

$$\underset{\text{Excess}}{2H_2S + 3O_2} \xrightarrow{\text{burning}} 2H_2O + SO_2$$

2. **Reaction with lead acetate**

$(CH_3COO)_2Pb + H_2S \longrightarrow PbS\downarrow + 2CH_3COOH$

Lead acetate — Lead sulphide (black ppt)

3. **Reducing reaction of H_2S gas**

a. **With acidified solution of potassium permanganate**

$2KMn^{+7}O_4+3H_2SO_4+5H_2S \rightarrow K_2SO_4+2MnSO_4+8H_2O+5S^\circ\downarrow$

pink — manganese sulphate (colourless)

b. **With acidified solution of potassium dichromate**

$K_2Cr_2O_7+4H_2SO_4+3H_2S \longrightarrow K_2SO_4+Cr_2^{+3}(SO_4)_3+7H_2O+3S^\circ$

Orange red — Chromium sulphate Green colour

c. **With acidified solution of ferric chloride**

$FeCl_3+H_2S \xrightarrow{dil.HCl} FeCl_2+2HCl+S^\circ$

Ferric chloride — Ferrous chloride

Reddish brown — Pale yellow

4. **As an analytical regent (or qualitative reagent)**

H_2S gas is used as qualitative reagent for the identification of basic radicals by wet method. For example:

The sulphide of Cu, Pb, Hg, Cd, Bi, As, Sb and Sn are precipitated in the presence of dil. HCl, so they are included in IInd group on the basis of formation of preeipitate.

The sulphides of Zn, Co, Ni, and Mn are precipitated in the presence of NH_4Cl, NH_4OH (excess), so they are included in group IV of qualitative analysis.

$$Cu^{++} + H_2S \xrightarrow[\text{Dil.HCl}]{\text{Acidic medium}} \underset{\text{Black ppt}}{CuS\downarrow} + 2H^+$$

$$Cu^{++} + H_2S \xrightarrow[NH_4OH]{\text{Basic medium}} \underset{\text{Black ppt}}{CuS\downarrow} + 2H^+$$

$$Ni^{++} + H_2S \xrightarrow[\text{Dil.HCl}]{\text{Acidic medium}} \text{no precipitate}$$

$$Ni^{++} + H_2S \xrightarrow[NH_4OH]{\text{Basic medium}} \underset{\text{Black ppt}}{NiS\downarrow} + 2H^+$$

$$Zn^{++} + H_2S \xrightarrow[NH_4OH]{\text{Alkaline medium}} \underset{\substack{\text{Zinc sulphide} \\ \text{White precipitate}}}{ZnS\downarrow} + 2H^+$$

Result:

Hydrogen sulphide gas is prepared in the laboratory and its properties are tested.

Conclusion

Thus, hydrogen sulphide gas can be prepared by the reaction of ferrous sulphide with dil. sulphuric acid and investigated its different properties.

Preparation of H_2S gas by Kipp's apparatus

Hydrogen sulphide gas is required in the laboratory frequently, but intermittently for the precipitation of salts. For this special apparatus is used, which is known as Kipp's apparatus. It consists of two parts (a) bulb A with long stem (b) base with two communicating bulbs B and C like shown in the figure 14.

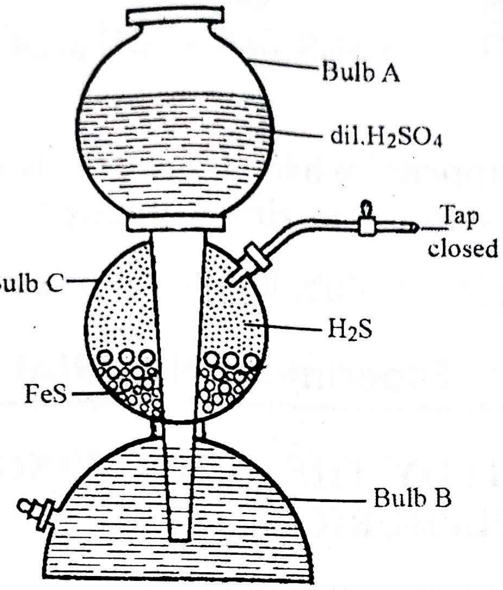


Fig. 14 : Kipp's apparatus for H_2S gas

Pieces of iron sulphide are introduced into the central bulb C and dil. sulphuric acid is poured down into the upper bulb A till the pieces of ferrous sulphide (FeS) just covered by the acid with the tap open. The acid combines with ferrous sulphide producing H_2S gas.

When the tap T is closed, the gas evolves for sometime and very soon the pressure developed in the central bulb forces the acid up into the upper bulb A, as a result, the contact between the acid and FeS breaks and the gas stops to evolve until the tap T is opened again.

Questions

1. **Can we use conc. H_2SO_4 in the preparation of H_2S gas?**

Ans: No, because it oxidises H_2S to sulphur

$H_2S^{--} + \text{conc. } H_2S^{+6}O_4 \longrightarrow 2H_2O + S^{+4}O_2 + S\downarrow$

2. **How do you test H_2S gas in lab?**

Ans: With lead acetate paper, it turns into black colour due to formation of lead sulphide (PbS).

$$(CH_3COO)_2Pb + H_2S \longrightarrow 2CH_3COOH + PbS\downarrow$$

Lead acetate Black ppt

3. What happens when H_2S gas is passed through acidified solution of zinc sulphate?

Ans: No precipitate of ZnS, because it dissolves in dil. HCl.

Experiment No. 19(A)

Object: TO STUDY THE FOLLOWING PROPERTIES OF SULPHURIC ACID

a. **Solubility with water**
b. **litmus paper test (acid test)**
c. **Precipitating reaction**
d. **Dehydrating reaction**

Apparatus required:

i.	Test tube	ii.	Test tube stand
iii.	Washing bottle	iv.	Brush

Chemicals required:

i.	H_2SO_4	ii.	$BaCl_2$ Solution
iii.	Copper turnings	iv.	Oxalic acid
v.	Phenolphthalein	vi.	Litmus paper

Theory:

a. **Sulphuric acid as an acid**

Sulphuric acid has two replaceable hydrogen atoms, so it is diprotic acid. The acidic properties of sulphuric acid can be easily tested by indicator such as litmus paper, methyl orange,

chemically with hydroxide, oxide, carbonate, sulphite, etc.

$$H_2SO_4 \xrightarrow{} 2H^+ + SO_4^{--}$$
(aq.)

$\Rightarrow$ $NaOH + H_2SO_4 \longrightarrow NaHSO_4 + H_2O$

$\Rightarrow$ $NaOH + NaHSO_4 \longrightarrow Na_2SO_4 + H_2O$

Sod. hydrogen sulphate — Sod. sulphate

$\Rightarrow$ $CuO + \text{dil. } H_2SO_4 \longrightarrow CuSO_4 + H_2O$

Base — acid — Salt

$\Rightarrow$ $Na_2CO_3 + \text{dil. } H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + CO_2\uparrow$

b. **Sulphuric acid as an oxidising agent**

Conc. $H_2SO_4 \longrightarrow H_2O + SO_2 + [O]$

$Cu^0 + \text{conc. } H_2SO_4 \longrightarrow CuSO_4 + H_2O + SO_2$

Here, Conc. H_2SO_4 oxidises cupper metal into copper sulphate. So it is oxidising agent

c. **Sulphuric acid as dehydrating reagent**

Those chemical substances, which absorb moisture are known as dehydrating reagent.

For example:

$$C_{12}H_{22}O_{11} \xrightarrow[\Delta]{\text{Conc. } H_2SO_4} 12C + 11H_2O$$

Sucrose (Table sugar) — Black mass

Observation Table

Experiment	Observation	Inference
1. **Solubility Test:** Take about 1 mL of Conc. H_2SO_4 slowly in test tube containing 2-5 mL of distilled water.	1. Acid dissolves in water and test tube becomes warm.	1. Sulphuric acid is soluble in water and exothermic in nature.

2. Test with indicators: **a. With litmus paper :** Test dilute solution of H_2SO_4 with moist blue and red litmus paper.	2. a. Blue litmus paper turns into red but no any change in red litmus paper.	2. a. Sulphuric acid is acidic in nature.
b. Test with methyl orange : Test dilute solution of H_2SO_4 with 1-2 drop of methyl orange	b. The solution turns into pink.	b. It is acidic in nature.
c. With phenolphthalein : To a little NaOH solution coloured with a drop of phenolphthalein (pink colour), add dil. H_2SO_4 acid dropwise	c. Pink colour of phenolphthalein disappears	c. Due to acidic in nature.
3. As an acid: a. With cupric oxide (black oxide) To a little black oxide of CuO in a test tube add some dilute H_2SO_4 and warm the solution.	a. The black oxide (CuO) dissolves and a blue solution is formed.	a. Due to formation of cupric sulphate

b. **With magnesium metal:** To a small piece of Mg in test tube, add dilute sulphuric acid	b. Effervescence with evolution of hydrogen gas	b. Magnesium metal displaces hydrogen gas from H_2SO_4
c. **With sodium carbonate** Take some amount of sodium carbonate in test tube and add dil. H_2SO_4 solution.	c. Effervescence of colourless gas appears which turns colourless limewater into milky colour.	c. Sodium carbonate is decomposed by dil. H_2SO_4 and CO_2 gas is produced.
d. **With the sodium sulphite:** Take some amount of Na in test tube and add dil. H_2SO_4 solution	d. Effervescence of colourless gas with smell similar to burning sulphur	d. Sodium sulphite is decomposed by dil. H_2SO_4.
e. **With copper turning:** Take some copper turning in test tube and add dil. H_2SO_4 acid	e. No reaction	e. Copper cannot displace H_2 from dil.H_2SO_4 because it lies below the hydrogen in electrochemical series.
f. **with potassium iodide:** Take 1-2 ml of KI solution in test tube and add 1-2 mL of dil. H_2SO_4	f. No brown colour of iodine	f. Dil. H_2SO_4 do not liberate iodine from iodide salts.

Reactions involved

a. **With cupric oxide (black powder)**

$$\underset{\text{Black powder}}{CuO} + \text{dil. } H_2SO_4 \xrightarrow{\text{Warm}} \underset{\text{Blue colour}}{CuSO_4} + H_2O$$

b. **With magnesium metal**

$$Mg + \text{dil. } H_2SO_4 \longrightarrow MgSO_4 + H_2$$

c. **With sodium carbonate (Na_2CO_3)**

$$Na_2CO_3 + \text{dil. } H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + CO_2\uparrow$$

$$CO_2 + \underset{\substack{\text{Lime water}\\ \text{Colourless}}}{Ca(OH)_2} \longrightarrow \underset{\text{milky colour}}{CaCO_3\downarrow} + H_2O$$

d. **With sodium sulphite (Na_2SO_3)**

$$Na_2SO_3 + \text{dil. } H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + SO_2$$

e. **With copper turnings**

$$Cu + \text{dil. } H_2SO_4 \longrightarrow \text{No reaction}$$

f. **With potassium iodide solution (KI)**

$$KI + \text{dil. } H_2SO_4 \longrightarrow \text{No reaction}$$

Experiment	Observation	Inference
4. As precipitating reagent : **a. With $BaCl_2$ solution:** Take about 1-2 mL of $BaCl_2$ solution in test tube and add 1-2 drops of dil. H_2SO_4.	a. White precipitates appears, which is insoluble in dil. HCl	a. Due to formation of insoluble barium sulphate

b. With lead nitrate solution Take about 1-2 mL of $Pb(NO_3)_2$ solution in test tube and add dil. H_2SO_4 drop wise	b. White precipitated appears	b. Due to formation of lead sulphate ($PbSO_4$)

Reactions involved

a. **With $BaCl_2$ solution:**

$$BaCl_2 + \text{dil. } H_2SO_4 \xrightarrow{\text{Warm}} \underset{\text{White ppt}}{BaSO_4\downarrow} + 2HCl$$

b. **With lead nitrate solution:**

$$Pb(NO_3)_2 + \text{dil. } H_2SO_4 \longrightarrow PbSO_4\downarrow + 2HNO_3$$

Experiment	Observation	Inference
5. As oxidising agent: **a. With copper turnings:** Take pieces of copper in test tube and add 1-2 ml of conc. H_2SO_4 and heat carefully	a. Colourless suffocating gas evolves, which turns acidified solution of $K_2Cr_2O_7$ into green.	a. Due to formation of sulphur dioxide gas
b. With KI solution: Take about 1–2 mL of KI solution in test tube and add few drops of conc. H_2SO_4	b. Reddish brown coloured iodine, which turns starch solution blue.	b. Due to liberation of I_2 from potassium iodide.

Reactions involved

a. **With copper turnings**

$$Cu^0 + \text{conc. } 2H_2SO_4 \xrightarrow{\Delta} CuSO_4 + 2H_2O + SO_2\uparrow$$

b. **With potassium iodide solution**

$$2KI + \text{Conc. } H_2SO_4 \xrightarrow{\Delta} K_2SO_4 + 5HI$$

$$2HI + H_2SO_4 \xrightarrow{\Delta} SO_2 + 2H_2O + I_2$$

$$\text{Starch} + I_2 \longrightarrow \text{Starch iodide blue complex}$$

Experiment	Observation	Inference
6. As dehydrating reagent **a. Test with sugar:** Take about 5 gram of sugar in porcelain basin and add 1-2 mL of Conc. H_2SO_4	a. Sugar turns black due to charring	a. conc. H_2SO_4 abstracts water from sugar to give black mass
b. With filter paper: Put 1-2 drops of conc. H_2SO_4 on filter paper.	b. The filter paper gets charred.	b. conc. H_2SO_4 abstracts water from cellulose.

Reactions involved

a. **With sugar**

$$\underset{\text{Table sugar}}{C_{12}H_{22}O_{11}} + \text{conc. } H_2SO_4 \xrightarrow{\Delta} \underset{\text{black mass}}{12C} + 11H_2O$$

b. **With cellulose**

$$(C_6H_{10}O_5)n \xrightarrow[\Delta]{\text{Conc. } H_2SO_4} nC + 5nH_2O$$

Cellulose

C. **With sodium chloride (common salt)**

$$NaCl + \text{conc. } H_2SO_4 \xrightarrow{\Delta} NaHSO_4 + HCl\uparrow$$

$$AgNO_3 + HCl \longrightarrow \underset{\text{White ppt}}{AgCl\downarrow} + HNO_3$$

Result:

Properties of sulphuric acid are investigated.

Conclusion

Thus, acidic, oxidising and dehydrating properties of sulphuric acid can be performed.

Precaution

All experiments should be handled carefully.

Experiment No. 19(B)

Object: **TO STUDY THE PROPERTIES OF NITRIC ACID**

Apparatus required:

i. Test tube
ii. Test tube stand
iii. Test tube holder

Chemicals required:

i. Nitric acid
ii. Cu turnings
iii. KI solution

Theory:

a. **Nitric acid as an acid**

Nitric acid has one replaceable hydrogen, so it is monoprotic or monobasic acid. It reacts with oxide, hydroxide, carbonates, bicarbonates, etc.

$$HNO_3 + NaOH \longrightarrow NaNO_3 + H_2O$$

$$2HNO_3 + CuO \longrightarrow Cu(NO_3)_2 + H_2O$$

$$2HNO + CaCO_3 \longrightarrow Ca(NO_3)_2 + H_2O + CO_2\uparrow$$

b. **Nitric acid as an oxidising agent**

$$Cu^0 + HNO_3(1:1) \longrightarrow Cu(NO_3)_2 + H_2O + NO$$

Here Cu reduces HNO_3 to nitric oxide, so it a reducing agent and HNO_3 oxidises copper to cupric nitrate, so it is oxidising agent.

c. **Action of nitric acid on metals**

Generally nitric acid does not produce hydrogen with metal (except Mg and Mn)

Dilute nitric acid favours the formation of colourless nitric oxide (NO) and conc. HNO_3 produces formation of brown nitrogen dioxide (NO_2).

Observation Table

A. **Reactions of dilute nitric acid**

Experiment	Observation	Inference
1. **With phenolphthalein :** To a little NaOH solution in test tube add I drop phenolphthalein and then add dilute nitric acid.	1. The pink colour of the solution becomes colourless,	1. Due to acidic nature and formation of sodium nitrate.

2. **With black oxide of copper oxide:** To a little black oxide of cupric oxide taken in a test tube, add few mL of dil. HNO_3 and warm.	2. The black oxide of CuO dissolves and a blue solution is formed.	2. Due to formation of cupric nitrate.
3. **With limestone:** To a little $CaCO_3$ taken in test tube, add few cc of dil. HNO_3 acid.	3. Effervescence of (evolution of) CO_2 gas is observed.	3. Due to formation of CO_2 gas
4. **With potassium iodide solution:** To I mL of KI solution in test tube, add 1 mL of dil. HNO_3 acid	4. Brown colour of iodine is observed which is further test with a drop of starch solution.	4. Due to formation iodine from iodide salt.
5. **With sodium sulphite (Na_2SO_3)** To a little Na_2SO_3 taken in test tube add few cc. of dil. HNO_3 acid.	5. Evolution of SO_2 gas with smell of burning sulphur, test with acidified solution of $K_2Cr_2O_7$ solution.	6. Due to formation of SO_2 gas.
6. **With magnesium metal:** To a small piece of Mg add very dil. HNO_3 (~2%)	6. Colourless and odourless gas is evolved.	6. Due to formation of hydrogen gas.
7. With copper turnings	7. Brown colour gas is evolved.	7. Due to formation of nitric oxide (NO).

B. With conc. Nitric acid

Experiment	Observation	Inference
1. **With copper turnings:** To a small piece of copper taken in test tube add 1-2 mL of conc. HNO_3 and warm.	1. Brown fumes evolved.	Due to formation of nitrogen dioxide gas (NO_2)
2. **With $FeSO_4$ and NH_4OH** Take 5 drops of aq. $FeSO_4$ solution in a test tube, add 5 drops of conc. HNO_3 and warm then add 1–2 mL of NH_4OH solution.	2. Brown precipitate appears.	2. Due to formation of ferric hydroxide [Fe^{++}ion converted into Fe^{++} ferric ion.
3. **With potassium iodide solution (KI)** To 1-2 mL of KI solution, in test tube, add 1-2 drops of conc. HNO_3.	3. Brown colour of iodine is observed which is further tested with starch solution.	3. Due to formation of iodine from iodide salts.

Reactions with dilute nitric acid solution

i. $NaOH + dil.HNO_3 \longrightarrow NaNO_3 + H_2O$

ii. $\underset{\text{Cupric oxide (black)}}{CuO} + dil.\ HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O$

iii. $\underset{\text{Limestone}}{CaCO_3} + dil.\ 2HNO_3 \longrightarrow Ca(NO_3)_2 + H_2O + CO_2\uparrow$

iv. $2KI + 4HNO_3 \longrightarrow 2KNO_3 + 2NO + I_2 + 2H_2O$

v. $Na_2SO_3 + 2HNO_3 \longrightarrow 2NaNO_3 + SO_2 + H_2O$

vi. $Mg + \text{dil. } HNO_3 \longrightarrow Mg(NO_3)_2 + H_2$

vii $Cu + HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O + NO$

$2HNO_3 \longrightarrow H_2O + 2NO + 3[O]$

$[Cu + [O] \longrightarrow CuO] \times 3$

$CuO + 2HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O] \times 3$

$3Cu + 8HNO_3 \longrightarrow 3Cu(NO_3)_2 + 4H_2O + 2NO$

Reactions with conc. HNO_3

1. $Cu + HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O + NO_2$

$2HNO_3 \longrightarrow H_2O + 2NO_2 + [O]$

$Cu + [O] \longrightarrow CuO$

$CuO + 2HNO_3 \longrightarrow Cu(NO_3)_2 + H_2O]$

$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 4H_2O + 2NO_2$

ii. $2Fe^{++} + 4H^{+} + 2NO^{-}_3 \longrightarrow Fe^{+++} + 2NO_2 + 2H_2O$

iii. $2KI + 4HNO_3 \longrightarrow 2KNO_3 + NO_2 + 2H_2O + I_2$

Result:

Properties of nitric acid are investigated.

Conclusion:

Thus, different properties of dilute and conc. Nitric acid can be studied.

✦✦

Chapter–six

E. Experiments on qualitative analysis

Salt Analysis

I. Introduction

In any branch of physical science, knowledge grows mainly through experiment, observation and analysis which leads to some definite conclusions.

Chemical analysis deals with the determination of constituents and the proportion of Constituents of a substance.

Analytical chemistry is such a branch of chemistry which deals with chemical composition, structure and behaviour of matter.

Analytical chemistry can broadly be divided into two parts:

a. Qualitative analysis

b. Quantitative analysis

a. Qualitative analysis

The process of identification of the various elements or radicals present in a particular chemical substance by physical and chemical test is known as qualitative analysis.

For example: Salt analysis

b. Quantitative analysis

It deals with the estimation of the relative proportion by weight of the elements, radicals or compounds. Simply it determines the proportion of constituents. This process of estimation of constituents in sample by weight is known as quantitative analysis. For example: Volumetric analysis.

The purpose of this book is to make students familiar with the elementary procedures of qualitative analysis and quantitative analysis. Every student should remember that there are two methods for experimental procedures in qualitative analysis.

A. Dry Tests

These tests are performed directly with the sample in dry and solid state.

B. Wet Tests

These tests are performed with the sample in the form of solution with some suitable solvent (e.g. water, dilute HCl)

Every simple salt consists of two parts viz. a positive part or basic radical (for example Na^+, Ca^{++}, Al^{+++}, Si^{++++}) and negative part or acid radical (for example: Cl^- ,CO_3^{--},SO_4^{--} ,NO_3^-, etc.). So, the systematic salt analysis should be performed in the following order.

1. Preliminary dry tests for basic radicals.
2. Preliminary dry tests for acid radicals
3. Wet tests for basic radicals, followed by confirmation with individual tests.
4. Wet test for acid radicals.

Having the preliminary idea about the nature of the salt by a dry way, it is easier to perform wet tests to get fruitful result.

II. Dry Tests for Basic Radicals

Some important explanations of dry test for basic radicals:

1. Action of heat on dry salts in dry test tube

At first test tube is dried with the help of clamp or paper mouth facing downward with constant rotating.

Fusion:

It is process, by which a solid chemical substance in the dry test tube is converted into the liquid or molten state on heating. For example, potassium or sodium nitrate, sodium thiosulphate, etc.

Decripitation

Some crystalline salts with no water of crystallisation, on heating produces cracking sound which is known as decripitation. For example: NaCl.

Chlorides

Certain chlorides decompose on heating with the evolution of chlorine gas. For example,

$$2FeCl_3 \xrightarrow{\Delta} 2FeCl_2 + Cl_2\uparrow$$

$$\underset{\text{Cupric chloride}}{2CuCl_2} \xrightarrow{\Delta} \underset{\text{Cuprous chloride}}{Cu_2Cl_2} + Cl_2\uparrow$$

Sulphates

Certain sulphates of heavy metals evolves SO_2 and SO_3 gas on heating strongly, leaving behind oxides. For example,

$$\underset{\text{Ferrous sulphate}}{2FeSO_4} \xrightarrow{\Delta} \underset{\text{Ferric oxide}}{Fe_2O_3} + SO_2 + SO_3$$

Nitrates

Certain nitrates of heavy metals decompose on heating into metallic oxides, brown nitrogen peroxide gas and oxygen (colourless)

$$2Cu(NO_3)_2 \xrightarrow{\Delta} 2CuO + 4NO_2\uparrow + O_2\uparrow$$

Carbonates

Certain carbonates of the alkali earth metals (except Na_2CO_3, K_2CO_3) decompose on heating like

$$CaCO_3 \xrightarrow{\Delta} CaO + CO_2\uparrow$$

$$MgCO_3 \xrightarrow{\Delta} MgO + CO_2\uparrow$$

Ammonium salts

Most of the ammonium salts, when heated with any alkali, we get ammonia gas,

$$2NH_4Cl + Na_2CO_3 \xrightarrow{\Delta} 2NaCl + H_2O + 2NH_3\uparrow + CO_2\uparrow$$

Mercury salts

They decompose into metallic mercury with sodium carbonate.

$$2HgCl_2 + 2Na_2CO_3 \xrightarrow{\Delta} 2HgCO_3 + 2NaCl$$

$$HgCO_3 \xrightarrow{\Delta} HgO + CO_2\uparrow$$

$$\underset{\text{Mercuric oxide}}{2HgO} \xrightarrow{\Delta} 2Hg + O_2\uparrow$$

2. Change in colour of crystalline compounds due to dehydration (removal of water)

Water of crystallization is an integral part of the constituents of hydrated crystalline solid due to which the crystalline solid possesses particular colour and shape. When hydrated crystalline solid is heated, it becomes anhydrous (no water) and its colour and structure changes like

i) $\underset{\text{Blue vitriol (crystalline)}}{CuSO_4 \cdot 5H_2O} \xrightarrow{\Delta} \underset{\text{white (amorphous)}}{CuSO_4 + 5H_2O}$

ii) $FeSO_4 \cdot 7H_2O \xrightarrow{\Delta} FeSO_4 + 7H_2O$

Green vitriol — white

iii) $CoCl_2 \cdot 6H_2O \xrightarrow{120°C} CoCl_2 + 6H_2O$

Cobalt chloride Pink — blue (anhydrous)

3. Cobalt Nitrate Test (Filter ash test)

Suitable for infusible (not melt) and white coloured salt

Salts like Zn, Al and Mg are dissolved in suitable solvent (eg. dil. HCl), and 2–3 drops of cobalt nitrate solution is added in test tube, insert a piece of filter paper and burnt to ashes. The oxides thus formed combine with the cobalt oxide to give characteristic coloured compounds like

$$Co(NO_3)_2 \xrightarrow{\Delta} CoO + NO_2\uparrow + O_2\uparrow$$

$$ZnCO_3 \xrightarrow{\Delta} ZnO + CO_2\uparrow$$

$$ZnO + CoO \xrightarrow{\Delta} CoZnO_2$$

Cobalt zincate (green)

$$Al_2O_3 + CoO \xrightarrow{\Delta} Co(AlO_2)_2$$

Cobalt metaluminate (blue)

$$MgO + CoO \longrightarrow CoMgO_2$$

Cobalt magnesate (light pink)

$$BaO + CoO \longrightarrow CoBaO_2$$

grey colour

4. Borax Bead (for coloured salts)

Molecular formula of Borax powder is $Na_2B_4O_7.10H_2O$ (sodium pyroborate decahydrate).

When borax powder is heated, it swells up in platinum loop, fuses into clear transparent glassy bead containing sodium metaborate and boric anhydride. The metallic oxide formed from the sample (given salt) combines with boric anhydride giving characteristic coloured beads. For example:

$$Na_2B_4O_7.10H_2O \xrightarrow{\Delta} Na_2B_4O_7 + 10H_2O$$
Borax powder

$$Na_2B_4O_7 \xrightarrow{\Delta} NaBO_2 + B_2O_3$$
Sod. metaborate (Boric anhydride)

$$CuSO_4 \longrightarrow CuO + SO_3$$

$$CuO + B_2O_3 \longrightarrow Cu(BO_2)_2$$
Cupric metaborate

In the reducing flame, the cupric metaborate is reduced to colouress cuprous metaborate and finally to metallic copper

$$2Cu(BO_2)_2 + C \longrightarrow 2CuBO_2 + B_2O_3 + CO\uparrow$$
Colourless

$$2CuBO_2 + C \longrightarrow 2Cu + B_2O_3 + CO\uparrow$$

Table for borax bead test

Experiment	**Observation**		**Inference**
	In reducing flame	**In Oxidizing flame**	
Borax bead test	1. Green when hot and blue when cold.	Opaque red	Cu salt
	2. Reddish yellow when hot, pale yellow when cold	Very light green	Fe salt
	3. Yellow brown	Opaque grey	Ni salt
	4. Deep violet	Colourless	Mn salt
	5. Deep green	Deep green	Cr salt
	6. Deep blue	Deep blue	Co salt

5. Flame Test (for white coloured salts)

The object of moistening the salts with Conc. Hydrochloric acid in platinum wire is simply to convert the salts into the chlorides, which are most volatile and are easily vapourised. At high temperature, imparting (produces) different colours characteristics to the metallic basic radicals to the flame. Thus the identification of the metallic atoms from their characteristic flame colours is the basis of spectrum analysis. For example: Sodium produces golden yellow to the flame.

The use of blue glass in flame test is useful for distinction between sodium and potassium, calcium and strontium. For example, when flame of sodium and potassium metal is viewed through the blue glass, only the pink colour due to potassium will be visible, the colour of sodium being completely absorbed by the glass.

Experiment No. 20

Object: **TO DETECT BASIC RADICAL PRESENT IN GIVEN INORGANIC SALT BY DRY TEST**

Sample number: B_1, B_2, B_3...

Physical characters:

Sometimes an inorganic salt (sample) is known by the physical properties, so before carrying out any particular experiment, note down the colour, odour and state like solid (crystalline or amorphous)

State: Solid
- Crystalline or granular e.g. NaCl
- Amorphous or powder form e.g. $CaCO_3$

Colour :

Theory:

Salt: The chemical substance (compound), which is made up of positive and negative ions or radicals is known as salt. It is a substance formed other than water in neutralization process.

For example: NaCl, KNO_3 $CaCO_3$ KNO_2 etc.

Radicals: Atoms or group of atoms, which carry positive or negative charge and behave as a single unit during a chemical reaction.

For example: NH_4^+,SO_3^{--},PO_4^{---},NO_3^-,NO_2^-, etc.

Type of radicals:

(i) **Simple radicals (ions):** Single atom containing positive or negative charge is known as simple radical or ion.

For example: Na^+, Mg^{++}, Al^{+++}, Si^{++++} etc.

They are also known as cations.

Cl^-, Br^-,O^{--} N^{---} etc. They are known as anions.

(ii) **Compound radicals:** Group of atoms containing positive or negative charge and behave as a single unit during a chemical reaction are known as compound radicals.

For example:

Ammonium $\longrightarrow NH_4^+$

Nitrate $\longrightarrow NO_3^-$

Sulphate $\longrightarrow SO_4^{--}$

Phosphate $\longrightarrow PO_4^{---}$

Nitrite $\longrightarrow NO_2^-$

Sulphite $\longrightarrow SO_3^{--}$

Phosphite $\longrightarrow PO_3^{---}$

Carbonate $\longrightarrow CO_3^{--}$, etc.

(iii) **Electropositive or basic radicals:** Those radicals, which come from base side (or metal side) and have positive charge are known as electropositive radicals. For example:

Na^{+} come from NaOH, Ca^{++} come from $Ca(OH)_2$

Al^{+++} come from $Al(OH)_3$, NH_4^{+} come from NH_4OH

(iv) **Electronegative or acid radicals:** Those radicals, which come from acid side and have negative charge are known as electronegative radicals. For example:

Cl^{-} come from HC1

NO_3^{-} come from HNO_3

NO_2^{-} come from HNO_2 (nitrous acid),

SO_4^{--} come from H_2SO_4

SO_3^{--} come from H_2SO_3 (sulphurous acid)

PO_4^{---} come from H_3PO_4

CO_3^{--} come from H_2CO_3 etc.

Dry Test : The test perform directly with the sample in dry and solid state is called dry test.

Note: Experiment, observation are generally written in past tense and passive voice and inference (conclusion) in present tense.

1. Action of Heat on Dry Salts

Take a little quantity (1 pinch) sample (given salt) into a clean and dry test-tube and heat it first gently and then strongly in horizontal position and out of face to prevent from cracking and accident.

Experiment	Observation	Inference
Take a little (1 pinch) of the sample in a clean and dry test tube and heat it first gently and then strongly.	i. **Moisture** condenses on the upper part of the test tube. ii. The chemical substance **melts** and **solidifies** on cooling. iii. The chemical substance **decrepitates** **Gas evolves** ***A. It is coloured.*** **a. Brown colour:** it turns freshly prepared $FeSO_4$ solution into black. **b. Brown colour:** It does not turn ferrous sulphate solution black but turns starch iodide paper blue. **c. Greenish yellow colour:** It turns starch iodide paper blue and bleaches (decoloursises) moist litmus paper. **d. Violet vapours:** It turns starch paper into blue.	i. Salts containing water of crystallisation e.g.$CuSO_4.5H_2O$ (blue vitrol) ii. Salts of alkali metals e.g. KNO_3, $NaNO_3$. iii. Salts having no water of crystallization e.g. NaCl, KBr, $Pb(NO_3)_2$ etc. a. Nitrogen peroxide $(NO)_2$ gas from certain nitrates NO^-_3 and nitrites (NO^-_2) b. Br_2 gas from certain bromide salts. c. Chlorine gas from certain chlorides e.g. NaCl. d. Iodine from iodide salts.

If a sublimate is formed in the test tube mix one part of dry sample and two parts of Na_2CO_3 and heat strongly.	***B. The gas is colourless but has odour***	
	a. Similar to burning sulphur: It decolourises $KMnO_4$ solution (pink) and turns acidified $K_2Cr_2O_7$ (orange yellow) into green colour.	a. Sulphur dioxide gas evolves from sulphite e.g. $CaSO_3$
	b. Similar to rotten eggs: This gas turns lead acetate paper into black.	b. Hydrogen sulphide (H_2S) gas evolves from sulphide salts e.g: FeS
	c. Similar to ammonia (pungent): It turns moist red litmus paper blue and mercurous nitrate paper into black.	c. Ammonia (NH_3) gas evolves from certain ammonium salts, eg. NH_4Cl
	C. The gas is both colourless and odourless	
	a. It supports the combustion of a burning candle.	a. Oxygen gas evolves from compounds rich in oxygen e.g. KNO_3, $KClO_3$.
	b. It turns lime water milky.	b. CO_2 gas evolves from certain carbonates, e.g $CaCO_3$.

	◆ **If sublimate is white** It produces irritating smell similar to ammonia gas, which turns red litmus paper blue and mercurous nitrate paper black. ◆ **If sublimate is yellow** and turns red on rubbing with a glass rod. ◆ **If sublimate is orange**	◆ Presence of NH_4^+ Salt ◆ May be mercuric iodide (HgI_2) ◆ May be antimony sulphide (Sb_2S_3)

Note:

i) *If a gas evolved is to be tested with a reagent solution, dip a glass tube into the solution, thumb it and show near the mouth of the test tube.*

ii) *If it is to be tested with a piece of paper, soak the piece into the required solution, hold it near the mouth of the test tube.*

Experiment	Observation	Inference
	v. Change in colour due to dehydration on heating a. From original light green to white, when changes to anhydrous (no water).	a. May be $FeSO_4.7H_2O$ and other ferrous salts.

	b. From blue to white (blue colour reappears back with a drop of water)	b. May be blue vitriol ($CuSO_4.5\ H_2O$)
	c. From green to whitish yellow, when anhydrous	c. May be $NiSO_4.\ 7H_2O$ and other nickel salts.
	d. From pink to blue or violet when anhydrous	d. May be cobalt salts
	vi. The residue is coloured due to decomposition on strong heating	
	a. Dark brown or black	a. May bc Fe. Co, Ni, Mn, Cu salt.
	b. Yellow when hot, white on cooling	b. May be Zn salt
	c. Blue to black	c. May be Cu salt
	d. Black when hot reddish on cooling	d. May be Fe salt
	e. Reddish brown, when hot, yellow on cooling	e. May be Pb, Bi salts
	f. Infusible (not melt) white (similar to original) colour on strong heating	f. May be Ca, Sr, Ba, Mg, Al salts
	g. Pink to black colour	g. May be Co salt
	h. Yellow or green to ash colour,	h. May be Cr salt
	i. Colourless to brown when cold	i. May be Ca salt

2. **Charcoal Reduction (Now a days generally not performed)**

Take one part given sample and two parts of sodium carbonate in the cavity of charcoal, moisten it with a drop of water and press before heating with reducing blow-pipe flame or luminous zone (or reducing flame).

3. **Cobalt Nitrate Test (Only for infusible non-coloured or white salts)**

Take a few cc of original solution of the given salt in water or dil. hydrochloric acid in test tube and add 2-3 drops of cobalt nitrate solution. Soak or moist a piece of filter paper in the resulting solution and burn the paper completely in flame. Note the colour of ash.

Experiment	Observation	Inference
Perform the cobalt nitrate or filter ash test of the sample	**The residue or ash is**	
	a. Green coloured	Zn salt
	b. Blue coloured	Al salt
	c. Light pink coloured	Mg salt
	d. Grey coloured	May be Ca, Ba and Sr salts

Reactions

$$Co(NO_3)_2 \xrightarrow{\Delta} CoO + NO_2\uparrow + O_2\uparrow$$

$$ZnCO_3 \xrightarrow{\Delta} ZnO + CO_2\uparrow$$

$$ZnO + CoO \xrightarrow{\Delta} \underset{\text{Cobalt zincate (green)}}{CoZnO_2}$$

$$Al_2O_3 + CoO \xrightarrow{\Delta} \underset{\text{Cobalt meta-aluminate (blue)}}{Co(AlO_2)_2}$$

$$MgO + CoO \longrightarrow CoMgO_2$$

Cobalt magnesate (light pink)

$$BaO + CoO \longrightarrow BaCoO_2$$

grey colour

4. Borax Bead Test (Only for coloured (salts)

At first a small circular loop at the tip of the platinum wire is prepared and heat the loop in the faint blue (non-luminous or oxidising flame) zone of burner, dip it into the powdered borax. Repeat this process two or three times until a clear and transparent bead is prepared.

Now touch the sample with bead and heat first in the oxidising flame (non-luminous zone) and note the colour of bead. Then dip the bead in the stannous chloride solution and heat it in the reducing flame (luminous zone) of the burner and also note the colour.

Experiment	Observation		Inference
	In oxidising flame (Non-luminous flame)	In reducing flame (Luminous flame)	
Perform the borax bead test for coloured salts	i. Yellow brown	Opaque grey	May be Ni-salt
	ii. Reddish yellow when hot, pale yellow when cold	Light green	May be Fe-salt
	iii. Green when cold	Colourless	May be Cu-salt
	iv. Deep violet	Colourless	May be Mn-salt
	v. Deep green	Deep green	May be Cr-salt
	vi. Deep blue	Deep blue	May be Co-salt

5. Flame Test (Only for white salts or non-coloured)

At first clean platinum wire by repeatedly heating and dipping the tip into conc. HC1 taken in a watch glass till the wire simply glows and produces no colour to the flame. Now dip the tip of the platinum wire in fresh Conc. HC1, touch the given sample with the tip of the wire and keep near the outer-nonluminous flame. Note the colour of the flame first with naked eye and then through the blue glass.

Experiment	**Observation**		**Inference**
	Flame colouration through naked eye	**Flame colouration through blue glass**	
Perform the flame test of non-coloured sample	i. Golden yellow	Colourless	May be Na salt
	ii. Violet or rose colour	Pink	May be K salt
	iii. Orange red	Faint green	May be Ca salt
	iv. Apple green	Same	May be Ba salt
	v. Intense or deep crimson	Same	May be Sr salt
	vi. Bluish white	White	May be Pb, Sb, As and Bi salt

Note:

i) *Do not use same conc. hydrochloric acid for testing two different samples.*

ii) *It is the best test for alkali and alkaline earth metals.*

iii) *If suspect for lead, do not use platinum wire but use asbestos fibre or graphite pencil tip.*

Conclusion:

Thus, the given sample B_1 contains .... radical.

III. Dry Test for Acid Radical

Explanations of dry test for acid radicals

A. Dilute Acid Test for CO_3^{--}, NO_2^{-}, SO_3^{--}, S^{--}

Carbonates (CO_3^{--}): Most of the carbonates react with dil. HCl or H_2SO_4 to give colourless carbon dioxide, which turns lime water (freshly prepared) into milky colour. For example:

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

$$MgCO_3 + 2HCl \longrightarrow MgCl_2 + H_2O + CO_2\uparrow$$

$$\underset{\text{Lime water (colourless)}}{Ca(OH)_2} + CO_2 \longrightarrow \underset{\text{milky colour}}{CaCO_3\downarrow} + H_2O$$

Carbon dioxide gas reacts with sodium carbonate to give sodium bicarbonate, which does not give pink colour with phenolphthalein because sodium bicarbonate is acidic in nature.

$$Na_2CO_3 + CO_2 \longrightarrow 2NaHCO_3$$

Sulphites (SO_3^{--}): Most of the suiphite salts react with dil. HCl or H_2SO_4 to give sulphur dioxide gas, which turns acidified solution of potassium dichromate (orange yellow) into green coloured chromium sulphate compound by reduction reaction.

$$\underset{\text{Sod. sulphite}}{Na_2SO_3} + H_2SO_4 \longrightarrow 2NaCl + H_2O + SO_2\uparrow$$

$$Na_2SO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + H_2O + SO_2\uparrow$$

$$\underset{\text{Orange yellow}}{K_2Cr_2O_7} + \text{dil.}H_2SO_4 + 3SO_2 \rightarrow K_2SO_4 + \underset{\text{green colour}}{Cr_2(SO_4)_3} + H_2O$$

Nitrites (NO_2^-): Nitrites of the metal react with dil. HCl to give colourless fumes of nitric oxide (NO) and brown fumes of nitrogen peroxide (NO_2) which turns freshly prepared ferrous sulphate into dark brown or black due to formation of nitrosyl ferrous sulphate like

$$KNO_2 + HCl \longrightarrow KCl + HNO_2 \text{ (nitrous acid)}$$

$$3HNO_2 \longrightarrow H_2O + HNO_3 + 2NO\uparrow$$

$$2NO + O_2 \longrightarrow \underset{\text{Brown}}{2NO_2\uparrow}$$

$$FeSO_4 + NO \longrightarrow FeSO_4.NO \text{ (Nitrosyl ferrous sulphate)}$$

Dark brown or black coloured (Ring test)

Sulphides (S^{--}) : Sulphides of the metal react with dil. HC1 or dil. H_2SO_4 to give hydrogen sulphide (H_2S) gas, which turns lead acetate to black precipitate due to formation of lead sulphide.

$$FeS + H_2SO_4 \longrightarrow FeSO_4 + H_2S\uparrow$$

$$\underset{\text{Lead acetate}}{(CH_3COO)_2Pb} + H_2S \longrightarrow \underset{\text{acetic acid}}{CH_3COOH} + \underset{\text{lead sulphide (black ppt)}}{PbS\downarrow}$$

B. Conc. sulphuric acid test (for halides)

Hales = sea salt, gens = to produce. For example: NaCl, KI, $MgBr_2$ etc.

Halogens: $X_2 = Cl_2\ Br_2\ I_2$

Halides: $X^- = Cl^-, Br^-, I^-)$

Halogen acid: H X (where X = Cl, Br, I)

Salt containing halides react with conc. sulphuric acid to give fumes of HC1, HBr and HI but HBr and HI being reducing agent reacts with oxidising agent (H_2SO_4) to give Br_2 and I_2.

$$NaC1 + \text{conc. } H_2SO_4 \xrightarrow{\Delta} NaHSO_4 + HC1\uparrow$$

$$NaBr + H_2SO_4 \xrightarrow{\Delta} NaHSO_4 + HBr\uparrow$$

$$2HBr + H_2SO_4 \xrightarrow{\Delta} 2H_2O + SO_2 + Br_2$$

$$KI + H_2SO_4 \xrightarrow{\Delta} KHSO_4 + HI\uparrow$$

$$2HI + H_2SO_4 \xrightarrow{\Delta} 2H_2O + SO_2 + I_2$$

$$8HI + H_2SO_4 \xrightarrow{\Delta} 4H_2O + H_2S + 4I_2$$

All the white fumes of HC1, HBr, and HI give precipitate with silver nitrate ($AgNO_3$) solution like

$$HC1 + AgNO_3\ (aq.) \rightarrow HNO_3 + AgCl\downarrow \text{ (white ppt)}$$

$$HBr + AgNO_3\ (aq.) \rightarrow HNO_3 + AgBr\downarrow \text{ (Pale yellow ppt)}$$

$$HI + AgNO_3\ (aq.) \rightarrow HNO_3 + AgI\downarrow \text{ (deep yellow ppt)}$$

HCl gives dense white fumes of ammonium chloride with liquor ammonia.

$$HCl + NH_3 \longrightarrow \underset{\text{Dense white fumes}}{NH_4Cl}$$

Whether the original salt is a chloride or bromide or iodide (I^-), a pinch of manganese dioxide (MnO_2) is added to the mixture of salt and conc. H_2SO_4 for the liberation of element halogen gases (Cl_2, Br_2, I_2), which have specific colour.

$$2NaX + 3H_2SO_4 + MnO_2 \xrightarrow{\Delta} 2NaHSO_4 + MnSO + 2H_2O + X_2\uparrow$$

$X^- = Cl^-, Br^-, I^-$ $\quad\quad$ $X_2 = Cl_2, Br_2, I_2$

For example:

$$2NaCl + 3H_2SO_4 + MnO_2 \xrightarrow{\Delta} 2NaHSO_4 + MnSO_4 + 2H_2O + Cl_2\uparrow$$

Test with starch iodide paper:

The pieces of filter paper soaked in the mixed solution of starch and potassium iodide is known as starch iodide paper. As we know reactivity of halogens $Cl_2 > Br_2 > I_2$, so chlorine and bromine can liberate iodine, which turns starch blue immediately due to formation of starch iodide (blue).

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

$$Br_2 + KI \longrightarrow 2KBr + I_2$$

$$\text{Starch} + \text{Iodine} \longrightarrow \text{starch iodide (blue)}$$

C. Dry test for nitrate (NO_3^-)

The salt sample containing nitrate is allowed to react with conc. H_2SO_4 and copper turning. On warming, it results blue solution of copper nitrate and brown gas of nitrogen dioxide.

$$NO^-_3 + H_2SO_4 \longrightarrow HSO_4^- + HNO_3$$

$$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 2NO_2\uparrow + 2H_2O$$

D. Dry Test for Sulphate (SO_4^{--})

Silver Coin Test (Hepar Method): The salt (sample) containing sulphate acid radical react with sodium carbonate in charcoal block to give sodium sulphide (Na_2S) which reacts with water to give hydrogen sulphide (H_2S) gas, which causes black stain on silver coin due to formation of silver sulphide (Ag_2S) like

$$ZnSO_4 + Na_2CO_3 \longrightarrow ZnCO_3 + Na_2SO_4$$

$$Na_2SO_4 + 4C \longrightarrow Na_2S + 4CO\uparrow$$

$$2Na_2S + 4H_2O \longrightarrow 4NaOH + 2H_2S\uparrow$$

$$2H_2S + 4Ag + O_2 \longrightarrow \underset{\text{Black stain}}{2Ag_2S\downarrow} + 2H_2O$$

The sodium sulphide formed in the above reaction reacts with freshly prepared sodium nitroprusside solution to give violet colour due to formation of tetrasodium thionitroprusside.

$$Na_2S + \underset{\text{Sod. Nitroprusside}}{Na_2[Fe(CN)_5NO]} \longrightarrow \underset{\text{Violet colour}}{Na_4[Fe(CN)_5NOS]}$$

Note: This test is not performed due to have costly silver coin.

Experiment No. 21

Object: TO DETECT ACID RADICAL PRESENT IN THE GIVEN INORGANIC SALT BY DRY TEST

Sample Number: A_1, A_2 A_3 ....

Physical characters:

State: Crystalline or amorphous

Colour:

Theory

Define: Salts, acids and basic radicals with suitable examples, dry test.

1. **Dilute hydrochloric acid test (CO_3^{--}, NO_2^-, SO_3^{--}, S^{--})**

Experiment	Observation	Inference
Take little sample (1 pinch) in test tube and add 1-2 ml of dilute HCl	**A gas is evolved :** **A. Colourless and odourless gas** evolves with rapid effervescence (coming out bubbling) and turns fresh lime water milky, which discharges the colour of the filter paper soaked in phenolphthalein solution made alkaline with NaOH or Na_2CO_3 solution.	CO_2 gas evolves from carbonate (CO_3^{--}).
	B. Brown coloured gas evolves, which turns freshly prepared $FeSO_4$ solution into black.	NO_2 (Nitrogen dioxide) gas evolves from nitrite (NO_2^-)
	C. Colourless gas with smell similar to burning sulphur evolves, which turns acidified potassium dichromate paper solution into green.	SO_2 evolves from sulphite (SO_3^{--})
	D. Colourless gas with smell similar to rotten eggs evolves, which decolourises acidified solution $KMnO_4$ paper and turns lead acetate paper into shining black.	H_2S gas evolves from sulphide (S^{--})

Lime Water Test for Carbonate (CO_3^{--})

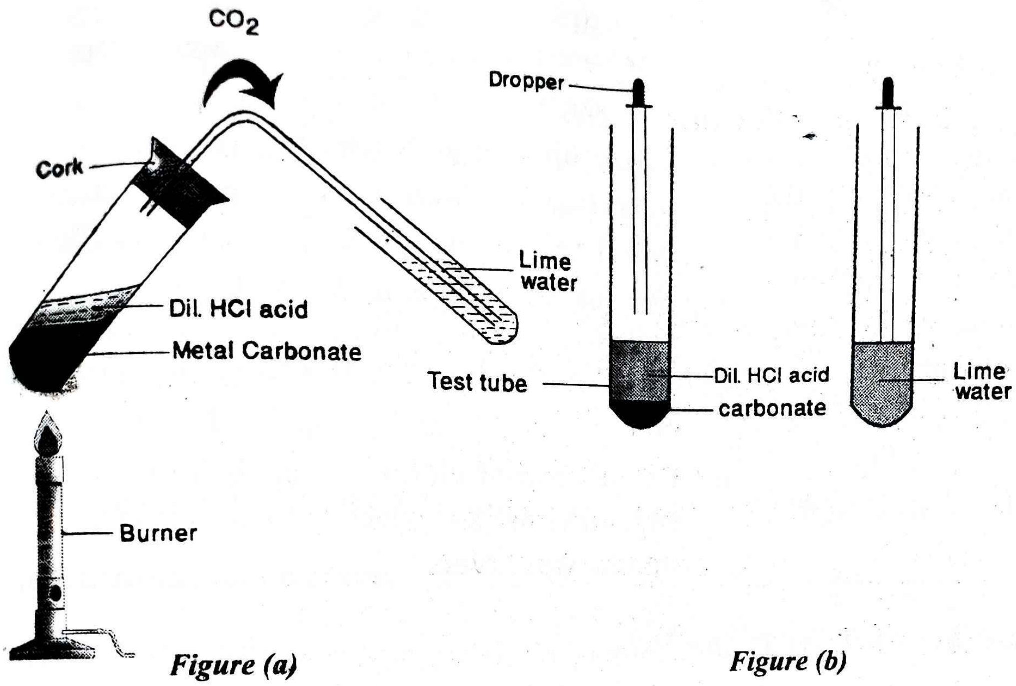


Figure (a) *Figure (b)*

Some carbonate (sample) is taken in test tube and add dilute HC1 acid. There is effervescence of CO_2 gas, which turns lime water (colourless) into milky colour due to formation of calcium carbonate as shown in the figure (a) and (b).

2. Concentrated Sulphuric Acid Test (For halogen)

Experiment	Observation	Inference
Take a little the sample (given salt) a clean test tube and add few drops Conc. H_2SO_4 Warm if needed, not boiled.	a. **Formation of a white dense fumes,** with irritating smell, gives white precipitate with $AgNO_3$ solution more white fumes of NH_4Cl with liquor ammonia (glass rod) solution b. **Formation of copious reddish brown fumes,** which gives pale yellow precipitate with $AgNO_3$ solution	a. HCl gas evolves from chloride (Cl^-) b. Br_2 and HBr evolves from bromide (Br^-)

	c. **Formation of violet vapours,** which gives deep yellow precipitate with $AgNO_3$ solution	c. I_2 and HI gas evolves from iodide (I^-)
If halides are suspected from the above test, add a pinch of MnO_2 to the same test tube and warm.	a. **Formation of reddish brown gas,** which turns starch iodide paper blue. b. **Formation of greenish yellow gas,** which turns starch iodide paper blue. c. **Formation of violet vapours,** which turns starch paper blue.	a. Br_2 gas evolves from bromide (Br^-) b. Cl_2 gas evolves from chloride (Cl^-) c. I_2 from iodide (I^-)

Dry Test for Nitrate (NO_3^-)

Experiment	Observation	Inference
Take a little sample in a clean test tube and add copper turning and Conc. H_2SO_4 and warm.	**Brown fumes** evolves, which turns freshly prepared $FeSO_4$ solution into black	NO_2 gas evolves from nitrate (NO_3^-)

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

Experiment	Observation	Inference
Take a little sample and three times its bulk of Na_2CO_3 in charcoal cavity fused in reducing flame. Keep the fused mass on a silver coin, add one drop of water, press with a pestle and wash.	A black stain is formed	Due to presence of SO_4^{--}

Alternatively

Experiment	Observation	Inference
Insert the above fused mass into the freshly prepared dilute solution of sodium nitroprusside in a test tube and shake.	Formation of violet colouration, which disappears rapidly	Due to presence of SO_4^{--}

IV. Wet Test for Basic Radicals (Zn^{++}, Al^{+++}, $NH4^{+}$, Ca^{++}, Na^{+})

Theoretical Discussion on Wet Tests for Basic Radicals

Common Ion Effect:

The degree of ionisation of weak electrolyte is decreased or suppressed or depressed, when we add another strong electrolyte containing common ion in solution is known as its common ion effect. For example:

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

Acetic acid (weak)

According to Law of Mass Action

$$K_e = \frac{[CH_3COO^-][H^+]}{[CH_3COOH]}$$

If we add strong electrolyte like sodium acetate

$$CH_3COONa \longrightarrow CH_2\,COO^- + Na^+$$

The degree of ionisation of weak electrolyte, acetic acid is suppressed due to common ion CH_3COO^- (acetate ion).

Similarly,

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$

Weak electrolyte

According to law of Mass Action

$$K_c = \frac{[NH_4^+][OH^-]}{[NH_4OH]}$$

When we add strong electrolyte

$$NH_4Cl \longrightarrow NH_4^+ + Cl^-$$

The degree of ionisation of ammonium hydroxide is suppressed, due to common ion NH_4^+ ion.

Solubility Product

Chemical substances such as AgCl, $BaSO_4$, $PbSO_4$ etc. are insoluble in water but in fact there is no any chemical substance, which is completely insoluble in water. Every substance gets dissolved in water even to a very small extent. So, they are called sparingly (slightly) soluble in water.

$$\text{AB solid} \rightleftharpoons \text{AB dissolves} \rightleftharpoons A^+ + B^-$$

For example:

$$\text{AgCl (solid)} \rightleftharpoons \text{AgCl dissolves} \rightleftharpoons Ag^+ + Cl^-$$

Condition for solubility product

i. Solution is saturated and
ii. Temperature is constant

According to Law of Mass Action:

$$K_e = \frac{[Ag^+][Cl^-]}{[AgCl]}$$

Solubility Product

The ionic product of sparingly soluble electrolyte in saturated solution at fixed temperature is constant, is known as its **solubility product.**

Rules Governing Precipitation

i. There is no precipitation in unsaturated solution because its ionic product is less than solubility products and also no precipitation occurs in saturated solution because its ionic product is equal to its solubility product.

ii. Generally in supersaturated solution, where the ionic product exceeds the solubility product, the excess ions are thrown out of solution in the form of insoluble precipitate, until the ionic product value comes down to the solubility product value of that particular chemical substance

iii The lesser the solubility product of a chemical substance, the more easily and completely it gets precipitate

Solubility product of $Fe(OH)_2 = 1 \times 10^{-18}$

Solubility product of $Fe(OH)_3 = 1.1 \times 10^{-36}$

So, it is always better to precipitate iron in the form of $Fe(OH)_3$ (ferric hydroxide)

Applications of Solubility Product Principles in group separation

Group I

Group reagent : dil. HCl

The addition of dilute hydrochloric acid to the soluble salts like Hg^+ (ous), Ag^+ and Pb^{++} (plumbous) exceeds the solubility product of their respective chlorides and hence, these metals are precipitated as insoluble chlorides in group I e.g. Hg_2Cl_2, AgCl, $PbCl_2$.

Group II

Group reagent : dil. HCl and H_2S

Some soluble salts like Cu^{++},Pb^{++},Hg^{++} etc. give precipitate in sulphide form with hydrogen sulphide gas in acidic medium (dil. HC1) because they have low solubility product but sulphide of Zn^{++}, Co^{++}, Ni^{++} and Mn^{++} do not give precipitate with H_2S (because they have high solubility product.

Sulphides Group II	Solubility product	Sulphides Group IIIB	Solubility Product
CuS	8.5×10^{-45}	ZnS	1.0×10^{-20}
HgS	4×10^{-53}	NiS	2.0×10^{-24}
PbS	4.20×10^{-28}	CoS	7.0×10^{-23}
CdS	3.6×10^{-29}	MnS	1.40×10^{-15}

HCl and H_2S ionise as given below:

$$HCl \longrightarrow H^+ + Cl^- \text{ (complete ionisation)}$$

$$H_2S \rightleftharpoons 2H^+ + S^{--} \text{ (partial ionisation)}$$

Both of them give common ion (H^+) in solution, but the complete ionisation of HC1 suppresses the ionisation of the weak electrolyte H_2S and reducing the concentration of the sulphide ions, consequently the S^{--} ions in the solution will be sufficient to exceed the solubility products of group II metal sulphides and give precipitate but ionic product of sulphide of group IIIB metals not exceed to its solubility products, so they do not give precipitate in a acidic medium with H_2S.

Group III A (Al^{+++}, Fe^{+++},Cr^{+++})

Group reagent: NH_4Cl and NH_4OH

$$\text{O.S.} + NH_4Cl + \underset{\text{Excess}}{NH_4OH} = \underset{\text{hydroxide}}{M(OH)_3}$$

Here ammonium chloride is used not as a precipitant but as an agent to suppress the ionisation of the weak electrolyte NH_4OH (base), so that the hydroxyl ions (OH^-) obtained from the latter may be just enough to precipitate only Fe^{+++}, Al^{+++} and Cr^{+++} as insoluble hydroxides but not Zn^{++}, Mn^{++} and Mg^{++} of the subsequent groups.

$$NH_4Cl \longrightarrow NH_4^+ + Cl^- \text{ (complete ionisation)}$$

$$NH_4OH \rightleftharpoons NH_4^+ + OH^- \text{ (partial)}$$

Note: *If NH_4Cl is added insufficiently, there is possibility of Zn, Mn and Mg also being precipitated as hydroxides earlier in the group IIIA causing serious complications. So, the necessity of adding NH_4Cl (solid) prior to NH_4OH*

Group III B (ZnS, MnS, NiS, CoS)

Group reagent: NH_4Cl, NH_4OH and H_2S

$$O.S. + NH_4Cl + NH_4OH + H_2S = \underset{\text{Metal sulphide}}{MS\downarrow}$$

When H_2S gas is passed through alkaline solution, ammonium sulphide is formed, which being a salt, ionizes almost completely to give large concentration of S^{--} ions enough to exceed the solubility products ZnS, MnS, CoS, and NiS to give precipitate.

Group IV ($CaCO_3$, $SrCO_3$, $BaCO_3$)

Group reagent : NH_4Cl, NH_4OH and $(NH_4)_2CO_3$

$$O.S. + NH_4Cl + NH_4OH + (NH_4)_2CO_3 \rightarrow \underset{\text{Metal carbonate}}{MCO_3\downarrow}$$

Strong electrolyte NH_4Cl suppresses the concentration of CO_3^{--} due to common ion effect, so ionic product of $CaCO_3$, $SrCO_3$ and $BaCO_3$ exceeds its solubility product and give precipitate, but ionic product of $MgCO_3$ not exceed to its solubility product so it does not produce precipitate.

A. Classification of basic radicals into groups

For the detection of basic radicals or cations by wet way, they are classified into six groups on the basis of precipitation.

Group reagents : Those chemical substances, which are used to detect basic radicals in particular salts are known as group reagents.

For example,

Group reagent for 1st group is dil. HCl

Group reagent for IInd group is dil. HCl + H_2S

Group	Group reagents	Cations or basic radicals	Formation of perecipitate
I	Dilute HCl acid	Pb^{++}, Hg^{+} (ous), Ag^{+}	As chlorides $(Cl)^{-}$
II	Dil. HCl + H_2S	Cu^{++}, Pb^{++}, Hg^{++}, Bi^{++} Cd^{++}, As^{+++}, Sb^{+++}, Sn^{++}	As sulphide (S^{--})
IIIA	$NH_4Cl + NH_4OH$ (excess)	Fe^{+++}, Al^{+++}, Cr^{+++}	As hydroxide (OH^{-})
IIIB	$NH_4Cl + NH_4OH + H_2S$	Zn^{++},Co^{++}, Ni^{++},Mn^{++}	As sulphides (S^{--})
IV	$NH_4Cl + NH_4OH + (NH_4)_2CO_3$	Ca^{++}, Sr^{++}, Ba^{++}	As carbonate (CO_3^{--})
V	$NH_4Cl + NH_4OH + Na_2HPO_4$ (disodium hydrogen phosphate)	Mg^{++}	As magnesium ammonium phosphate $MgNH_4PO_4$
VI	No general group reagent	Na^{+}, K^{+}, NH_4^{+}	

B. Preparation of original solution (O.S.) in suitable solvent (It is important aspect to choose suitable solvent)

Try by dissolving a little of the given inorganic salts (sample about 0.5–1 gram) in test tube in the following solvents in the given Order (first cold and then hot).

i) Water $\begin{cases} \text{cold} \\ \text{Hot} \end{cases}$

ii) Dilute hydrochloric acid $\begin{cases} \text{cold} \\ \text{Hot} \end{cases}$

iii) Concentrated hydrochloric acid $\begin{cases} \text{cold} \\ \text{Hot} \end{cases}$

Selecting the suitable solvent, make about 15-20 cc (i.e. ¾ of a test tube or full), transparent solution, this solution is known as original solution (O.S.). From this original solution start group separation.

Note:

(a) If HCl is used as solvent, try to dissolve the sample (given salt) in minimum quantity of acid and add water to make up the required volume (15 - 20cc)

(b) Lead chloride is one of the chloride that is insoluble in cold water, but soluble in hot water, so do not forget to see by boiling it.

(c) If precipitate, is not obtained up to group V. the salt must contain Na^+, K^+ and NH_4^+ as basic radicals.

(d) The group separations should be done step by step in the given order.

(e) Start next group only, when you are sure that the preceding group is completely absent.

(f) After getting group, stop and confirm individual basic radical present in inorganic salts systematically.

GROUP SEPERATION TABLE A

(To be performed strictly in the order given)

I. To the first part of O.S. add dilute HCl dropwise: (In case of the sample dissolves in dilute HCl, pass on directly to the Group II						
	II Warm the solution from I (In case Group I metals are absent) and pass H_2S gas for about 10 sec. at the rate of one bubble per sec.					
		III A To the second part of O.S. (add 2 or 3 drops of Conc. HNO_3 and boil only if Fe is suspected from dry test) add double the volume of ammonium chloride, NH_4Cl (shake) and then NH_4OH till distinctly alkaline (smell of ammonia by shaking).				
			III B Warm the solution from IIIA. (in case of Group IIIA, metals ions are absent) and pass H_2S.			
				IV To the third part of O.S. Add 1-2 gm solid. NH_4Cl and then NH_4OH till **distinctly alkaline**. Warm and add $(NH_4)_2CO_3$.		
					V To the fourth part of O.S. add NH_4Cl (solid 1-2 gm), then NH_4OH till **distinctly alkaline** and then Na_2HPO_4 Shake well.	
White ppt Presence of **Group I metal ions** $PbCl_2$ (white) Hg_2Cl_2 (white) $AgCl$ (white)	**Coloured ppt.** Presence of **Group II metal** ions HgS (black) PbS (black) CuS (black) Bi_2S_3(black-brown) CdS (yellow) As_2S_3 (yellow) Sb_2S_3 (yellow) SnS (brown) N.B. Reject whitish yellow ppt. of sulphur	**ppt obtained Presence of Group IIIA metal ions** $Fe(OH)_3$ (brown) $Al(OH)_3$ (white gelatinous) $Cr(OH)_3$ (green)	**ppt obtained** Presence of **Group IIIB metals** ZnS (white) MnS (flesh coloured) CoS (black) NiS (black)	**White ppt Presence of Group IV metal ions** $CaCO_3$ (white) $SrCO_3$ (white) $BaCO_3$ (white)	White ppt of $MgNH_4PO_4$	

Note: If the given salt is light green, better test for Fe^{++} ion before passing on the group IIIA, as follows 2 c.c. of O.S. +2 drops potassium ferricyanide → deep blue ppt i.e. Fe^{++} ion is present. Only if Fe^{++} ion is present, add Conc. HNO_3 in group IIIA, otherwise not.

GROUP SEPARATION TABLE B

(To be performed strictly in the order given)

Experiment	Observation	Inference
I. To the first part of O.S. add dilute HCl dropwise. (In case the sample dissolved in dil. HCl, pass on directly to group II)	**White ppt obtained** $PbCl_2$ (white) Hg_2Cl_2 (white) AgCl (white)	Presence of **group I** metal ions.
II. Warm the solution from I and pass H_2S gas slowly for about 10 sec.	**Coloured precipitate obtained** HgS (black) PbS (black) CuS (black) Bi_2S_3 (black brown) CdS (yellow) As_2S_3 (yellow) Sb_2S_3 (orange) SnS (brown) N.B. If only whitish yellow ppt is formed, reject it.	Presence of **group II** metal ions.
IIIA To the second part of O.S. (add 2 or 3 drops of conc. HNO_3 and boil only if iron is suspected from dry tests otherwise not) and add double the volume of NH_4Cl (shake), and then NH_4OH till distinctly alkaline (smell of ammonia by shaking)	**White ppt obtained** $Fe(OH)_3$ (brown) $Al(OH)_3$ (white gelatinous) $Cr(OH)_3$ (green)	Presence of **group IIIA** metal ions.

III B Warm the solution from IIIA (if IIIA metal ions are absent) and pass H_2S gas.	**Precipitate obtained** ZnS (white) CoS (Black) NiS (Black) MnS (Flesh coloured)	Presence of **group IIIB** metal ions.
IV. To the third part of O.S. add about 1-2 gm solid NH_4Cl, and then NH_4OH till distinctly alkaline. Warm and add $(NH_4)_2CO_3$ solution,	**White ppt obtained** $CaCO_3$(white) $SrCO_3$ (white) $BaCO_3$(white)	Presence of **group IV** metal ions.
V. To the fourth part of O.S. add NH_4Cl (solid 1-2 gm) and NH_4OH till distinctly alkaline and then Na_2HPO_4 Shake well.	**White ppt obtained** $MgNH_4PO_4$ (white crystalline)	Presence of **group V** metal ion Mg^{++}. (basic radicals)

* *Note: **If** the given salt is light green, better test for Fe^{++} ion before passing on to group IIIA, as follows 2 cc of O.S. + 2 **drops** potassium ferricyanide → deep blue ppt i.e. Fe^{++} ion is present. Only if Fe^{++} ion is present, add conc. HNO_3 **in group** IIIA, otherwise not.*

O.S. = Original solution of given sample

Confirmatory tests for the basic radicals (metal ions or cations)

Important points to remember, while performing confirmatory tests :

i. The original solution (prepared solution) must be neutral to litmus paper (Blue and red).

ii. For each individual test, take about 1–2 cc of the original solution (O.S.) in clean test tube.

iii. Add only a few drops of the reagents.

iv. Avoid excess of reagents (the chemical substance, which is used to detect)

Group I Basic Radicals or Metal ions [(Ag^+, Pb^{++}, Hg^+ (ous)]

Silver (Ag^+)

Experiment	Observation	Inference
i. Take 1-2 cc of original solution in test tube and add dil. HCl.	Curdy white precipitate appears, which is soluble in liquor ammonia and precipitate reappears on addition of conc. HNO_3	Presence of silver (Ag^+)
ii. Take O.S. in test tube and add KI (potassium iodide) solution	Yellowish white precipitate appears, which is insoluble in NH_4OH solution (ammonia)	Presence of silver (Ag^+)
iii. Take O.S. in test tube and add potassium chromate solution	Brick red precipitate appears, which is soluble in NH_4OH solution	Presence of silver (Ag^+)

Lead (Pb^{++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add dil. H_2SO_4	White ppt appears, which is insoluble in mineral acids but soluble in hot ammonium acetate solution	Presence of lead (Pb^{++})
ii. Take O.S. in test tube and add potassium chromate (K_2CrO_4) solution	Yellow ppt appears which is soluble in NaOH solution	Presence of lead
iii. Take O.S., in test tube and add KI solution	Yellow ppt appears, dissolves when boiled and reappears when cooled	Presence of lead (Pb^{++})

Mercurous (Hg^+ or Hg_2^+)

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add KI solution	Yellowish green precipitate appears	Presence of mercurous (Hg_2^+)
ii. Take 1-2 cc of O.S. in test tube and add acidified $SnCl_2$ (stannous chloride) solution with dil HCl	Grey ppt appears	Presence of (Hg_2^+) (ous)
iii. Take 1-2 cc of in test tube and add NaOH Solution	Black ppt appears	Presence of mercurous (Hg_2^+)

Reactions involved

Silver (Ag^+):

i. $Ag^+ + HCl \longrightarrow AgCl\downarrow + H^+$

white ppt

$AgCl + 2NH_3 \longrightarrow Ag(NH_3)_2Cl$

Diamine silver (I) chloride

soluble in water

$[Ag(NN_3)_2]Cl + 2HNO_3 \longrightarrow AgCl\downarrow + 2NH_4NO_3$

white ppt reappears

ii. $Ag^+ + KI \longrightarrow AgI\downarrow + K^+$

Potassium Iodide — Yellowish white ppt

iii. $2Ag + K_2CrO_4 \longrightarrow Ag_2CrO_4\downarrow + 2K^+$

Pot. chromate — Brick red ppt

Silver chromate

Lead (Pb^{++}):

i. $Pb^{++} + H_2SO_4 \longrightarrow PbSO_4\downarrow + 2H^+$

White ppt

ii. $Pb^{++} + K_2CrO_4 \longrightarrow PbCrO_4\downarrow + 2K$

Yellow ppt

iii. $Pb^{++} + KI \longrightarrow PbI_2\downarrow + 2K^+$

Yellow ppt

Mercurous (Hg^+ or Hg_2^{++}):

i. $2H^+g + 2Kl \longrightarrow Hg_2I_2\downarrow + 2K^+$

Yellowish green ppt

ii. $Hg^+ + SnCl_2 + 2HCl \longrightarrow Hg\downarrow + SnCl_4 + 2H^+$

Grey ppt

iii. $2Hg^+ + NaOH \longrightarrow Hg_2O\downarrow + Na^+ + H^+$

Black ppt

Group II Basic Radicals (Metal ions)

(Cu^{++}, Pb^{++}, Hg^{++}, Cd^{++}, Sn^{++}, Sb^{+++}, Bi^{+++})

Cupric (Cu^{++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add NaOH solution	Bluish white precipitate appears, which turns black when boiled.	Presence of Cu^{++}
ii. Take 1-2 c of O.S. in test tube add NH_4OH (ammonia solution)	Bluish white ppt appears, with little ammonia, but gives deep blue coloured solution with excess of NH_4OH solution	Presence of Cu^{++}
iii. Take 1-2 cc of O.S. in test tube & add potassium ferrocyanide solution	Reddish brown (chocolate) precipitate appears	Presence of Cu^{++}
iv. Take 1-2 cc of O.S. in test tube and add KI solution	Brown precipitate of cuprous iodide appears which gives white ppt with sodium thiosulphate solution	Presence of Cu^{++}

Mercuric (Hg^{++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add KI solution	Red (at first yellow) ppt appears, which is soluble in excess of potassium iodide solution	Presence of Hg^{++}
ii. Take 1-2 c of O.S. in test tube and add NaOH solution	Yellow ppt appears, which turns red on heating	Presence of Hg^{++}
iii. O.S. + stannous chloride solution	White to grey ppt appears	Presence of Hg^{++}
iv. Take 1-2 cc of O.S. in test tube and add ammonium thiocyanide solution	Deep blue crystalline precipitate appears	Presence of Hg^{++}

Confirmatory test for lead as done in Group I

Bismuth (Bi^{+++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add water in excess	White precipitate appears, which is soluble in mineral acids e.g.dil. HCl, H_2SO_4	Presence of Bi^{+++}
ii. Take 1-2 c of O.S. in test tube, and add NaOH solution	White precipitate appears, which is soluble in mineral acids and turns yellow when boiled	Presence of Bi^{+++}

Cadmium (Cd^{++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S in test tube and add NaOH solution	White precipitate, Soluble in excess of the reagent	Presence of stannous (Sn^{++}) Presence of Stannic (Sn^{4+})
ii. Take 1-2 cc of O.S in test tube and add mercuric chloride ($HgCl_2$) solution	No precipitate White precipitate appears, which turns grey on adding excess of the reagent	Presence of Stannous (Sn^{++}) Presence of Sn^{4+} (stannic)

Antimony (Sb^{+++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test and add water in excess.	White precipitate appears	Presence of Sb^{+++}
ii. Take 1-2 cc of O.S. in test and add NH_4OH solution	White precipitate appears	Presence of Sb^{+++}
iii. Take 1-2 cc of O.S. in test tube and add Zn metal.	Black precipitate appears	Presence of Sb^{+++}

Reactions involved

Copper (Cu^{++})

i. $Cu(OH)_2 + 2NH_4OH + 2NH_4^+ \rightarrow [Cu(NH_3)_4]^{++} + 4H_2O$

Deep blue coloured solution

ii. $2Cu^{++} + K_4[Fe(CN)_6] \longrightarrow Cu_2[Fe(CN)_6]\downarrow + 4K^+$

Pot. ferrocyanide — Reddish brown ppt

iii. $2Cu^{++} + 4KI \longrightarrow Cu_2I_2\downarrow + I_2 + 4K^+$

Brown ppt

Mercuric (Hg^{++})

i. $Hg^{++} + 2KI \longrightarrow HgI_2\downarrow + 2K^+$

Reddish brown ppt

$HgI_2 + 2KI \longrightarrow K_2HgI_4$ (potassium mercuric iodide)

Colourless

ii. $Hg^{++} + 2NaOH \longrightarrow HgO + H_2O + 2Na^+$

Yellow ppt

iii. $2Hg^{++} + Sn^{++} \longrightarrow 2Hg^+\downarrow + Sn^{4+}$

White to grey ppt

Bismuth (Bi^{+++})

i. $Bi^{+++} + H_2O \longrightarrow (BiO)^+ + 2H^+$

White ppt

ii. $Bi^{+++} + 3NaOH \xrightarrow{\text{Boiled}} Bi(OH)_3\downarrow + 3Na^+$

White ppt

$Bi(OH)_3 \xrightarrow{\text{Boiled}} BiO(OH)\downarrow + H_2O$

Yellow ppt

iii. $2Bi^{+++} + 3NaHSnO_2 + 3H_2O \longrightarrow 3NaHSnO_3 + 6H^+ + 2Bi\downarrow$

Black ppt

Cadmium (Cd^{++})

i. $Cd^{++} + 2NaOH \longrightarrow \underset{\text{White ppt}}{Cd(OH)_2\downarrow} + 2Na^+$

ii. $Cd^{++} + 2NH_4OH \longrightarrow \underset{\text{White ppt}}{Cd(OH)_2} + 2NH_4^+$

iii. $Cd(OH)_2 + 2NH_4OH + 2NH_4^+ \longrightarrow [Cd(NH_3)_4]^{++} + 4H_2O$

Tin(Sn^{++} and Sn^{++++})

i. $Sn^{++} + 2NaOH \longrightarrow \underset{\text{White ppt}}{Sn(OH)_2\downarrow} + 2Na^+$

$Sn(OH)_2 + 2NaOH \longrightarrow Na_2SnO_2 + 2H_2O$

$Sn^{4+} + 4NaOH \longrightarrow \underset{\text{White gelatinous ppt}}{Sn(OH)_4\downarrow} + 2Na^+$

$Sn(OH)_4 + 2NaOH \longrightarrow \underset{\text{Sodium stannite}}{Na_2SnO_3} + 3H_2O$

ii. $Sn^{++} + 2HgCl_2 \longrightarrow SnCl_4 + 2Hg^+\downarrow$

White ppt

$Sn^{4+} + HgCl_2 \longrightarrow$ No reaction

Antimony (Sb^{+++})

i. $Sb^{+++} + H_2O \longrightarrow \underset{\text{White ppt}}{(SbO)^+\downarrow} + 2H$

ii. $Sb^{+++} + 6NH_4OH \longrightarrow \underset{\text{White ppt}}{SbO_3} + 6NH_4^+ + 3H_2O$

iii. $2Sb^{+++} + 3Zn \longrightarrow \underset{\text{Black ppt}}{2Sb\downarrow} + 3Zn^{++}$

Group IIIA Basic Radicals (Metal ions)

Iron [Fe^{++} ferrous and Fe^{+++} (ferric)]

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add potassium ferrocyanide	No precipitate Deep blue precipitate	Presence of Fe^{++} Presence of Fe^{+++}
ii. Take 1-2 cc of O.S. in test tube and add potassium ferricyanide solution.	Deep blue ppt Greenish colouration appear	Presence of Fe^{++} Presence of Fe^{+++}
iii. Take 1-2 cc of O.S. in test tube and add ammonium thiocyanate	No ppt or colouration Deep red colouration appears	Presence of Fe^{++} Presence of Fe^{+++}
iv. Take 1-2 cc of O.S. in test tube and add NH_4OH solution	Dirty green ppt appears Brown ppt appears	Presence of Fe^{++}. Presence of Fe^{+++}
v. Take 1-2 cc of O.S. in test tube, add dil. H_2SO_4 and 1-2 drops of $KMnO_4$	Pink colour of $KMnO_4$ is discharged Pink colour is not discharged	Presence of Fe^{++} Presence of Fe^{+++}

Aluminium (Al^{+++})

Experiment	Observation	Inference
i. Take, 1-2 cc of O.S. in test tube and add NaOH solution	White ppt appears which is soluble in excess of the reagent and reprecipitated by NH_4Cl	Presence of Al^{+++}
ii. Take 1-2 cc of O.S. in test tube and add NH_4OH	White gelatinous ppt	Presence of Al^{+++}
iii. Take 1-2 cc of O.S. in test tube and add disodium hydrogen phosphate solution	White ppt appears, which is insoluble in acetic acid	Presence of Al^{+++}

Chromium (Cr^{+++})

Experiment	Observation	Inference
i. Take 1-2 cc of O.S. in test tube and add NaOH solution	Green ppt appears, which is soluble in excess of the reagent giving green solution	Presence of Cr^{+++}
ii. Take 1-2 cc of O.S. in test tube and add disodium hydrogen phosphate	Green ppt appears, which is soluble in mineral acids.	Presence of Cr^{+++}
iii. O.S. + NaOH + Br_2 water. Boil it with acetic acid and add lead acetate.	Yellow precipitate appears	Presence of Cr^{+++}

Reactions involved

Iron

1. $Fe^{++} + K_4Fe(CN)_6 \longrightarrow K_2Fe[Fe(CN_6)] + 2K^+$

Potassium ferroferricyanide (white)

$4Fe^{+++} + 3K_4[Fe(CN)_6] \longrightarrow Fe_4[Fe(CN)_6]_3 \downarrow + 12K^+$

Pot. ferrrocyanide — deep blue ppt. Ferric ferrocyanate

2. $3Fe^{2+} + 3K_3[Fe(CN)_6] \longrightarrow Fe_3[Fe(CN)_6]_2\downarrow + 6K^+$

Pot. ferricyaflide — deep blue ppt ferrous ferricyanide

$Fe^{+++} + K_3[Fe(CN)_6] \longrightarrow Fe[Fe(CN)_6] + 3K^+$

Ferriferricyanide (green)

3. $Fe^{++} + NH_4SCN \longrightarrow$ No reaction

$Fe^{3+} + 3NH_4CNS \longrightarrow Fe(CNS)_3 + 3NH_4^+$

Amm. thiocyanate — Ferric thiocyanate

4. $Fe^{++} + 2NH_4OH \longrightarrow Fe(OH)_2\downarrow + 2NH_4^+$

Green ppt

$Fe^{3+} + 3NH_4OH \longrightarrow Fe(OH)_3 \downarrow + 3NH_4^+$

Reddish brown ppt
Ferric hydroxide

5. $4Fe^{++} + 2KMnO_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 2Fe_2(SO_4)_3 + 3H_2O$

$Fe^{+++} + KMnO_4 + H_2SO_4 \longrightarrow$ No reaction

Aluminium (Al^{3+})

1. $Al^{3+} + 3NaOH \longrightarrow Al(OH)_3\downarrow + 3Na^+$

Gelatinous white ppt

$Al(OH)_3 + NaOH \longrightarrow NaAlO_2 + 2H_2O$

Sod. Meta-aluminate

2. $Al^{+++} + 3NH_4OH \longrightarrow Al(OH)_3\downarrow + 3NH_4^+$

White gelatinous ppt

3. $2Al^{+++} + 4Na_2HPO_4 \longrightarrow 2AlPO_4\downarrow + 6Na^+ + 2NaH_2PO_4$

Disodium hydrogen Phosphate — White ppt Al. phosphate — Sod. dihydrogen phosphate

Chromium (Cr^{+++})

1. $Cr^{+++} + 3NaOH \longrightarrow Cr(OH)_3\downarrow + 3Na^+$

Green ppt

2. $Cr^{+++} + Na_2HPO_4 \longrightarrow CrPO_4\downarrow + 3Na^+ + NaH_2PO_4$

Green ppt
Chromium phosphate

3. $Br_2 + 2NaOH \longrightarrow NaBrO + NaBr + H_2O$

Sod. Hypobromite

$NaBrO \longrightarrow NaBr + [O]$

$Cr^{+++} + 3NaOH \longrightarrow Cr(OH)_3 + 3Na^+$

$2Cr(OH)_3 + 4NaOH + 3[O] \longrightarrow 2Na_2CrO_4 + 5H_2O$

Sod. chromate

Group III B basic radicals (Metal ions: Zn^{2+}, Ni^{2+}, Co^{2+}, Mn^{2+}) Zinc (Zn^{++})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube and add NaOH solution	White ppt appears which is soluble in excess of the sodium hydroxides solution	Presence of Zn^{++}
2. Take 1-2 cc of O.S. in test tubes & add potassium ferrocyanide solution	White precipitate appears	Presence of Zn^{++}

Nickel (Ni^{2+})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube and add NaOH solution	Apple green ppt appears	Presence of Ni^{++}
2. Take 1-2 cc of O.S. in test tube and add few drops of NH_4OH and dimethyl glyoxime (DMG)	Red ppt of nickel dimethyl glyoxime	Presence of Ni^{++}

Cobalt (Co^{2+})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S in test tube and add NaOH solution.	1. Blue precipitate appears	1. Presence of Co^{++}
2. Take O.S. in test tube and add Ammonium thiocyanate, ethyl alcohol and shake nicely	2. Blue layer of ethyl alcohol	2. Presence of Co^{++}
3. Take 1-2 cc of O.S in test tube and add solid ammonium thiocyanate and at last add $HgCl_2$ solution.	3. Deep blue precipitate appears	3. Presence of Co^{++}

Manganese (Mn^{++})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S in test tube and add conc. HNO_3+ Red lead and heat	1. Purple colouration appears	1. Presence of Mn^{++}
2. Take 1-2 cc of O.S. in test tube and add ammonium sulphide	2. Flesh coloured ppt appears	2. Presence of Mn^{++}
3. Take 1-2 cc of O.S. in test tube and add NaOH solution	3. White ppt appears which turns black on addition of Br_2 water	3. Presence of Mn^{++}
4. Take given salt + Na_2CO_3 + little KNO_3. Heat in platinum wire loop to prepare bead	4. The bead is green	4. Presence of Mn^{++}
5. Dissolve the bead in water and acidify	5. Pink coloured solution appears.	5. Presence of Mn^{++}

Reactions involved

Zinc

1. $Zn^{++} + 2NaOH \longrightarrow \underset{\text{White ppt}}{Zn(OH)_2\downarrow} + 2Na^+$

2. $Zn(OH)_2 + 2NaOH \longrightarrow \underset{\text{Sodium zincate}}{Na_2ZnO_2} + 2H_2O$

3. $2Zn^{++} + \underset{\text{Potassium ferrocyanide}}{K_4[Fe(CN)_6]} \longrightarrow \underset{\text{Zinc ferrocyanide}}{Zn_2[Fe(CN)_6]} + 4K^+$

Nickel

1. $Ni^{++} + 2NaOH \longrightarrow \underset{\text{Apple green ppt}}{Ni(OH)_2\downarrow} + 2Na^+$

2. $Ni^{++} + 2NH_3 + 2C_4H_8O_2N_2 \rightarrow (C_4H_7O_2N_2)_2 Ni \downarrow + 2NH_4^+$

DMG Red ppt

Cobalt

1. $Co^{++} + 2NaOH \longrightarrow Co(OH)_2\downarrow + 2Na^+$

Blue ppt

2. $Co^{++} + 4NH_4CNS \longrightarrow (NH_4)_2 [Co(CNS)_4] + 2NH_4^+$

Flesh coloured ppt

3. $Mn^{++} + 2NaOH \longrightarrow Mn(OH)_2\downarrow + 2Na^+$

White ppt

$Mn(OH)_2 + [O] \longrightarrow MnO_2\downarrow + H_2O$

Black ppt

4. $MnO + Na_2CO_3 + O_2 + Na_2MnO_4 + CO_2$

$3Na_2MnO_4 + 2H_2O \longrightarrow 2NaMnO_4 + MnO_2 + 4NaOH$

Sod. manganate Sod. permanganate (pink)

Group IV Basic radicals (Metal ions)

(Ca^{++}, Ba^{++}, Sr^{++})

Experiment	Observation	Inference
1. Take 1-2cc of O.S. in test tube and add potassium chromate solution.	1. a. No precipitate b. Yellow ppt, which is soluble in acetic acid. c. Yellow ppt, which is insoluble in acetic acid	1. a. Presence of Ca^{++} b. Presence of Sr^{++} c. Presence of Ba^{++}

Calcium (Ca^{++})

Experiment	Observation	Inference
1. Take 1-2cc of O.S. in test tube and add ammonium carbonate solution	1. White ppt appears, which is soluble in acetic and mineral acid with effervescence (coming out bubble)	1. Presence of Ca^{++}
2. Take 1-2 cc of O.S. in test tube and add ammonium oxalate solution.	2. White ppt appears, which is insoluble in acetic acid hut readily soluble in dil. HCl	2. Presence of Ca^{++}
3. Take 1-2 cc of O.S. in test tube and add few cc of $CaSO_4$ solution	3. White ppt appears, which is insoluble in acetic acid but readily soluble in dil. HCl	3. Presence of Ca^{++}
4. Take 1-2 cc of O.S. in test tube and add few cc of potassium ferrocyanide solution.	4. No precipitate	4. Presence of Ca^{++}

Barium (Ba^{++})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube and add $(NH_4)_2CO_3$ solution	1. White ppt appears which is soluble in acetic acid and dil HCl	1. Presence of Ba^{++}
2. Take 1-2 cc of O.S. in test tube and add ammonium oxalate solution	2. While ppt appears, which is soluble in acetic acid and dil. HCl	2. Presence of Ba^{++}
3. Take 1-2 cc of O.S in test tube and add $CaSO_4$ Solution	3. White precipitate appears, which insoluble in dilute.HCl	3. Presence of Ba^{++}

Strontium (Sr^{++})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube and add $(NH_4)_2CO_3$ solution	1. White ppt appears which is soluble in acetic acid and dil. HCl	1. Presence of Sr^{++}
2. Take 1-2 cc of O.S. in test tube and add ammonium oxalate solution	2. White ppt appears which is slightly soluble in acetic acid but readily soluble in dil. HCl	2. Presence of Sr^{++}
3. Take 1-2 cc of O.S. in test tube and add $CaSO_4$ solution	3. White ppt appears, when boiled or on standing	3. Presence of Sr^{++}

Calcium

1. $Ca^{++} + (NH_4)_2CO_3 \longrightarrow \underset{\text{White ppt}}{CaCO_3\downarrow} + 2NH_4^+$

2. $Ca^{++} + \underset{\text{Amm. Oxalate}}{(NH_4)_2C_2O_4} \longrightarrow \underset{\text{Cal. oxalate}}{CaC_2O_4} + 2NH_4^+$

   $CaC_2O_4 + 2HCl \longrightarrow \underset{\text{Oxalic acid}}{H_2C_2O_4} + CaCl_2$

3. $Ca^{++} + K_4[Fe(CN)_6] \longrightarrow CaK_2[Fe(CN)_6] + 2K^+$

Barium

1. $Ba^{++} + (NH_4)_2CO_3 \longrightarrow \underset{\text{White ppt}}{BaCO_3\downarrow} + 2NH_4^+$

   $BaCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ba + CO_2 + H_2O$

2. $Ba^{++} + (NH_4)_2C_2O_4 \longrightarrow BaC_2O_4\downarrow + 2NH_4^+$

White ppt (Barium oxalate)

3. $Ba^{++} + CaSO_4 \longrightarrow BaSO_4\downarrow + Ca^{++}$

White ppt

Strontium

1. $Sr^{++} + (NH_4)_2 CO_3 \longrightarrow SrCO_3 \downarrow + 2NH_4^+$

Amm. Carbonate — White ppt

$SrCO_3 + 2CH_3COOH \longrightarrow (CH_3COO)_2Sr + CO_2 + H_2O$

Strontium acetate

2. $Sr^{++} + (NH_4)_2C_2O_4 \longrightarrow SrC_2O_4 \downarrow 2NH_4^+$

Amm, Oxalate — White ppt

$SrC_2O_4 + 2HCI \longrightarrow SrCl_2 + H_2C_2O_4$ (Oxalic acid)

3. $Sr^{++} + CaSO_4 \longrightarrow SrSO_4 \downarrow + Ca^{++}$

White ppt

Group V Basic radicals (Metal ions)

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube and add NaOH solution.	1. White precipitate appears, which is soluble in NH_4Cl	1. Presence of Mg^{++}
2. Take 1-2 c of O.S. in test-tube and add NH_4OH solution.	2. White gelatinous ppt appears, which is insoluble in NH_4Cl solution. *Note: Test No. II may not be positive with Mg- solution, made in dil. HCl*	2. Presence of Mg^{++}
3. Take 1-2 cc O.S. in test tube and add disodium hydrogen phosphate	3. White crystalline (granular) precipitate soluble in acetic acid and dil. mineral acid,	3. Presence of Mg^{++}

Reactions involved

Magnesium

1. $Mg^{++} + 2NaOH \longrightarrow Mg(OH)_2 \downarrow + 2Na^+$

White ppt

$Mg(OH)_2 + 2NH_4Cl \longrightarrow MgCl_2 + 2NH_4OH$

2. $Mg^{++} + 2NH_4OH \longrightarrow Mg(OH)_2 \downarrow + 2NH_4^+$

White ppt

$Mg(OH)_2 + 2NH_4Cl \longrightarrow MgCl_2 + 2NH_4OH$

3. $Mg^{++} + Na_2HPO_4 + NH_4OH \rightarrow Mg(NH_4)PO_4 + 2Na^+ + H_2O$

Disodium hydrogen Phosphate — white granular ppt magnesium ammonium phosphate

Special Confirmatory Test for Na^+, K^+ and NH_4^+

Sodium (Na^+)

Experiment	Observation	Inference
1. Perform flame test with the salt	1. Brilliant golden yellow through naked eye and colourless through blue glass	1. Presence of Na^+

Potassium (K^+)

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test-tube and add conc. tartaric acid.	1. White crystalline precipitate appears.	1. Presence of K^+
2. Take 1-2 cc of O.S. in test tube and add sodium cobalt nitrite solution	2. Yellow crystalline ppt appears.	2. Presence of K^+
3. Perform flame test with the salt	3. Violet colour through naked eye and pink through blue glass	3. Presence of K^+

Ammonium (NH_4^+)

Observation	Observation	Inference
1. Take a little salt in test tube, add NaOH solution and warm.	1. Smell of ammonia gas, which turns mercurous nitrate solution paper black.	1. Presence of NH^+_4
2. Take 1-2 cc of O.S. in test tube and add Nessler's reagent.	2. Brown ppt or brown colouration appears.	2. Presence of NH_4^+

Reactions involved

Potassium (K^+)

1. $K^+ + H_2C_4H_4O_6 \longrightarrow KHC_4H_4O_6\downarrow + H^+$

   Tartaric acid — Potassium tartarate

2. $2K^+ + Na_3[Co(NO_2)_3] \longrightarrow K_2Na[Co(NO_2)_6]\downarrow + 2Na^+$

   Sod. Cobalt (III) nitrite — Yellow ppt (Pot. Sodium cobaltnitrite)

Ammonium (NH_4^+)

1. $NH_4^+ + NaOH \longrightarrow NH_3\uparrow + Na^+ + H_2O$

   $4NH_3 + 2Hg_2(NO_3)_2 + H_2O \rightarrow Hg_2ONH_2NO_3\downarrow + 2Hg\downarrow + 3NH_4NO_3$

   Mercurous nitrate — black ppt

2. $2K_2HgI_4 + NH_3 + 3KOH \longrightarrow NH_2HgOHgI\downarrow + 7KI + 2H_2O$

   Potassium Mercuric iodide — Brown ppt (Million's base)

V. Wet Test for Acid Radicals

Experiment No. 22

Object: TO DETECT ACID RADICAL IN THE GIVEN INORGANIC SALTS

Sample Number: A_1, A_2,...

Physical Characters:

State: Crystalline (granular)

Colour:

Solubility: In water

Note: *If the salt is insoluble in water cold or hot, reject the following tests and perform dry tests for acid radicals.*

Theory

Define salts, acid and basic radicals with suitable examples.

Test I: Silver Nitrate Test For (Cl^-, Br^-, I^-)

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube, add dil. HNO_3 and boil 3-5 minutes, cool in water and add $AgNO_3$ solution.	1. A curdy white precipitate appears, which is soluble in ammonia (NH_4OH) solution and ppt reappears on addition of conc. HNO_3.	1. Presence of Cl^- (Chloride)
	2. A pale yellow precipitate appears which is slightly soluble in ammonia solution.	2. Presence of Br^- (bromide)
	3. A curdy yellow ppt appears which is insoluble in NH_4OH.	3. Presence of I^- (iodide)

Confirmatory Test for Bromide (Br^-) and Iodide (I^-)

Experiment	Observation	Inference
1. 1-2 cc of O.S. in test tube and add 1-2 cc of $CHCl_3$ or CS_2 solvent and chlorine water and shake nicely or take 1cc of conc. HCl, add small crystal of potassium permanganate, 1-2 cc of O.S. and 1-2 cc of chloroform or carbon tetrachloride and shake nicely.	1. The bottom layers of chloroform is coloured. i. Brown ii. Violet	i. Presence of Br^-. ii. Presence of I^-

Reactions involved

$Cl^- + AgNO_3 \longrightarrow AgCl + NO_3^-$

Curdy white ppt

$AgCl + 2NH_3 \longrightarrow [Ag(NH_3)_2]Cl$

Soluble complex compound

Diamine silver chloride

$Br^- + AgNO_3 \longrightarrow AgBr \downarrow + NO_3^-$

Pale yellow ppt

$AgBr + 2NH_3 \longrightarrow [Ag(NH_3)_2]Br$

$I^- + AgNO_3 \longrightarrow AgI \downarrow + NO_3^-$

Deep yellow ppt

Q. Why original solution (O.S.) is boiled with Conc. HNO_3 before the test of halides?

Ans: To overcome S^{--} and CN^- in the form of H_2S and HCN gas, it is boiled with conc. HNO_3 acid.

$$S^{--} + HNO_3 \longrightarrow H_2S\uparrow + NO_3^-$$

$$CN^- + HNO_3 \longrightarrow HCN\uparrow + NO_3^-$$

Confirmatory Test for Br^- and I^-

Chlorine liberates bromine and iodine from bromide and iodide salt respectively. Chloroform or carbon tetrachloride or carbon disulphide absorbs Br_2 and I_2, thus liberated to give brown and violet colouration respectively.

$$2Br^- + Cl_2 \longrightarrow \underset{\text{Brown}}{Br_2} + 2Cl^-$$

$$2I^- + Cl_2 \longrightarrow \underset{\text{Violet}}{I_2} + 2Cl^-$$

Test 2: Barium chloride test for CO_3^{--}, SO_4^{--}, SO_3^{--}

Experiment	Observation	Inference
(1)Take 1-2 cc of O.S. in test tube, add few drops of $BaCl_2$ solution	(1)White precipitate appears.	(1) Presence of SO_4^{--}, CO_3^{--}, SO_3^{--}
(2) Now in white precipitate add dil. HCl	(2)(a)white ppt does not dissolve in dil. HCl.	Presence of SO_4^{--} (sulphate)
	(b)The precipitate dissolves with the evolution of (i) CO_2 gas, which turns lime water milky colour	Presence of CO_3^{--}
	(ii) SO_2 gas, which turns acidified solution of potassium dichromate solution into green colour.	Presence of SO_3^{--} (sulphite)

Reactions involved

$BaCl_2 + CO_3^{--} \longrightarrow BaCO_3\downarrow + 2Cl^-$

White ppt

$BaCO_3 + dil.2HCl \longrightarrow BaCl_2 + H_2O + CO_2\uparrow$

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

Lime water — Milky colour

$BaCl_2 + SO_3^{--} \longrightarrow BaSO_3 + 2Cl^-$

$BaSO_3 + dil.\ 2HCl \longrightarrow BaCl_2 + H_2O + SO_2\uparrow$

$K_2Cr_2O_7 + dil.H_2SO_4 + SO_2 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + H_2O$

Orange yellow — Chromium sulphate (green)

$BaCl_2 + SO_4^{--} \longrightarrow BaSO_4\downarrow + 2Cl^-$

White ppt

$BaSO_4 + dil.HCl \longrightarrow$ No reaction

Test 3: For Nitrate (NO_3^-)

Experiment	Observation	Inference
1. Take 1-2 cc O.S. in test tube, add 1-2 cc of conc. H_2SO_4, cool and add freshly prepared $FeSO_4$ solution slowly along with the walls of test tube.	1. A brown ring appears at the junction of the liquid layers.	1. Presence of nitrate (NO_3^-)
2. Take 1-2 cc of O.S. in test tube, add metallic zinc, acetic acid, warm and add KI solution and test with starch solution.	Blue colouration	Presence of nitrate (NO_3^-)

Reactions involved

i) $NO_3^- + H_2SO_4 \longrightarrow HNO_3 + HSO_4^-$

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

$FeSO_4 + NO + 5H_2O \longrightarrow Fe[(H_2O)_5NO]SO_4$

Penta aquanitrosyl ferrous sulphate
(Brown ring)

or

$FeSO_4 + NO \longrightarrow FeSO_4.NO$

(Nitrosyl ferrous sulphate)
Brown ring

ii) $2CH_3COOH + Zn \longrightarrow (CH_2COO)_2\,Zn + 2[H]$

Zinc acetate

$KNO_3 + 2[H] \longrightarrow KNO_2 + H_2O$

Pot. Nitrate — potassium nitrite

$2KNO_2 + 4CH_3COOH + 2KI \rightarrow 4CH_3COOK + I_2 + 2NO + 2H_2O$

Test 4: For Nitrite (NO_2^-)

Experiment	Observation	Inference
1. Take 1-2 cc of O.S in test tube, add acidified solution of potassium permanganate.	1. The pink colour is discharged	1. Presence of nitrite (NO_2^-)
2. Take 1-2 cc of O.S. in test tube and acetic acid + KI solution add starch solution	2. Blue colouration	2. Presence of nitrite (NO_2^-)
3. Take 1-2 cc of O.S. in test tube, add 2 drops of acetic acid and 2 cc of ferrous sulphate solution.	3. Dark brown solution appears	3. Presence of Nitrite (NO_2^-)

Reactions involved

i) Nitrites (NO_2^-) are oxidized to nitrates (NO_3^-) by acidified solution of $KMnO_4$.(Potassium permanganate)

$$2KMnO_4 + 3H_2SO_4 + 5KNO_2 \rightarrow K_2SO_4 + 2MnSO_4 + 5KNO_3 + 3H_2O$$

ii) Nitrites liberates free iodine from iodide in acidified solution, thus liberated iodine turns starch blue.

$$2KNO_2 + 4CH_3COOH + 2K \rightarrow 4CH_3COOK + I_2 + 2NO + 2H_2O$$

iii) $KNO_2 + CH_3CHOOH \longrightarrow CH_3COOK + HNO_2$

Test 5: For sulphide (S^{--})

Experiment	Observation	Inference
1. Take 1-2 cc of O.S. in test tube, add NaOH solution and freshly prepared sodium nitroprusside solution.	1. Violet colouration appears	1. Presence of sulphide (S^{--})
2. Take 1-2 cc of O.S. in test tube and add lead acetate solution.	2. Black ppt appears	2. Presence of S^{--}

i. A soluble sulphide (S^{--}) in alkaline solution gives violet colour with freshly prepared sodium nitroprusside solution.

$$Na_2S + Na_2[Fe(CN)_5NO] \longrightarrow \underset{\text{Sodium thionitroprusside}}{Na_4[Fe(CN)_5NOS]}$$

ii. A soluble sulphide precipitates as black lead sulphide (PbS) from any soluble lead salt solution.

$$Na_2S + (CH_3COO)_2Pb \longrightarrow \underset{\text{Black ppt}}{PbS\downarrow} + 2CH_3COONa$$

Viva Voice

1. What are basic and acid radicals?
2. What are salts? Give suitable example.
3. What are group reagents? Give the group reagents for group II.
4. In which form group IIIB give precipitate? Give two examples.
5. Why group IIA metals give precipitate with H_2S in acidic medium but group IIIB do not give?
6. Why Group IIIB give precipitate with H_2S in alkaline medium?
7. Explain $CaCO_3$, $BaCO_3$ and $SrCO_3$, give precipitate with $(NH_4)_2CO_3$ in presence of NH_4Cl and NH_4OH but not $MgCO_3$.
8. What is common ions effect?
9. What is solubility product?
10. Give the principle of precipitation.
11. What is brown ring test? Give the reaction involved.
12. What is filter ash test (Cobalt nitrate test). Give the reaction.
13. What is flame test? What type of salts are confirmed?
14. What is borax bead test? What type of salts are confirmed?
15. Why original solution is boiled with Conc. HNO_3 before the test of halides?
16. Give the use of silver nitrate in acid radical test and also give the reaction involved.
17. What is $BaCl_2$ test? Give the reaction involved.
18. What is Nessler's reagent? Give its use.
19. Define precipitation, sublimation, distillation, crystallisation and neutralisation with suitable example.
20. What is original solution?

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Table 1 : Vapour pressure of water (aqueous tension)
Table 2 : Surface tension of liquids at different temperatures
Table 3 : Viscosity of some liquids at different temperature in milli poise.
Table 4 : Solubilities of some common compounds at different temperature gram/100 gram H_2O.
Table 5 : Symbol, atomic number and relative atomic mass

Vapour pressure of water (aqueous tension)

Temperature	Vapour pressure (mm Mercury)	Temperature (°C)	Vapour pressure (mm of mercury)
9	8.58	26	24.99
10	9.18	27	26.50
11	9.81	28	28.10
12	10.48	29	29.78
13	11.19	30	31.55
14	11.94	31	33.42
15	12.73	32	35.37
16	13.56	33	37.43
17	14.45	34	39.59
18	15.38	35	41.85
19	16.37	36	42.02
20	17.41	37	46.07
21	18.50	38	49.03
22	19.07	39	52.00
23	20.09	40	54.09
24	22.18	45	71.09
25	23.54	50	92.05

Surface tension of liquids at different temperatures

Substance	γ in dynes/cm at temperature in °C						
	15	20	25	30	35	40	45
Acetone	23.4	23.0	22.5	22.1	–	20.16	–
Benzene	29.6	28.9	28.2	27.5	26.8	26.1	25.4
Carbon tetrachloride	27.55	26.95	26.35	25.75	25.15	24.55	23.95
Ethyl alcohol	22.1	21.7	21.3	20.9	20.5	20.1	19.7
Water	73.50	72.85	72.09	71.34	70.58	69.82	68.00

Viscosities of some liquids at different temperature in millipoise

Substance	15°C	20°C	30°C	40°C	50°C
Acetone	–	3.3	2.95	–	–
Benzene	6.97	6.49	5.62	4.92	4.37
Carbon tetrachloride	–	9.69	8.41	7.38	6.53
Diethyl ether	–	2.322	–	–	–
Ethyl alcohol	–	11.9	9.89	8.27	6.97
Water	11.40	10.05	8.01	6.56	5.49

Solubilities of some common compounds at different temperature gram/100 gram H_2O

S.N	Chemical substance	20°C	25°C	30°C	35°C	40°C	45°C	50°C	60°C	100°C
1.	Blue vitriol	20.7		25	28.5	40.2		33.3	40	75.4
2.	Green vitriol	26.6	29.8	32.9	36.4	40.3	44.1	78.5	55	36
3.	Potassium chloride	34.5	36	37.5	38.9	160	41.7	43.1	45.9	56.08
4.	Pot. Iodide	144.2	148.4	152.3	156	65.5	164	168	176	209
5.	Pot. Nitrate	33.0	39.7	46.7	55.6	36.6	75.1	84.1	110.1	248.6
6.	Sodium Chloride	35.9	36.1	36.2	36.34	105	36.83	37	37.3	39.8
7.	Sodium nitrate	88.1	92.13	96.15	101	238	110	115	124	177
8.	Cane sugar	204	211.4	219.5	228.4	45.8	248.7	260	287	487.2
9	Amm. chloride	37.28	39.3	41.4	43.6	297	48	50.4	55.2	77.3
.10	Amm. nitrate	192	214	242	265		–	344	421	87
11.	Barium chloride	35.7	37	38.2	39.5	40.7	42.2	43.6	46.4	58
12.	Calcium chloride	74	85.9	100.8	107.5	11.5	13.04	132.6	136.8	159.1

Table 5 : Atomic weights and other constants of some commonly used elements

Elements	Symbol	At. No.	At. Wt.	Density (g/mL)	Specific heat
Hydrogen	H	1	1.008	0.00008987	–
Carbon	C	6	12.011	3.51	0.17
Nitrogen	N	7	14.007	0.0012507	–
Oxygen	O	8	15.9994	0.001437	–
Fluorine	F	9	18.998	0.001694	–
Sodium	Na	11	22.990	0971	0.293
Magnesium	Mg	12	24.312	1.74	0.248
Aluminum	Al	13	26.98	2.703	0.214
Phosphorus	P	15	30.974	1.83	0.202
Sulphur	S	16	32.064	1.96	0.174
Chlorine	Cl	17	35.453	0.00322	–
Potassium	K	19	39.102	0.875	0.166
Calcium	Ca	20	40.08	1.548	0.149
Chromium	Cr	24	51.996	6.22	0.112
Manganese	Mn	25	54.938	7.06	0.122
Copper	Cu	29	63.24	8.95	0.093
Zinc	Zn	30	65.37	7.1	0.093
Arsenic	As	33	74.92	5.73	0.083
Bromine	Br	35	79.909	3.12	0.107
Silver	Ag	47	107.870	10.5	0.058
Tin	Sn	50	118.69	7.2	0.055
Barium	Ba	56	137.34	3.78	0.068
Iodine	I	53	126.904	4.94	0.054
Lead	Pb	82	207.19	11.4	0.0305
Gold	Au	79	196.967	19.3	0.031
Mercury	Hg	80	200.59	13.515	0.033

Viva Voice

1. What are ions?
2. Define chemical reactions with suitable examples.
3. What is sublimation?
4. Give two examples of substances that sublimizes.
5. How can you distinguish Fe^{++} & Fe^{+++} ion present in the solution?
6. Equivalent weight of magnesium is 12. What does it mean?
7. Equivalent weight of Cu can't be determined by hydrogen displacement method. Why?
8. Define surface tension. Give its unit.
9. Define viscosity. Give its unit.
10. How do you test for Cl^-, SO_4^{--} & NO_3^- ?
11. What happens when dil. HCl is added over marble chips?
12. What are oxidizing & reducing agents?
13. How would you detect CO_2 gas is acidic?
14. Show that ammonia is basic in nature.
15. Define crystals, crystallization, water of crystallisation, mother liquor.
16. Define saturated solution, supersaturated solution, millicrystals.
17. What are qualitative and quantitative analysis?
18. Define dry and wet test.
19. Define acid and basic radicals with examples.
20. Why is lead (Pb^{++}) kept in group I as well as group II?
21. Name the salts which produce cracking noise on heating. Why do these salts produce cracking noise on heating?

22. What is blue lake?

23. In the third group precipitation NH_4Cl is used in excess but NH_4OH is not used in excess, why?

24. Why freshly prepared $FeSO_4$ solution is used in the ring test?

25. Define the following terms:

    a. Common ion effect
    b. Ionic product
    c. Solubility product
    d. Law of mass action
    e. Solubility product principle

26. What are the basis of classification of basic radicals into different groups?

27. Write the molecular formula of lime water and milk of lime.

28. Ammonium hydroxide is stored in stoppered bottles, why?

29. Silver nitrate solution is stored in dark brown bottle, why?

30. Lime water is stored in stoppered bottle, why?

31. What is fume cupboard? Write its use.

32. How will you distinguish NH_4OH and NaOH solution?

33. How will you distinguish between CO_3^{--} and SO_3^{--}?

34. Can conc. HCl be used as group reagent for Group I instead of dil. HCl?

35. In the precipitation of Group IIIA, which should be added first, NH_4Cl or NH_4OH? Why?

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Books on Chemistry for Class XI & XII

- A Textbook of Higher Secondary Chemistry, Class XI
- Higher Secondary Chemistry, Class XII
- Higher Secondary Practical Chemistry, Volume I
- Higher Secondary Practical Chemistry, Volume II

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