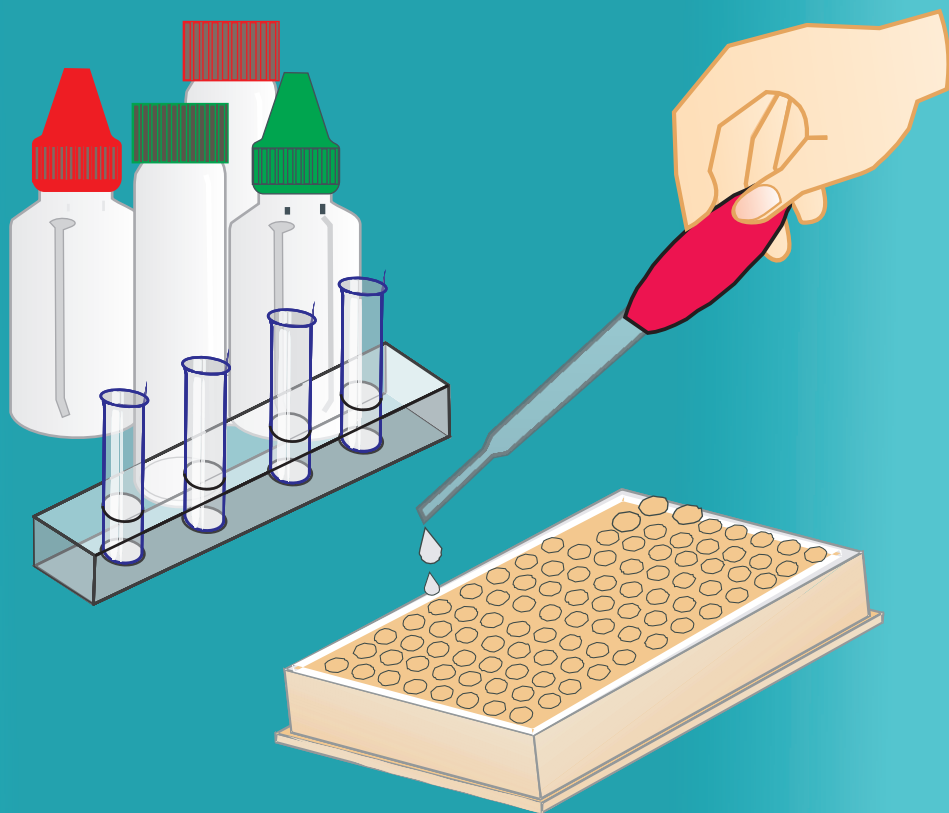


Manual of Microscale Chemistry Laboratory Kit

For Classes XI and XII



MANUAL OF MICROSCALE CHEMISTRY LABORATORY KIT

For Classes XI and XII

विद्यया ऽ मृतमश्नुते



एन सी ई आर टी
NCERT

राष्ट्रीय शैक्षिक अनुसंधान और प्रशिक्षण परिषद्
NATIONAL COUNCIL OF EDUCATIONAL RESEARCH AND TRAINING

ISBN 978-93-5007-861-7

First Edition

April 2018 Chaitra 1940

PD 2T BS

© **National Council of
Educational Research and
Training, 2018**

₹ 70.00

Printed on 80 GSM paper

Published at the Publication
Division by the Secretary,
National Council of Educational
Research and Training,
Sri Aurobindo Marg, New Delhi
110016 and printed at

ALL RIGHTS RESERVED

- ❑ No part of this publication may be reproduced, stored in a retrieval system or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise without the prior permission of the publisher.
- ❑ This book is sold subject to the condition that it shall not, by way of trade, be lent, re-sold, hired out or otherwise disposed of without the publisher's consent, in any form of binding or cover other than that in which it is published.
- ❑ The correct price of this publication is the price printed on this page. Any revised price indicated by a rubber stamp or by a sticker or by any other means is incorrect and should be unacceptable.

OFFICES OF THE PUBLICATION

DIVISION, NCERT

NCERT Campus
Sri Aurobindo Marg
New Delhi 110 016
Phone : 011-26562708

108, 100 Feet Road
Hosdakere Halli Extension
Banashankari III Stage
Bengaluru 560 085
Phone : 080-26725740

Navjivan Trust Building
P.O. Navjivan
Ahmedabad 380 014
Phone : 079-27541446

CWC Campus
Opp. Dhankal Bus Stop
Panihati
Kolkata 700 114
Phone : 033-25530454

CWC Complex
Maligaon
Guwahati 781 021
Phone : 0361-2674869

Publication Team

| | |
|----------------------------|------------------|
| Head, Publication Division | : M. Siraj Anwar |
| Chief Editor | : Shveta Uppal |
| Chief Business Manager | : Gautam Ganguly |
| Chief Production Officer | : Arun Chitkara |
| Editor | : Bijnan Sutar |
| Production Assistant | : ? |

PREFACE

The present day Chemistry laboratory has the following features:

- lot of wastage of chemicals and increased cost of setting up and maintaining chemistry laboratory.
- increased water and air pollution due to excessive use of chemicals and obnoxious and toxic fumes, leading to environmental pollution.
- many accidents due to fire and hazardous chemicals.
- cook-book approach to practicals, giving little time for thinking, increases information load rather than clarifying the concepts.

How to maintain a pollution-free environment, how to cut chemistry laboratory costs and to handle chemical wastes are the present day concerns. These concerns have been addressed by designing and developing an alternative way of performing chemistry experiments in the laboratory known as Microscale Chemistry Laboratory Techniques. It may be mentioned that the present Kit, based on the National Curriculum Framework (NCF), highlights the use of microscale chemistry techniques in schools.

We have developed special apparatus, which limits the volume of liquid reagent and the quantity of the solid chemicals used without compromising on the observation skills, chemical principles involved, accuracy, etc. Moreover, doing chemistry experiments this way saves costly heating system based on gas burners in traditional laboratories.

Volumetric methods have been simplified and physical chemistry experiments could now be done using this Kit, which, otherwise, are hardly done in schools. Conventional laboratory wares are replaced by items based on microscale chemistry.

We hope this Kit will be useful for students, teachers and schools. Feedback for improving this Kit is welcome.

New Delhi
October 2017

R.K. Parashar
Professor
Division of Educational Kits

DEVELOPMENT TEAM

Akhileshwar Mishra, *Head Master*, DMS, Bhubaneswar.

Alka Malhotra, *Professor*, DESM, NCERT, New Delhi.

Anjni Koul, *Professor*, DESM, NCERT, New Delhi.

Brahm Prakash, *Professor (Retd.)*, DESM, NCERT, New Delhi.

K.K. Arora, *Associate Professor*, Zakir Hussain College, New Delhi.

L.K. Tiwary, *Associate Professor*, DESM, RIE-Bhopal, Madhya Pradesh.

Pramila Tanwar, *Assistant Professor*, DESM, NCERT, New Delhi.

R.B. Pareek, *Assistant Professor*, DESM, RIE-Ajmer, Rajasthan.

R.S. Sindhu, *Professor (Retd.)*, DESM, NCERT, New Delhi.

Ruchi Verma, *Associate Professor*, DESM, NCERT, New Delhi.

V.P. Singh, *Professor*, DESM, RIE-Ajmer, Rajasthan.

Vijay Sarda, *Associate Professor (Retd.)*, Zakir Hussain College, New Delhi.

MEMBER AND COORDINATOR

R.K. Parashar, *Professor*, DEK, NCERT, New Delhi.

ACKNOWLEDGEMENTS

The Council acknowledges the contribution of H.O. Gupta, *former Head* of NIE workshop, under whose supervision the Microscale Chemistry Laboratory Kit was developed. The Kit has now been modified as per the feedback received from users. Thanks are due to the following subject experts for carrying out the experiments and enriching the Kit and its manual.

Tarun Vir Rathore, *PGT*, SBV, Timarpur, Delhi; Amina Ansari, *PGT*, Sardar Patel Vidyalaya, New Delhi; S.K. Arora, *Associate Professor*, Government College, Ajmer; Manisha Jain, *Associate Professor*, Acharya Narendra Dev College, New Delhi; Seema Gupta, *Associate Professor*, Acharya Narendra Dev College, New Delhi; R.K. Upadhyay, *Associate Professor*, Government College, Ajmer; S.P. Kamat, *Professor (Retd.)*, Goa University, Goa; Savia P. Torres, *Assistant Professor*, Carmel College for Women, Nuvem, Goa; Sarbjeet Sachdeva, *Professor*, St. Columbas School, New Delhi; A. Mishra, *Head Master*, D.M. School, RIE- Bhubaneswar; S.P. Singh, *Associate Professor*, A.N. College, Patna; N.V.S. Naidu, *Professor*, S.V. University, Tirupati; Swastika Sharma, *PGT*, DAV Public School, Vasant Kunj, Delhi; Sameer Vyas, *Assistant Research Officer*, CSMRS, New Delhi; P. Tamil Salvan, *Associate Professor*, RIE-Mysuru; Archana Rani, *Associate Professor*, Delhi Technical University; New Delhi.

The Council acknowledges the contribution of DEK staff, V.B. Patil, *Technical Officer*, Anil Nayal, *Draftsman* for making illustrations, Satish Kumar, Komal Vashistha, *JPF*, Nivedita, *JPF*, and Nargis Islam, *DTP Operator*.

CONTENTS

| | | |
|--|--|--------------|
| Preface | | <i>iii</i> |
| Introduction to Microscale Chemistry Laboratory Kit | | 1–16 |
| CHAPTER 1 | Characterisation and Purification of Chemical Substances | 17–22 |
| Experiment 1.1 | To determine the boiling point of an organic compound | 17–18 |
| Experiment 1.2 | To determine the melting point of an organic compound | 19–20 |
| Experiment 1.3 | To purify the given compound by crystallisation | 21–22 |
| CHAPTER 2 | Volumetric Analysis | 23–44 |
| Experiment 2.1 | To prepare a standard solution of 0.1 M Oxalic acid | 23–24 |
| Experiment 2.2 | To determine the strength of a given solution of Sodium hydroxide by titrating it against a standard solution of Oxalic acid | 25–28 |
| Experiment 2.3 | To prepare a standard solution of 0.05 M Na_2CO_3 | 29–30 |
| Experiment 2.4 | To determine the strength of a given solution of Hydrochloric acid by titrating it against a standard Na_2CO_3 solution | 31–34 |
| Experiment 2.5 | To determine the strength/ molarity of KMnO_4 solution by titrating it against a 0.05 M standard solution of Oxalic acid | 35–39 |
| Experiment 2.6 | To determine the strength/ molarity of KMnO_4 solution by titrating it against a standard solution of Ferrous ammonium sulphate (Mohr's salt) | 40–44 |

| | | |
|------------------|--|--------------|
| CHAPTER 3 | Qualitative Analysis (Inorganic) | 45–67 |
| Experiment 3.1 | To detect one cation and one anion | 45–67 |
| CHAPTER 4 | Qualitative Analysis (Organic) | 68–77 |
| Experiment 4.1 | To detect extra elements in the given organic compound | 68–70 |
| Experiment 4.2 | To identify one functional group present in an organic compound from the following: | 71–74 |
| | 1. Unsaturation | 71 |
| | 2. Alcoholic | 72 |
| | 3. Phenolic | 72 |
| | 4. Aldehydic and Ketonic | 72 |
| | 5. Carboxylic acid | 73 |
| | 6. Amino group (primary) | 73 |
| Experiment 4.3 | To characterise carbohydrates, fats and proteins in pure form and detect their presence in the given food stuff | 75–77 |
| CHAPTER 5 | Chromatography | 78–84 |
| Experiment 5.1 | To study the separation of pigments from extracts of leaves and flowers by paper chromatography and determination of their R_f values | 81–82 |
| Experiment 5.2 | To separate the constituents present in an inorganic mixture containing two cations only (Pb^{2+} and Cd^{2+}) using the technique of paper chromatography | 83–84 |
| CHAPTER 6 | Preparation of Compounds | 85–97 |
| Experiment 6.1 | To prepare double salts: (i) Potash alum (ii) Ferrous ammonium sulphate (Mohr's salt) | 85–86 |

| | | |
|------------------|--|----------------|
| Experiment 6.2 | To prepare Potassium trioxalatoferrate (III) | 87 |
| Experiment 6.3 | To prepare Acetanilide | 88–89 |
| Experiment 6.4 | To prepare Dibenzalacetone (Dibenzylidene acetone) | 90–91 |
| Experiment 6.5 | To prepare <i>p</i> -nitroacetanilide | 92–93 |
| Experiment 6.6 | To prepare Phenyl-azo- β -naphthol (an azo dye) | 94–95 |
| Experiment 6.7 | To prepare Iodoform | 96–97 |
| CHAPTER 7 | Chemical Equilibrium | 98–102 |
| Experiment 7.1 | To study the pH change by common ion effect in case of weak acids and bases | 98–99 |
| Experiment 7.2 | To study the shift in equilibrium between the chemical systems by either increasing the concentration of ions or the temperature | 100–102 |
| CHAPTER 8 | Surface Chemistry | 103–109 |
| Experiment 8.1 | To prepare lyophilic and lyophobic sols | 104–105 |
| Experiment 8.2 | To purify prepared sol by dialysis | 106 |
| Experiment 8.3 | To study the role of emulsifying agents in stabilising the emulsions of different oils | 107–109 |
| CHAPTER 9 | Chemical Kinetics | 110–117 |
| Experiment 9.1 | To study the effect of change in concentration on the rate of reaction between Sodium thiosulphate and Hydrochloric acid | 111–112 |
| Experiment 9.2 | To study the effect of temperature on the rate of the reaction between Sodium thiosulphate and Hydrochloric acid. | 113 |

| | | |
|-------------------|--|------------------|
| Experiment 9.3 | To study the effect of variation of concentration on the rate of reaction of Iodide ions with Hydrogen peroxide at room temperature (clock reaction) | 114–115 |
| Experiment 9.4 | To study the rate of reaction between Potassium iodate (KIO_3) and Sodium sulphite (Na_2SO_3) using starch solution as indicator | 116–117 |
| CHAPTER 10 | Thermochemistry | 118 – 124 |
| Experiment 10.1 | To determine the enthalpy of the solution of Potassium nitrate (1:200 molar ratio) | 120–122 |
| Experiment 10.2 | To determine the enthalpy of neutralisation of HCl and NaOH | 123–124 |
| CHAPTER 11 | Electrochemistry | 125 – 128 |
| Experiment 11.1 | To study the variation of cell potential of the cell $\text{Zn} \text{Zn}^{2+} \text{Cu}^{2+} \text{Cu}$ with change in the concentration of electrolytes at room temperature | 125–128 |
| APPENDIX | | 129 – 134 |

INTRODUCTION TO MICROSCALE CHEMISTRY LABORATORY KIT

The crucial role of practical work and experimentation in science curriculum is universally accepted. This is more so in chemistry, which is an experimental science. Most of the concepts in this area are better understood by doing practical work.

The aims and objectives of practical work in the laboratory can be stated as follows:

- better understanding of scientific concepts and principles.
- promotion of basic skills and competencies (procedural and manipulative skills, observational skills, drawing skills, reporting and interpretation skills).
- awakening and maintaining curiosity in the learning environment.

Laboratory work is an indispensable part of chemistry instructions. Despite several laudable efforts in the past, experiments, by and large, have continued to be marginalised in school education and have not received due importance. There are several factors which contribute towards the neglect of laboratory work. In particular, there is a crunch of financial resources for developing a separate conventional chemistry laboratory. The increasing cost of chemicals, handling a large number of students, no time to maintain equipment and accessories, no money to replace breakages, problems in the disposal of waste, handling toxic chemicals and working in shifts are some other major contributing factors.

In order to overcome the above problems, the desired modifications may be:

- change in facilities.
- modifications in laboratory programmes.
- optimisation in the use of resources.

- adaptation of cost-effective, safe and efficient techniques. a move towards 'Green Chemistry'.

MANUAL OF MICROSCALE CHEMISTRY LABORATORY (MCL) KIT

Alternative ideas to replace the conventional laboratory, while meeting the objectives of practical work, are being thought of. Recent developments include microscale chemistry experiments using small quantity of chemicals and simple equipment. The concept was first introduced by D.W. Mayo, S.S. Butcher, et al. at Bowdoin College in Brunswick about 30 years ago. In India, this concept has been developed and introduced in the school system by the NCERT with some innovative laboratory apparatus in the form of a kit known as Microscale Chemistry Laboratory Kit.

I. WHAT IS MICROSCALE CHEMISTRY LABORATORY (MCL) KIT?

The Microscale Chemistry Laboratory Kit enables students to perform experiments in an environmentally safe and pollution-free atmosphere, using small quantities of chemicals without compromising with the quality and standard of experiments. The experiments can be performed easily and quickly. They are safer and less polluting. The bigger size reagent bottles, glassware, plasticware, etc., in a conventional laboratory are being replaced by their smaller size counterparts. In short, this method is cost-effective, student, environment, administrator and teacher-friendly.

II. ADVANTAGES OF USING THE KIT

The MCL Kit uses solid chemicals in quantities of a few milligrams and liquid chemicals in few drops using special low-cost apparatus.

1. STUDENT-FRIENDLY

- The use of chemicals in small quantities reduces fumes and risk of accidents, acid burns, etc.
- The experiments are quick to perform, thus, saving time for performing more experiments.
- It develops the habit of conservation.

2. ENVIRONMENT-FRIENDLY

- It reduces use of chemicals resulting in waste reduction at the source.
- It offers vastly enhanced laboratory safety by improving the laboratory air quality through reduction in:
 - exposure to toxic chemicals.
 - fire and explosion hazards.
 - spills and accidents.

3. ADMINISTRATOR-FRIENDLY

- It sharply reduces laboratory cost.
- It lowers glass breakage cost.
- It saves storage space.

4. TEACHER-FRIENDLY

- It promotes better student discipline in the laboratory.
- It is pedagogically sound. Teachers can ask the students to do more experiments during the saved time to help in better conceptual understanding.

III. WHAT SPECIAL APPARATUS ARE PROVIDED IN THE KIT?

Following is a list of some special apparatus provided:

1. Small portable box for easy transportation of apparatus, and their storage.
2. Polyethylene dispensing bottles (squeeze type) to dispense liquid chemicals dropwise avoiding the contamination of chemicals.
3. Well plates for performing tests.
4. W-tubes for generating gases in small quantities and studying their properties.
5. Barrel pipettes.
6. Miniature glassware for the minimum consumption of chemicals.
7. Micro filtration unit.
8. Micro spatulas.

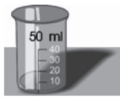



IV. COMPARISON OF MICROSCALE CHEMISTRY LABORATORY KIT AND TRADITIONAL CHEMISTRY LABORATORY





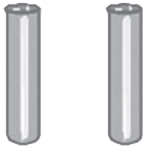

| S. No. | Traditional Chemistry Laboratory | Microscale Chemistry Laboratory |
|--------|---|--|
| 1. | Quantity of Chemicals Used: A few grams of solids and a few millilitre of solutions are used. | A few milligrams of solids and a few drops of solutions are used. A typical microscale experiment will consume only, say, less than 1% of the required quantity of reagents used in a corresponding traditional macro scale experiment. |
| 2. | Environment Protection: In a traditional chemistry laboratory, a large quantity of chemicals is drained. Also, a large quantity of toxic fumes causes air pollution. | In a microscale chemistry lab, the use of a sink may not be necessary. The drainage of chemicals is minimised. Toxic fumes are almost non-existent. |
| 3. | Accidental Hazards: Many accidents occur due to fire, acid burns, etc. | Flame intensity is minimised. Acid burns are almost non-existent. The risks of fire hazards and acid burns are reduced to minimum. |
| 4. | Breakage of Glassware: Possibility of breakage of glassware is more. | Breakage is minimised. |
| 5. | Time taken for an Experiment: A lot of time is required for a particular experiment. | Less time is required for an experiment. So, more experiments can be done in the allotted time. |
| 6. | Cost: More costly. | There is a lot of cost reduction. |


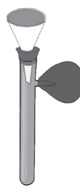
| | | |
|----|--|--|
| 7. | Space: A regular laboratory requires a lot of space for infrastructure, such as table racks, gas line, storage of glassware and chemicals, etc. | Only a small room with tables is sufficient. The storage space is drastically reduced. |
| 8. | Habit of Conservation: Students develop the habit of wasting a lot of water and chemicals. | Students develop the habit of conserving chemicals and water by minimising their use. |


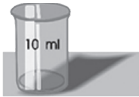


V. DESCRIPTION OF KIT ITEMS








(a) APPARATUS


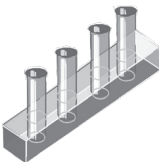


| S. No. | Items (Quantity/Kit) | Figure/ Setup | Uses |
|--------|---|---|--|
| 1. | Kit manual | | It contains information about all experiments. |
| 2. | Kit box (made of HDPE with attachable wheels at the base and has a lockable lid). | | It contains all kit items. |
| 3. | Graduated beaker- 50 mL (3) |  | Measuring the volume of liquids/ keeping the liquids. |
| 4. | Boiling tube (90 mm×20 mm) (borosilicate glass) (4) |  | For boiling of liquids. |
| 5. | Capillary tube (10 cm) (1 box) |  | Used for melting point and boiling point determination. |
| 6. | China dish (diameter: 8.5 cm) (4) |  | Used to heat small quantities to very high temperatures. Used as a water bath. |

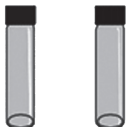

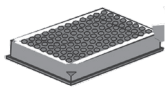

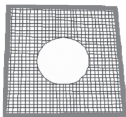


| | | | |
|-----|--|---|--|
| 7. | Conical flask Capacity: 25mL, 50mL, 100mL (1 each) |  | For multipurpose usage, including titrations. |
| 8. | Dispensing bottle 15mL (approx.) capacity (30) |  | Used to keep chemical reagents. Open the cap and squeeze the bottle to dispense one or two drops. Label the dispensing bottle. |
| 9. | Graduated dropper/pasteur pipette (made of polypropylene), LDPE Bulb: 4 cm Capacity: 3mL (20) |  | Used to transfer liquids. |
| 10. | Glass dropper with rubber bulb Glass tube length: 10.5 cm (4) |  | <ul style="list-style-type: none"> i. The dropper is used for transferring small amount of a liquid from one container to another. ii. Fix the rubber bulb to a small glass dropper. |
| 11. | Ignition tube/ fusion tube Standard size (20) |  | <ul style="list-style-type: none"> i. To determine the boiling point of a substance. ii. Preparation of Lassaigne's Extract. |
| 12. | Glass rod 15 cm length 0.5 cm diameter (4) |  | <ul style="list-style-type: none"> i. Used as a stirrer. ii. Used to pour liquid on to the filter paper during filtration. |



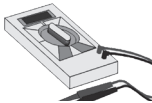



| | | | |
|-----|--|---|--|
| 13. | Micro funnel Diameter: 32 mm Overall size 80 mm (4) |  | Used for filtration. |
| 14. | Micro filtration unit (4) Borosilicate glass boiling tube, length 80 mm; dia 20 mm, having side tube for fitting smaller bulb, small rubber bulb, rubber cork fitted with small Hirsch funnel as per sample. |  | <p>Steps for use:</p> <ol style="list-style-type: none"> Bore the rubber cork to fit the stem of Hirsch funnel. Cut the filter paper to fit the mesh of the funnel. The size should exactly fit the mesh, neither big nor small. Fit the cork with funnel in the mouth of the boiling tube (having a side tube). Transfer the solution to be filtrated to the funnel. Press the bulb with hand to evacuate it and place it on the side tube of the boiling tube while keeping it pressed. Release the pressure. The bulb will suck the air inside the boiling tube allowing fast filtration. |




| | | | |
|-----|---|--|---|
| 15. | Micro test tube Borosilicate glass, length 45 mm, dia 10 mm with rim (2 mL) (24) |  | As a container for keeping and testing small quantities of liquids. |
| 16. | Micro beaker Borosilicate glass, height 34 mm, dia, 25 mm (10 mL) (12) |  | As a container for storing liquids. |
| 17. | Micro measuring cylinder (10 mL) (4) |  | To measure the volume of a liquid up to 10 mL. |
| 18. | Micro burette Borosilicate glass, rotating screw type flow stop cock with PTFE key and cup least count 0.5 mL (5 mL) (8) |  | <ul style="list-style-type: none"> i. The micro burette has 5 mL capacity with the least count of 0.05 mL. At the narrow end of the burette, a micro tip is attached with the help of a plastic tube. ii. Two burettes are used to avoid the use of a pipette. iii. Pour the liquid slowly and make sure that no air bubble remains inside the burettes. |

| | | | |
|-----|---|---|--|
| 19. | Micro test tube brush (4) |  | The test tube brush is used to easily clean the micro test tube. |
| 20. | Petri dish soda glass without cover (diameter 10 cm) (4 sets) |  | A shallow glass round dish used for chromatography. |
| 21. | Platinum wire/ Nichrome wire (4) |  | Platinum wire is used for flame test. |
| 22. | Micro spatula (Stainless steel) Flat portion: 4 cm, scoop: 1.3×0.7 cm, length 10 cm (4) |  | Used for transferring small amounts of solid. |
| 23. | Spirit lamp/ Bunsen burner steel body alongwith brass wick holder 6.5 cm dia, height 5 cm (04) |  | Used for heating substances. Spirit lamp can be used in place of a kerosene burner. |
| 24. | Circular whatman filter paper: good quality, dia 125 mm (20) |  | Filter paper is used to separate fine solids from liquids. |
| 25. | Mercury thermometer range -0°C to 250°C Least count -1°C (2) |  | For boiling point and melting point determination. |

| | | | |
|-----|---|---|--|
| 26. | Digital thermometer with button cell. Range -50°C to 300°C , and F selectable display, memory of last measure. Accuracy: 0.1°C . (1) | | Used for thermochemistry experiments. Caution: Do not dip the tip in concentrated solutions of acids and bases. |
| 27. | Tripod stand height 10 cm, cast iron top $7\times 7\times 7$ cm triangle, 10 g, 4 mm, dia iron (4) |  | Used as a stand for keeping apparatus for heating. |
| 28. | Micro test tube acrylic/plastic micro test tube stand 100 mm \times 27mm \times 13mm 4 mm, blind holes micro test tube (4) |  | The test tube rack is used to hold test tubes. |
| 29. | Two-way burette clamp and stand. |  | For base mild steel sheet 140 mm \times 160mm \times 16 SWG. For stand: mild steel rod 6 mm dia, 280 mm length, mild steel nut 6 mm, fly nut 6 mm, 6 mm washer, spring steel strip 80 mm \times 8 mm \times 20 SWG to hold two burettes. |
| 30. | Micro test tube wooden handle to hold 10 mm micro test tubes holders (4) |  | Used to hold test tubes. |

| | | | |
|-----|---|---|---|
| 31. | Vials 5.5 cm×1.5 cm PVC (20) |  | Used as a container to store solid chemicals. |
| 32. | Soda watch glass Diameter: 5 cm (4) |  | i. The watch glass is used to hold solids when being weighed. ii. It should never be heated. |
| 33. | Well plate Polystyrene well plate (4) |  | Flat plate with multiple 'wells' used as micro test tubes with 96 wells. |
| 34. | W-tube Borosilicate glass 2 mm thick × 8 mm dia, length of arm 72–75 mm with small bulb in 1 arm (8) |  | Used for the test, in which a gas has to be passed through a reagent. |
| 35. | Wire gauge with frame 100 mm×100 mm (4) |  | Used to provide uniform heat while heating. |
| 36. | Wash bottle 125 mL (LDPE) (2) |  | Used to contain water to wash the apparatus. |
| 37. | Calorimeter (50 mL Borosilicate glass beaker inserted in LDPE foam case 100×75×130 mm with the lid) (1) |  | Device used for measuring the enthalpy of chemical reactions or physical changes. |

| | | | |
|-----|---|---|--|
| 38. | Electrodes (Cu and Zn) 8 cm × 0.5 cm (4 pairs) |  | Used to generate voltage in an electrochemical reaction. |
| 39. | Emery paper (100 No.) (1) |  | To give a smooth, shiny finish to manufactured products. |
| 40. | Multimeter pocket size digital high impedance as per sample (1) |  | i. To check the continuity of a wire and circuit. ii. To measure resistance, voltage and current. |
| 41. | Parchment/cellophane paper (9 cm×14.5 cm) (100 leaves packet) |  | Used for dialysis. |
| 42. | Glass pipette graduated 5 mL accuracy: 0.05 mL least count Borosilicate glass (4) |  | Used to measure the volume of a solution accurately. |
| 43. | Stirrer (glass) (4) |  | i. Circular end is used to stir a mixture or a solution in a calorimeter. ii. The mounted stirrer is inserted in the foam lid of the calorimeter surrounding the thermometer. |

| | | | |
|-----|--|---|--|
| 44. | Stopwatch as per sample (2) |  | Used when time periods must be measured precisely and with a minimum of complications. In laboratory experiments, like clock reaction. |
| 45. | Thread small roll |  | Used to tie fusion tubes with thermometer m.p/bp |
| 46. | Volumetric flask Borosilicate glass with stopper 25 mL (4) |  | Used to make accurate solution by measuring a specific volume. |
| 47. | Universal indicator 1 paper box containing 10 booklets (2 paper box) | | |
| 48. | Red litmus paper and blue litmus paper (five booklets each) | | |
| 49. | Forecep, SS length 100 mm (04) | | |
| 50. | Plastic box 225×125×85 mm (1) 235×150×75 mm (1) | | To pack glass items. |
| 51. | Chromatography jar | | Soda glass height 200 mm × I. dia 50 mm with rubber cock fitted with hook (1) |

(B) CHEMICALS/REAGENTS

| | |
|---------------------------------------|-------------------------------------|
| 1. Alcohol | 29. Calcium carbide |
| 2. Acetic acid | 30. Carbon disulphide |
| 3. Glacial acetic acid | 31. Calcium oxide |
| 4. Acetaldehyde | 32. Castor oil |
| 5. Acetanilide | 33. Ceric Ammonium nitrate |
| 6. Acetone | 34. Chloroform/Carbon tetrachloride |
| 7. Acetyl chloride | 35. Chlorine water |
| 8. Activated charcoal | 36. Citric acid |
| 9. Alcoholic KOH | 37. Cobalt chloride |
| 10. Aluminium chloride | 38. Cobalt nitrate |
| 11. Aluminium sulphate | 39. Copper turnings |
| 12. Ammonium carbonate | 40. Copper sulphate |
| 13. Ammonium chloride | 41. Dimethyl glyoxime |
| 14. Ammonium sulphate | 42. Disodium hydrogen phosphate |
| 15. Ammonium phosphate | 43. Distilled water |
| 16. Ammonium oxalate | 44. 2, 4-DNP |
| 17. Ammonium molybdate | 45. Egg albumin |
| 18. Ammonium thiocyanate | 46. Ethyl acetate |
| 19. Aniline | 47. Fehling solution A |
| 20. Liquor ammonia | 48. Fehling solution B |
| 21. Aqueous potassium hydroxide | 49. Formaldehyde |
| 22. Arsenic oxide | 50. Ferric chloride |
| 23. Barium chloride | 51. Ferrous sulphate |
| 24. Benedict solution | 52. Hydrogen peroxide |
| 25. Benzene | 53. Hydrogen sulphide |
| 26. Benzaldehyde | 54. Dilute Hydrochloric acid |
| 27. Bromine water | 55. Concentrated Hydrochloric acid |
| 28. Butan-2-one (Ethyl methyl ketone) | 56. Iodine solution |

| | |
|-------------------------------------|--|
| 57. Iron fillings | 86. Phenol |
| 58. Iron sulphide | 87. Potassium iodate |
| 59. Liquid paraffin | 88. Potassium thiocyanate |
| 60. Lead acetate | 89. Potassium chloride |
| 61. Lime water | 90. Potassium chromate |
| 62. Manganese dioxide | 91. Potassium dichromate |
| 63. Machine oil | 92. Rectified spirit |
| 64. Methanol | 93. Resorcinol |
| 65. Mohr's salt | 94. Soap/Detergent |
| 66. Molisch's reagent | 95. Schiff's reagent |
| 67. Methyl orange | 96. Silver nitrate |
| 68. Mustard oil | 97. Sodium acetate |
| 69. Naphthalene | 98. Sodium bicarbonate |
| 70. Nessler's reagent | 99. Sodium bromide |
| 71. Ninhydrin | 100. Sodium chloride |
| 72. Dilute Nitric acid | 101. Sodium carbonate |
| 73. Concentrated Nitric acid | 102. Sodium hydrogen phosphate |
| 74. α -Naphthol (1-Naphthol) | 103. Sodium hypochlorite/ Sodium chlorate |
| 75. β -Naphthol (2-Naphthol) | 104. Sodium hydroxide |
| 76. Oxalic acid | 105. Sodium hydroxide pellets |
| 77. Parchment paper | 106. Sodium metal |
| 78. Phenolphthalein | 107. Sodium nitrite |
| 79. Potassium ferricyanide | 108. Sodium nitroprusside |
| 80. Potassium iodate | 109. Sodium sulphate |
| 81. Potassium nitrite | 110. Sodium sulphite |
| 82. Potassium nitrate | 111. Sodium hydrogen sulphite |
| 83. Potassium ferrocyanide | 112. Sodium sulphide solution |
| 84. Potassium hydroxide | 113. Sodium thiosulphate |
| 85. Potassium hydrogen sulphate | 114. Dilute Sulphuric acid |

| | |
|----------------------------------|---|
| 115. Concentrated Sulphuric acid | 120. Universal indicator solution/paper |
| 116. Stannous chloride | 121. Uranyl Zinc acetate |
| 117. Starch | 122. Zinc granules |
| 118. Tollen's reagent | 123. Zinc dust |
| 119. Urea | 124. Zinc sulphate |

CHAPTER 1

CHARACTERISATION AND PURIFICATION OF CHEMICAL SUBSTANCES

EXPERIMENT 1.1

To determine the boiling point of an organic compound.

APPARATUS REQUIRED

Beaker (50 mL), capillary tube, ignition tube, thermometer, iron stand (with clamp), wire gauze, tripod stand and burner/spirit lamp.

CHEMICALS REQUIRED

Organic liquids, such as Benzene and Ethyl methyl ketone for boiling point (b.p.) determination.

PROCEDURE

1. Take a 50 mL glass beaker and fill it half with water.
2. Take a capillary tube and seal its one end by heating it in the flame.
3. Transfer a few drops of the organic liquid compound in the ignition tube. Dip the open end of capillary tube into the liquid taken in the ignition tube.

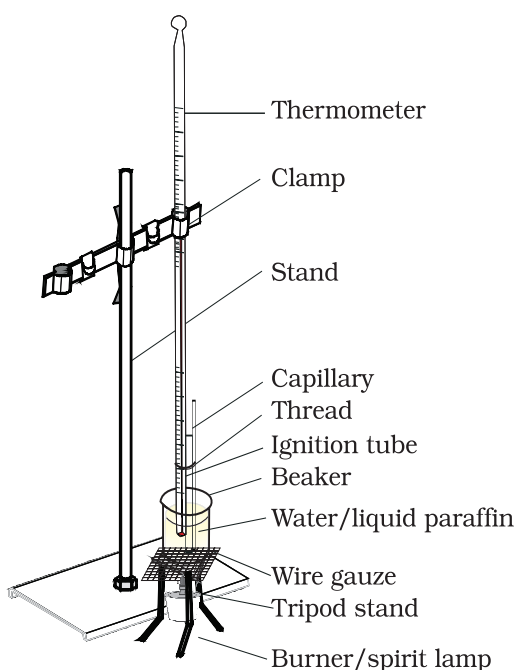


Fig. 1.1: Arrangement showing boiling point determination

4. Tie the ignition tube with the thermometer with the help of a thread in such a way that the lower end of the ignition tube is near the bulb of the thermometer.
5. Keep the 50 mL beaker on a wire gauze kept on a tripod stand. Fix the thermometer and the ignition tube with the help of thread in such a way that the bulb of the thermometer and half of the ignition tube are dipped in water.
6. Heat the beaker gently.
7. Note the temperature as soon as regular stream of bubbles are seen coming out of the liquid in the ignition tube.

RESULT

1. Boiling point of Benzene =°C
Literature value = 78°C
2. Boiling point of Ethyl methyl ketone =°C
Literature value = 80°C

NOTE

If the boiling point of the liquid is more than 95°C, take liquid paraffin in the beaker in place of water.

PRECAUTIONS

Fix the thermometer in the beaker in such a way that its bulb and the ignition tube do not touch the beaker.

EXPERIMENT 1.2

To determine the melting point of an organic compound.

APPARATUS REQUIRED

Beaker (50 mL), capillary tube, thermometer, iron stand (with clamp), tripod stand and burner/spirit lamp.

CHEMICALS REQUIRED

Organic compounds, such as Naphthalene, Benzoic acid, etc.

PROCEDURE

1. Take a capillary tube and seal its one end by heating it in the flame.
2. Take the given organic compound, say Naphthalene, on a tile and grind it into a fine powder.
3. Hold the closed end of the capillary tube between your thumb and finger. Now, insert its open end in the finely powdered organic compound. Gently tap the capillary tube on the table to fill the compound in it to a length of about 1–2 cm. Tie the capillary tube with a thermometer in a way that its lower end is at the same level as that of the bulb of the thermometer.
4. Take a 50 mL beaker, fill it half with water and put it on wire gauze kept on a tripod stand.

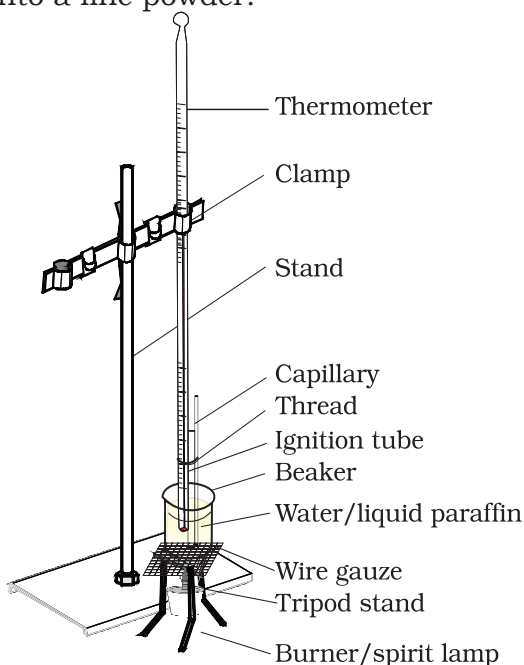


Fig. 1.2: Arrangement showing melting point determination

5. Clamp the thermometer with the capillary tube in the water taken in the beaker in a way that the bulb of the thermometer and the portion of the capillary tube, containing the organic compound, are dipped in the water. Heat the beaker gently.
6. Note the temperature as soon as the substance begins to melt.

RESULT

Melting point of the compound is°C.

[Literature value, melting point (m.p.) of Benzoic acid = 121°C, m.p. of Naphthalene = 80°C]

PRECAUTIONS

1. The compound should be packed tightly in the capillary tube.
2. Fix the thermometer in the beaker in such a way that its bulb and the capillary tube do not touch the beaker.

NOTE

Use liquid paraffin in the beaker in place of water, if the m.p. of the compound is more than 95°C.

EXPERIMENT 1.3

To purify the given compound by crystallisation.

APPARATUS REQUIRED

Two beakers (50 mL), watch glass, filtration unit and burner / spirit lamp.

CHEMICALS REQUIRED

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and dilute H_2SO_4

PROCEDURE

1. Prepare a clear solution of crude Copper sulphate by dissolving 1 g in about 10 mL of water. Add 1–2 drops of dilute H_2SO_4 to it.
2. Heat the solution till the saturation point is reached. Check it by dipping one end of a glass rod in the hot solution. Take it out and blow air on this end from your mouth. Small crystals should appear. If not, further heat the solution.
3. Filter the hot solution through cotton wool fixed in a micro funnel.
4. Transfer the solution from the filtration unit to another beaker. Allow it to cool for hours, crystals will appear.

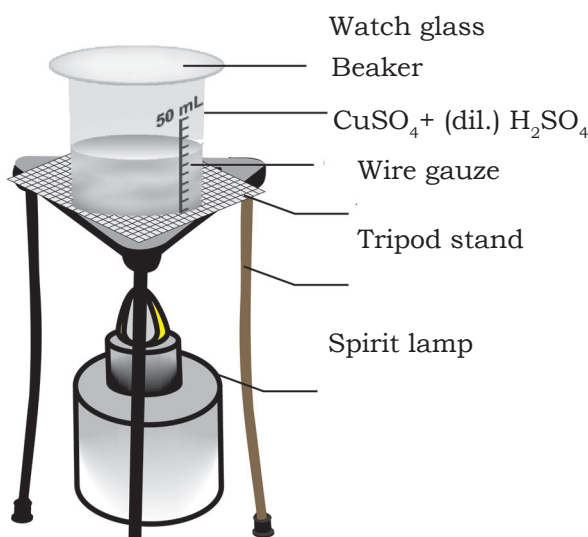


Fig. 1.3: Arrangement showing the preparation of Copper sulphate crystals

5. Filter, wash with little water and dry the crystals.
6. Weigh the crystals and report the yield.

RESULT

The mass of crystals obtained is g.

NOTE

1. H_2SO_4 is added to the Sulphate solution to prevent hydrolysis.
2. Crystallisation of alum, Benzoic acid and Iron sulphate can also be done by following the above procedure. The amount of substances required is as follows:
 - (a) For the purification of Ferrous sulphate, take about 0.5 g sample of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ + three drops of dilute H_2SO_4 + 3 mL H_2O .
 - (b) For the purification of Alum, take about 1 g Alum sample + two drops of dilute H_2SO_4 + 5 mL H_2O .
 - (c) For the purification of Benzoic acid, take about 0.5 g of Benzoic acid + 8 mL H_2O .

CHAPTER 2

VOLUMETRIC ANALYSIS

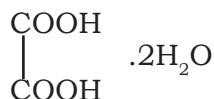
EXPERIMENT 2.1

To prepare a standard solution of 0.1 M Oxalic acid.

THEORY

PRIMARY AND SECONDARY STANDARDS

A standard solution is one whose concentration is known accurately. A standard solution of an acid/base is used to determine the unknown concentration of a solution of base/acid by volumetric analysis. There are various ways of expressing the concentration of a standard solution. The concentration of a standard solution is usually expressed in moles per litre (molarity). For example, a standard solution of Oxalic acid can be used to determine the unknown concentration of an alkali solution. The formula of hydrated crystalline Oxalic acid is:



and its molar mass is 126 g mol^{-1} . If 126 g of Oxalic acid is present in one litre of a solution, it is known as one molar (1.0 M) solution.

For preparing 10 mL of $\frac{\text{M}}{20}$ Oxalic acid solution, we require:

$$\frac{126\text{g} \times 10\text{mL}}{20 \times 1000} = 0.0063 \text{ g of hydrated Oxalic acid.}$$

APPARATUS REQUIRED

Volumetric flask (10 mL), funnel, watch glass, wash bottle, balance and spatula.

CHEMICALS REQUIRED

Oxalic acid $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$

PROCEDURE

1. Weigh a clean and dry watch glass accurately.
2. Weigh 0.063 g Oxalic acid on the foil.
3. Transfer Oxalic acid carefully from the watch glass into the clean volumetric flask using a funnel. Wash the aluminium foil and the funnel several times with distilled water. While washing the funnel, water should be added in small amounts.
4. Swirl the volumetric flask till the solid Oxalic acid dissolves completely. Make the volume up to the mark on the volumetric flask with distilled water.
5. Remove the funnel from the mouth of the volumetric flask. Close the mouth of the flask with a stopper tightly and shake the contents thoroughly to make a solution.

EXPERIMENT 2.2

To determine the strength of a given solution of Sodium hydroxide by titrating it against a standard solution of Oxalic acid.

THEORY

In the titration of Oxalic acid (weak acid) against Sodium hydroxide (strong base), the following reaction takes place:



In this titration, Phenolphthalein is used as an indicator. The concentration of an unknown solution is calculated in g/L. The molarity of the solution can be calculated by using the formula

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where a_1 , M_1 , V_1 are respectively the basicity, molarity and volume of Oxalic acid used, and a_2 , M_2 and V_2 are the acidity, molarity and volume of NaOH, respectively, used in the titration.

The basicity of an acid is the number of replaceable H^+ ions and acidity of a base is the number of replaceable OH^- ions from one formula unit of the substance.

APPARATUS REQUIRED

Two burettes (5 mL), conical flask, beaker (10 mL), glazed tile, two-way clamp and standard volumetric flask (10 mL).

CHEMICALS REQUIRED

Standard solution of 0.05 M Oxalic acid, 0.10 M Sodium hydroxide (approximately) and Phenolphthalein.

PROCEDURE

1. Take two 5 mL burettes and wash them thoroughly with distilled water.
2. Rinse and fill the first burette with 0.05 M Oxalic acid solution. Rinse and fill the second burette with 0.10 M Sodium hydroxide solution.

3. Remove air bubbles, if any, from both the burettes by running the solution.
4. Adjust the level at zero mark.
5. Now, take 2 mL of Oxalic acid solution (V_1) from the first burette in a micro conical flask (titration flask). Add 1–2 drops of Phenolphthalein indicator to it.
6. Now, add NaOH solution dropwise from the second burette to the titration flask with constant swirling.
7. Continue this process till a permanent pink colour appears due to excess NaOH (Phenolphthalein becomes pink in alkaline medium and colourless in acidic medium). Record this reading as V_2 in the table.
8. Add another 0.5 mL of Oxalic acid solution from the first burette to the titration flask without adding any indicator. Record 2.5 mL as V_1 in Table 2.1 for the second reading. The solution becomes colourless.
9. Titrate it again with the NaOH solution. Record the final volume as V_2 in the table for the second reading.
10. Take 5–6 readings by adding Oxalic acid solution in increments of 0.5 mL and titrating it against the NaOH solution.

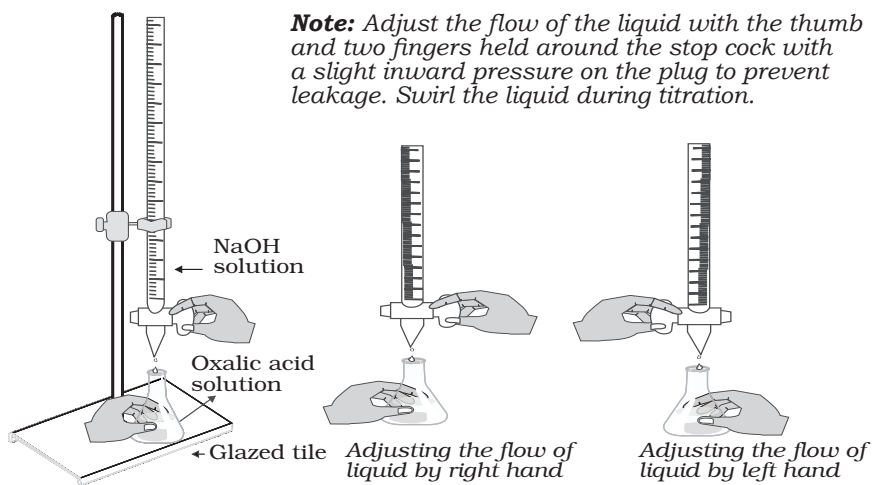


Fig. 2.1: Titrating the solution

Table 2.1

| S. No. | Volume of Oxalic acid in mL (V_1) | Volume of NaOH in mL (V_2) | Molarity of NaOH $M_2 = \frac{a_1 M_1 V_1}{a_2 V_2}$ |
|--------|---------------------------------------|--------------------------------|---|
| 1. | 2.0 | | |
| 2. | 2.5 | | |
| 3. | 3.0 | | |
| 4. | 3.5 | | |
| 5. | 4.0 | | |
| 6. | 4.5 | | |
| 7. | 5.0 | | |

CALCULATIONS

$$\begin{array}{ccc} a_1 M_1 V_1 & = & a_2 M_2 V_2 \\ \text{(Oxalic acid)} & & \text{(Sodium hydroxide)} \end{array}$$

where, M_1 and V_1 are the molarity and the volume of the Oxalic acid solution, respectively. M_2 and V_2 are the molarity and the volume of the Sodium hydroxide solution, respectively. a_1 and a_2 are the basicity of Oxalic acid and the acidity of Sodium hydroxide, respectively. In this case, $a_1 = 2$ and $a_2 = 1$.

Calculate the concentration of Sodium hydroxide solution in g/L by using the equation given below.

$$\text{Concentration (strength)} = (\text{Molarity} \times \text{Molar mass}) \text{ g/L}$$

ALTERNATIVE METHOD OF CALCULATION

Plot a graph of volume of NaOH added against the volume of Oxalic acid taken. It will be a straight line.

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

or

$$M_2 = \frac{a_1 M_1}{a_2} \times \frac{V_1}{V_2}$$

Since, V_1/V_2 is known from the graph (Fig. 2.2), M_2 can be calculated. The results obtained by the graphical method are

more accurate than those obtained by the earlier average calculation method.

$$\frac{v_1}{v_2} = \frac{y_2 - y_1}{x_2 - x_1}$$

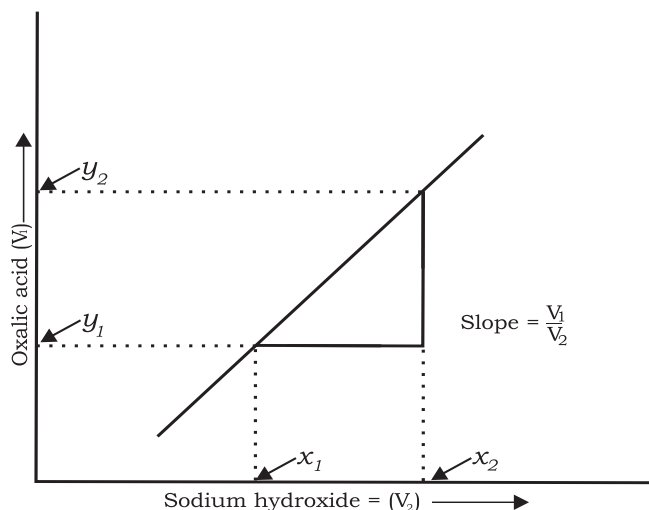


Fig. 2.2

RESULT

The strength of the given NaOH solution is.....g L⁻¹.

PRECAUTIONS

1. Always rinse the burette with the solution to be taken in it.
2. Always read the lower meniscus for all colourless solutions and the upper meniscus for all coloured solutions.
3. Place the eye parallel to the meniscus to note the burette readings.
4. The concentration (strength) of the solution must be calculated up to two places of decimal.
5. Remove air bubbles, if any, from the burette by running the solution.
6. Never rinse the conical flask with the solution to be taken in it for titration, otherwise the volume of the solution taken gets changed.

EXPERIMENT 2.3

To prepare a standard solution of 0.05 M Na_2CO_3 .

THEORY

Sodium carbonate is a primary standard. Therefore, its standard solution can be prepared by direct weighing and dissolving the desired volume of water.

Molar mass of anhydrous $\text{Na}_2\text{CO}_3 = 106 \text{ g mol}^{-1}$

$$\text{Molarity} = \frac{\text{Mass}}{\text{Molar Mass}} \times \frac{1000}{\text{Volume (mL)}}$$

The mass of anhydrous Na_2CO_3 required for the preparation of 10 mL of \underline{M} solution is

$$= \frac{106 \times 10}{20 \times 1000} = 0.053 \text{ g}$$

APPARATUS REQUIRED

10 mL measuring flask (volumetric flask), watch glass, wash bottle and balance.

CHEMICALS REQUIRED

Anhydrous Sodium carbonate (Na_2CO_3)

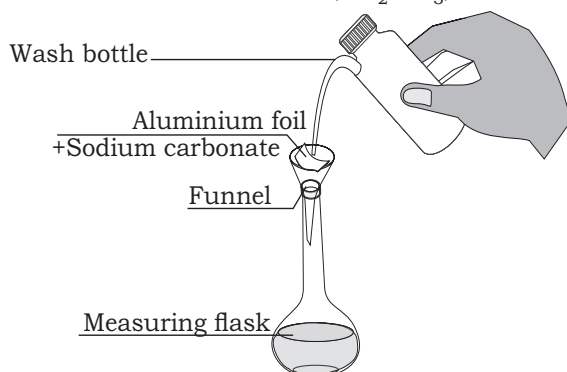


Fig. 2.3: Preparation of standard solution of Sodium carbonate

PROCEDURE

1. Weigh a clean and dry watch glass accurately.
2. Weigh 0.053 g of Na_2CO_3 by using a watch glass.
3. Transfer Sodium carbonate carefully from the watch glass

into a clean and dry measuring flask using a funnel. Wash the watch glass and the funnel thoroughly with distilled water. While washing the funnel, water should be added in small proportions.

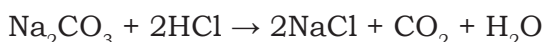
4. Swirl the measuring flask till the solid Sodium carbonate dissolves completely. Make up the volume to the mark on the measuring flask with distilled water.
5. Remove the funnel from the mouth of the measuring flask. Close the flask tightly with the stopper and shake the contents thoroughly to make the solution.

EXPERIMENT 2.4

To determine the strength of a given solution of Hydrochloric acid by titrating it against a standard Na_2CO_3 solution.

THEORY

The following reaction takes place in this titration:



Methyl orange is used as an indicator in this titration. The proton furnished by the acid neutralises Sodium carbonate solution. When the entire Sodium carbonate solution is neutralised, the next drop of the acid added produces the reddish orange colour, which is the end point. The concentration (strength) of the unknown solution is calculated in g/L. It is calculated from the molarity of the solution using the relation:

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where, a_1 and a_2 are the acidity of the base and the basicity of the acid, respectively. M_1 and M_2 are the molarities, V_1 and V_2 are the volumes of the base and the acid, respectively, used to neutralise each other.

APPARATUS REQUIRED

Two burettes (5 mL), two conical flasks, glazed tile and standard measuring flask/volumetric flask (10 mL).

CHEMICALS REQUIRED

0.05 M standard solution of Sodium carbonate, 0.1 M HCl solution (approximately) and Methyl orange solution.

PROCEDURE

1. Wash the burettes with distilled water.
2. Rinse and fill the first burette with the standard solution of Sodium carbonate. Adjust the level exactly at zero mark. Take 2 mL (V_1) of this solution in a conical flask (titration flask) and add 2–3 drops of Methyl orange indicator to it.
3. Rinse and fill the second burette with the Hydrochloric acid solution. Adjust the level exactly at zero mark.

4. Add HCl solution dropwise to the titration flask from the burette with constant swirling.
5. Continue this process till the colour changes to reddish orange. Record this reading as (V_2) in the table.
6. Add another 0.5 mL of Na_2CO_3 solution from the first burette to the titration flask without adding any indicator. (Record 2.5 mL as V_1 in table). The solution becomes yellow.
7. Titrate it against the HCl solution from the burette as earlier. Record the final volume as V_2 in the table.
8. Take 5–6 readings by adding Sodium carbonate solution in increments of 0.5 mL and titrating it against the HCl solution.

Table 2.2
Titration of Hydrochloric acid against Sodium carbonate (Na_2CO_3) Solution

| S. No. | Volume of Sodium carbonate in mL (V_1) | Volume of HCl in mL (V_2) | Molarity of HCl $M_2 = \frac{a_1 V_1 M_1}{a_2 V_2}$ |
|--------|--|-------------------------------|--|
| 1. | 2.0 | | |
| 2. | 2.5 | | |
| 3. | 3.0 | | |
| 4. | 3.5 | | |
| 5. | 4.0 | | |
| 6. | 4.5 | | |
| 7. | 5.0 | | |

CALCULATIONS

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

(Sodium carbonate) (Hydrochloric acid)

where, M_1 and V_1 are the molarity and the volume of the Sodium carbonate solution, respectively. M_2 and V_2 are the molarity and the volume of the Hydrochloric acid solution, respectively.

a_1 and a_2 are, respectively, the acidity of Sodium carbonate and the basicity of Hydrochloric acid. In this case, $a_1 = 2$ and $a_2 = 1$. Also, the molar mass of Sodium carbonate, $\text{Na}_2\text{CO}_3 = 106 \text{ g mol}^{-1}$ and the molar mass of Hydrochloric acid (HCl) = 36.5 g mol^{-1} . Calculate the average molarity. Calculate the strength of Hydrochloric acid solution in g/L by using the equation as given below.
 Strength = Molarity \times Molar mass g/L

ALTERNATIVE METHOD OF CALCULATION

Plot a graph of the volume of Hydrochloric acid added against the volume of Sodium carbonate taken. It will be a straight line.

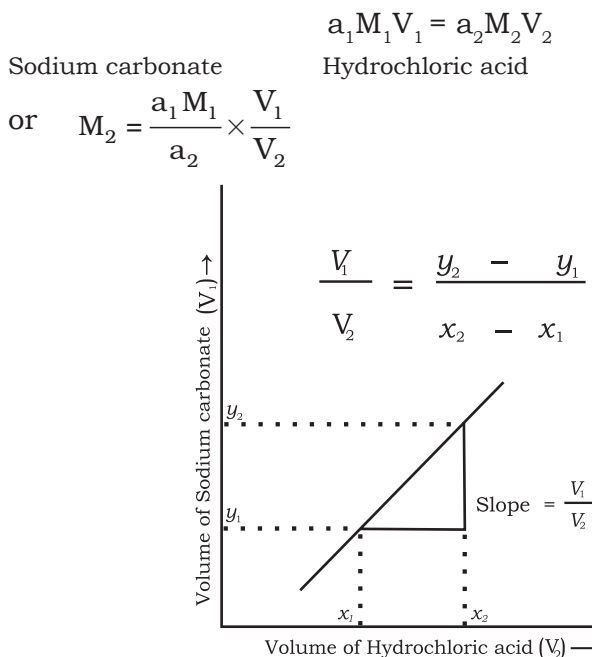


Fig. 2.4

Since V_1/V_2 is known by the graph (Fig. 2.4.), M_2 can be calculated. The results obtained by the graphical method are more accurate than those obtained by the earlier average calculation method.

RESULT

The strength of the given Hydrochloric acid solution is..... g L⁻¹.

PRECAUTIONS

1. Always rinse the burette with the solution which is to be taken in it.
2. Always read the lower meniscus for all colourless solutions.
3. Place the eye parallel to the meniscus to note the burette readings.
4. The strength of the solution must be calculated up to two places of decimal.
5. Remove air bubbles, if any, from the burette by running the solution.
6. Never rinse the conical flask with the solution to be taken in it for titration, otherwise the volume of the solution taken gets changed.

REDOX TITRATIONS

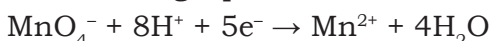
The oxidation and reduction reactions involve the transfer of electrons from one species to another. In the oxidation of a substance, electrons are lost from the species and in reduction, electrons are gained by the species. Oxidation and reduction reactions occur simultaneously, and therefore, they are known as 'redox reactions'. Titrations involving redox reactions are called 'redox titrations'. You know that in acid-base titrations, indicators which are sensitive to pH change are used to note the end point. Similarly, in redox titrations, there is a change in the electrode potential of the system. The indicators used in redox reactions are sensitive to change in electrode potential. The ideal oxidation-reduction indicators have an electrode potential intermediate between the values for the solution being titrated and the titrant. These values show sharp readily detectable colour change.

EXPERIMENT 2.5

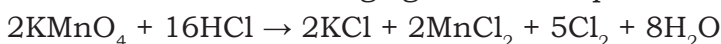
To determine the strength/molarity of KMnO_4 solution by titrating it against a 0.05 M standard solution of Oxalic acid.

THEORY

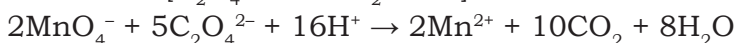
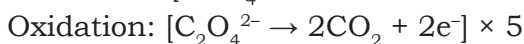
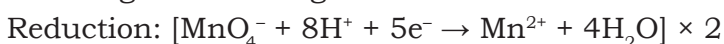
In the present experiment, Potassium permanganate acts as a powerful oxidising agent in acidic medium. The oxidising action of KMnO_4 in the acidic medium can be represented by the following equation:



The acid used in this titration is dilute Sulphuric acid. Nitric acid is not used as it is an oxidising agent and Hydrochloric acid is usually avoided because it reacts with KMnO_4 , according to the equation given below to produce Chlorine, which is also an oxidising agent in the aqueous solution.



Since, Oxalic acid acts as a reducing agent, it can be titrated against Potassium permanganate in the acidic medium involving the following reactions:



In these equations, MnO_4^- is reduced to Mn^{2+} and $\text{C}_2\text{O}_4^{2-}$ is oxidised to CO_2 . The oxidation number of Carbon in $\text{C}_2\text{O}_4^{2-}$ changes from +3 to +4 and that of Manganese in MnO_4^- from +7 to +2.

In these titrations, Potassium permanganate acts as a self-indicator. Initially, the colour of Potassium permanganate is discharged due to its reduction by Oxalic acid. After the complete consumption of oxalate ions, the end point is indicated by the appearance of a light pink colour produced by the addition of a little excess of unreacted Potassium permanganate. Further, during the titration, warming Oxalic acid solution ($50^\circ - 60^\circ \text{C}$) along with dilute H_2SO_4 is required. This is essential because the reaction is very slow at higher temperature. During the titration, Mn^{2+} ions are formed, which act as a catalyst for the reduction of KMnO_4 by Oxalic acid. Therefore, the heating of acid solution is required only in the initial stage.

APPARATUS REQUIRED

Two burettes (5 mL), two conical flasks, distilled water bottle and measuring cylinder.

CHEMICALS REQUIRED

Oxalic acid, Potassium permanganate and dilute Sulphuric acid.

PROCEDURE**I. Preparation of 0.05 M standard solution of Oxalic acid**

Prepare 0.05 M Oxalic acid solution as mentioned in experiment 2.1.

II. Titration of Oxalic acid solution against Potassium permanganate solution

1. Wash the burettes with distilled water.
2. Fill the first burette with standard Oxalic acid solution and the second burette with the given Potassium permanganate solution after rinsing.
3. Adjust the level at zero mark. Read the lower meniscus for Oxalic acid and the upper meniscus for Potassium permanganate solution.
4. Now, take 2 mL Oxalic acid in a titration flask (conical flask) from the burette. Record this reading as V_1 in the observation table.
5. Add 2 mL dilute H_2SO_4 solution to the titration flask. Heat the mixture to 50–60°C.
6. Add KMnO_4 solution dropwise to the titration flask from the burette with constant swirling.
7. Continue this process till a permanent pink colour appears. Now, record this reading as (V_2) in the observation table.
8. Add another 0.5 mL of Oxalic acid solution from the first burette to the titration flask (record 2.5 mL as V_1 in the table). The solution becomes colourless.
9. Titrate it against KMnO_4 solution from the burette as earlier. Record the final volume in continuation with the earlier volume.

Strength of KMnO_4 solution is given by the following equation:

Strength = Molarity \times Molar mass g/L

(Molar mass of KMnO_4 = 158 g/mole)

Calculate the average molarity.

ALTERNATIVE METHOD OF CALCULATION

Plot a graph of the volume of Potassium permanganate added against the volume of Oxalic acid taken. It will be a straight line.

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

Oxalic acid

Potassium permanganate

$$\text{or } M_2 = \frac{a_1 M_1}{a_2} \times \frac{V_1}{V_2}$$

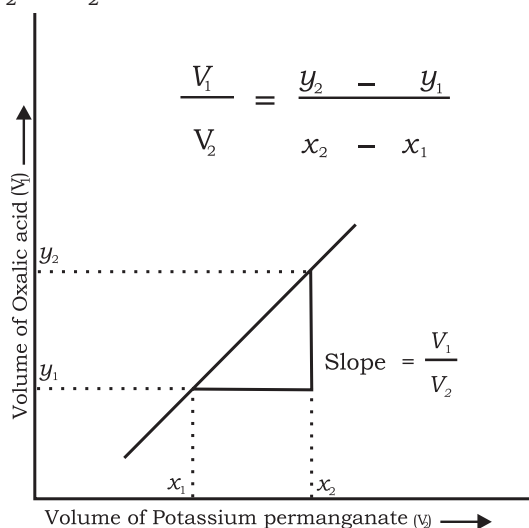


Fig. 2.5

Since V_1/V_2 is known by the graph (Fig. 2.5), M_2 can be calculated.

RESULT

Molarity of KMnO_4 solution is M.

Strength of KMnO_4 solution is g L^{-1} .

PRECAUTIONS

1. Always rinse the burettes with the solutions to be taken in them.
2. Always read the lower meniscus for all colourless solutions and the upper meniscus for coloured solutions.
3. Place the eye parallel to the meniscus to note the burette readings.
4. The strength of the solution must be calculated up to the second place of decimal.
5. Heat the mixture of Oxalic acid and H_2SO_4 solution to $50\text{--}60^\circ\text{C}$ before titrating it against Potassium permanganate.

NOTE

The heating of Oxalic acid and Sulphuric acid mixture may be avoided by adding 4–5 drops of Manganese sulphate solution in the beginning. It is because Mn^{2+} ions from MnSO_4 act as a catalyst.

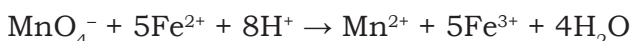
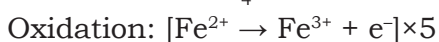
EXPERIMENT 2.6

To determine the strength/molarity of KMnO_4 solution by titrating it against a standard solution of Ferrous ammonium sulphate (Mohr's salt).

THEORY

Like Oxalic acid, Ferrous ammonium sulphate also acts as a reducing agent in titration against Potassium permanganate. The reaction which takes place is given below:

Ionic equation



The oxidation number of Iron in Mohr's salt is +2. Iron is oxidised during the reaction and its oxidation number changes from +2 to +3 and that of Manganese from +7 to +2. In this titration, the heating of Ferrous ammonium sulphate solution is not required because the reaction rate is very high even at room temperature.

APPARATUS REQUIRED

Two burettes (5 mL), conical flask and distilled water bottle.

CHEMICALS REQUIRED

0.01 M KMnO_4 solution, 0.05 M Mohr's salt solution and dilute H_2SO_4 .

PROCEDURE

I. Preparation of 0.05 M, standard solution of Ferrous ammonium sulphate

(Molar mass of $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} = 392 \text{ g mol}^{-1}$).

1. Weigh 0.196 g Ferrous ammonium sulphate accurately and transfer it into a 10 mL measuring flask through a funnel.
2. Transfer the solid sticking to the funnel with the help of distilled water into the flask and add dilute H_2SO_4 dropwise to get a clear solution.
3. Shake the flask till the substance dissolves and make the solution up to the mark.

II. Titration of Ferrous ammonium sulphate against Potassium permanganate solution

1. Wash the burettes with distilled water.
2. Fill the first burette with standard Ferrous ammonium sulphate solution and the second with the given Potassium permanganate solution.
3. Now, take 2 mL of Ferrous ammonium sulphate solution in a conical flask from the burette. Record this reading as V_1 in the observation table.
4. Add 1 mL dilute H_2SO_4 solution to the titration flask.
5. Add KMnO_4 solution dropwise to the conical flask (titrating flask) from the burette with constant swirling.
6. Continue this process till a permanent pink colour appears. Now, record this reading as (V_2) in the observation table.
7. Add another 0.5 mL of Ferrous ammonium sulphate solution from the first burette to the titration flask. (Record 2.5 mL as V_1 in table). The solution becomes colourless.
8. Titrate it again with KMnO_4 solution from the burette as earlier. Record the final volume in continuation with the earlier volume.
9. Take 5–6 readings by adding Mohr's salt solution in increments of 0.5 mL and titrating it against the KMnO_4 solution.

Table 2.4
Titration of Potassium permanganate Solution against
Standard Mohr's salt Solution

| S. No. | Volume of Ferrous ammonium sulphate in mL (V_1) | Volume of KMnO_4 in mL (V_2) | Molarity of Potassium permanganate |
|--------|---|---|------------------------------------|
| 1. | 2.0 | | |
| 2. | 2.5 | | |
| 3. | 3.0 | | |
| 4. | 3.5 | | |
| 5. | 4.0 | | |
| 6. | 4.5 | | |
| 7. | 5.0 | | |

CALCULATIONS

The strength of the unknown solution in terms of molarity may be determined by the following equation:

Ferrous ammonium
sulphate

Potassium permanganate

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

$a_1 = 1$, (number of electrons lost per formula unit of Ferrous ammonium sulphate in the balanced equation).

$a_2 = 5$, (number of electrons gained per formula unit of Potassium permanganate in a balanced equation).

M_1 and M_2 are the molarities, and V_1 and V_2 are the volumes of Ferrous ammonium sulphate and Potassium permanganate, respectively.

On putting the value of a_1 and a_2 in equation, we get

Ferrous ammonium sulphate

Potassium permanganate

$$1M_1V_1 = 5M_2V_2$$

$$M_2 = \frac{M_1V_1}{5V_2}$$

We can calculate the molarity of Potassium permanganate solution by using the above equation. The strength of the solution is given by the following equation:

$$\text{Strength} = \text{Molarity} \times \text{Molar mass g L}^{-1}$$

(Molar mass of $\text{KMnO}_4 = 158 \text{ g/Mol.}$)

ALTERNATIVE METHOD OF CALCULATION

Plot a graph of the volume of Potassium permanganate added against the volume of Ferrous ammonium sulphate taken. It will be a straight line.

Ferrous ammonium sulphate

$$a_1 M_1 V_1$$

$$\text{or } M_2 = \frac{a_1 M_1}{a_2} \times \frac{V_1}{V_2}$$

Potassium permanganate

$$= a_2 M_2 V_2$$

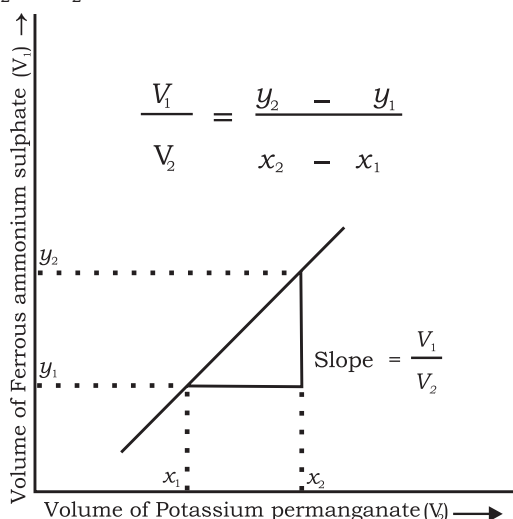


Fig. 2.6

Since V_1/V_2 is known from the graph (Fig. 2.6), M_2 can be calculated.

RESULT

Molarity of given KMnO_4 solution is M

The strength of the given KMnO_4 solution is g L^{-1} .

PRECAUTIONS

1. Always use a fresh sample of Ferrous ammonium sulphate to prepare its standard solution.
2. Other precautions are same as that in Experiment 2.5 except point number 5.

CHAPTER 3

QUALITATIVE ANALYSIS (INORGANIC)

EXPERIMENT 3.1

To detect one cation and one anion from the following:

CATIONS: NH_4^+ , Pb^{2+} , Cu^{2+} , As^{3+} , Al^{3+} , Fe^{3+} , Mn^{2+} , Ni^{2+} , Zn^{2+} , Co^{2+} , Ba^{2+} , Sr^{2+} , Ca^{2+} , Mg^{2+}

ANIONS: CO_3^{2-} , S^{2-} , SO_3^{2-} , NO_2^- , Cl^- , Br^- , I^- , NO_3^- , CH_3COO^- , $\text{C}_2\text{O}_4^{2-}$ (oxalate), SO_4^{2-} , PO_4^{3-}

(Insoluble salts to be excluded)

INTRODUCTION

Inorganic qualitative analysis involves the identification of ions present in a salt, which consists of an anion and a cation. The basis of identification is the observed chemical behaviour of the ions. The salt analysis may be systematically carried out in the following three steps:

- Preliminary examination
- Detection of anion and
- Detection of cation

PRELIMINARY EXAMINATION

Preliminary examination of the salt is carried out in the following order.

1. COLOUR OF THE SALT

Observe the colour of the salt carefully, which may provide useful information about the cations. Table 3.1 gives the characteristic colours of the salts of some cations.

Table 3.1
Characteristic Colours of Some Metal Ions

| Colour | Cation Indicated |
|---------------|------------------|
| Light green | Fe^{2+} |
| Yellow, Brown | Fe^{3+} |

| | |
|--------------|------------------|
| Blue | Cu^{2+} |
| Bright green | Ni^{2+} |
| Blue, Red | Co^{2+} |
| Violet, Pink | Mn^{2+} |

2. DRY HEATING TEST

- Take a few crystals of dry salt in a clean and dry micro test tube and note its colour.
- Heat it and observe the colour of the residue when it is hot. The changes give indications about the presence of cations in some salts, which may not be taken as conclusive evidence (see Table 3.2).

Table 3.2
Inferences from the Colour of the Salt when Cold and on Heating

| Colour when cold | Colour when hot | Inference |
|------------------|-----------------------|------------------|
| Blue | White | Cu^{2+} |
| Green | White | Fe^{2+} |
| White | Yellow | Zn^{2+} |
| Pink | Dirty white or yellow | Co^{2+} |

3. FLAME TEST

The chlorides of several metals impart characteristic colour to the non-luminous flame because they are volatile. This test is performed with the help of a platinum wire/nichrome wire as follows:

- To clean the wire, dip it into concentrated Hydrochloric acid and hold it on a non-luminous flame.
- Repeat step (i) until the wire imparts no colour to the flame.
- Add one drop of concentrated Hydrochloric acid to a pinch of salt taken on a clean watch glass to make a paste.
- Dip the clean wire in this paste and introduce it in the non-luminous (oxidising) flame.
- Observe the colour of the flame and identify the metal ion present in the salt as given in Table 3.3.

Table 3.3
Inference from the Flame Test

| Colour of the flame observed by naked eyes | Inference |
|--|------------------|
| Green flame with blue centre | Cu^{2+} |
| Crimson red | Sr^{2+} |
| Apple green | Ba^{2+} |
| Brick red | Ca^{2+} |

DETECTION OF ANIONS

Anions are divided into three groups on the basis of their reactions with dilute and concentrated H_2SO_4 as given below in Table 3.4.

Table 3.4
Grouping of Anions

| | |
|---|---|
| Give positive test with dilute Sulphuric acid | $\text{CO}_3^{2-}, \text{S}^{2-}, \text{SO}_3^{2-}, \text{NO}_2^-, \text{CH}_3\text{COO}^-$ |
| Give positive test with concentrated Sulphuric acid | $\text{Cl}^-, \text{Br}^-, \text{I}^-, \text{NO}_3^-, \text{C}_2\text{O}_4^{2-}$ |
| Do not give positive test with dilute or concentrated H_2SO_4 | $\text{SO}_4^{2-}, \text{PO}_4^{3-}$ |

I. REACTION WITH DILUTE SULPHURIC ACID

Take a pinch of the salt in a micro test tube, add 4–5 drops of dilute Sulphuric acid and heat. Look for the observations as given in Table 3.5.

Table 3.5
Preliminary Test with Dilute Sulphuric Acid

| Observations | Inference | |
|---|----------------------|----------------------------------|
| | Gas evolved | Possible anion |
| A colourless, odourless gas is evolved with brisk effervescence, which turns lime water milky. | CO_2 | Carbonate (CO_3^{2-}) |
| A colourless gas with the smell of rotten eggs is evolved, which turns paper dipped in Lead acetate solution black. | H_2S | Sulphide (S^{2-}) |

| | | |
|---|------------------------------|---|
| Colourless gas with the smell like that of burning Sulphur, which turns acidified Potassium dichromate solution green and lime water milky. | SO ₂ | Sulphite (SO ₃ ²⁻) |
| Brown fumes, which turn acidified Potassium iodide solution, containing starch, solution blue. | NO ₂ | Nitrite (NO ₂ ⁻) |
| Colourless vapour with the smell of vinegar turns wet blue litmus paper red. | CH ₃ COOH vapours | CH ₃ COO ⁻ |

W-tube may be used for testing the gas.

CONFIRMATORY TESTS FOR CO₃²⁻, S²⁻, SO₃²⁻, NO₂⁻ AND CH₃COO⁻

It is essential to prepare the salt solution for performing confirmatory tests of anions.

Depending upon the solubility of the given salt, its aqueous solution or Sodium carbonate extract is prepared.

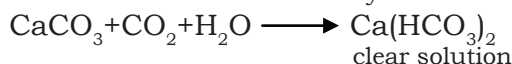
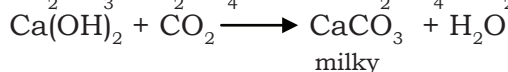
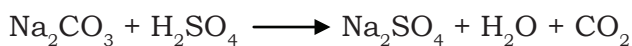
Boil a few milligrams of the salt with 2 mL distilled water for 2–3 minutes. If it dissolves completely, use it for further tests and if it does not, prepare its Sodium carbonate extract.

SODIUM CARBONATE EXTRACT (SE)

Mix one micro spatula of the salt with three micro spatulas of Na₂CO₃ in a boiling tube. Add about 10 mL distilled water and boil it for about five minutes. Cool and filter it. The filtrate is the Sodium carbonate extract (SE).

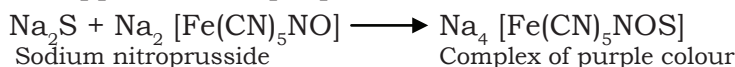
1. CONFIRMATORY TEST FOR CO₃²⁻

Take half a micro spatula of the salt in one arm of W-tube and transfer two drops of lime water in the other arm with the help of a dropper. Now, transfer 2–3 drops of dilute H₂SO₄ with the help of the dropper in the first arm and close it with your thumb. The gas from this arm bubbles through the lime water. The lime water turns milky. If gas is passed in excess through it, the milkiness disappears. CO₃²⁻ is confirmed.



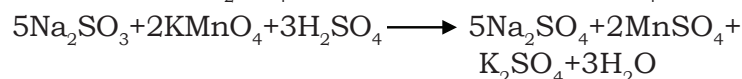
2. CONFIRMATORY TEST FOR S^{2-}

Take a few drops of aqueous salt solution/SE in the well of a well plate and add a drop of Sodium nitroprusside solution, the appearance of purple or violet colour confirms S^{2-} .



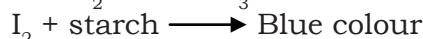
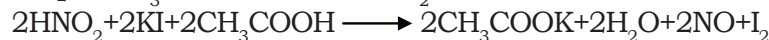
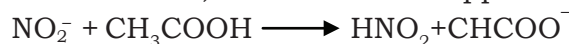
3. CONFIRMATORY TEST FOR SO_3^{2-}

Take a few drops of aqueous salt solution/SE in a micro test tube. Add one drop of KMnO_4 solution. Acidify it with one drop of dilute H_2SO_4 . The pink colour of KMnO_4 disappears.



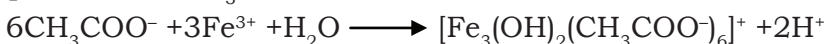
4. CONFIRMATORY TEST FOR NO_2^-

Take three drops of aqueous salt solution or SE in a micro test tube. Add three drops of KI solution and two drops of freshly prepared starch solution. Acidify with 2–3 drops of Acetic acid, the blue colour appears.



5. CONFIRMATORY TEST FOR CH_3COO^-

Take 3–4 drops of aqueous salt solution/SE and add a few drops of neutral FeCl_3 solution, a deep red colour is formed. On heating, it gives a brown-red ppt, which confirms the presence of CH_3COO^- ion.



II. REACTION WITH CONCENTRATED H_2SO_4

Take a micro spatula of salt in a micro test tube, add a few drops of concentrated H_2SO_4 and heat it. Look for anyone of the following observations as given in Table 3.6.

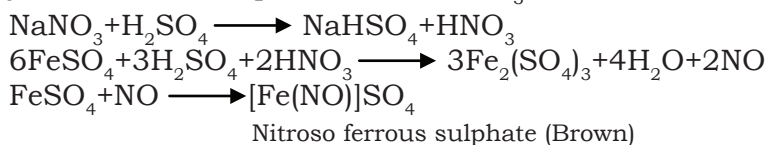
Table 3.6
Preliminary Test with Concentrated Sulphuric Acid

| Observations | Inference | |
|--|---------------------------|---|
| | Gas/ vapour evolved | Possible anion |
| A colourless gas with a pungent smell, which gives dense white fumes when a rod dipped in Ammonium hydroxide is brought near the mouth of the test tube. | HCl | Chloride (Cl^-) |
| Reddish brown gas with a pungent odour is evolved. The intensity of reddish gas increases on heating the reaction mixture after the addition of solid MnO_2 to the reaction mixture. The solution also acquires red colour. | Br_2 vapours | Bromide (Br^-) |
| Violet vapour, which turns starch paper blue, is evolved. Fumes become dense on adding MnO_2 to the reaction mixture. | I_2 vapours | Iodide (I^-) |
| Brown fumes evolve, which become dense upon heating the reaction mixture. After the addition of Copper turnings, the solution becomes blue. | NO_2 | Nitrate (NO_3^-) |
| Colourless and odourless gas is evolved, which turns lime water milky and the gas coming out of the lime water burns with a blue flame, if ignited. | CO and CO_2 | Oxalate ($\text{C}_2\text{O}_4^{2-}$) |

If anyone of the above is detected, carry out the confirmatory test as follows:

1. CONFIRMATORY TEST FOR NO_3^-

Transfer one drop of aqueous solution/SE in one of the wells of the well plate and add one drop of freshly prepared FeSO_4 solution. Add three drops of concentrated H_2SO_4 slowly along the side of the well — a brown ring at the junction of the liquids confirms NO_3^- .

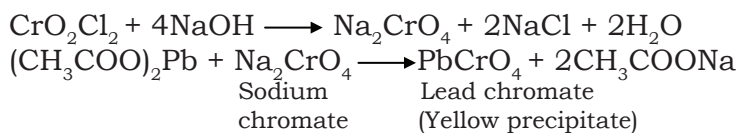
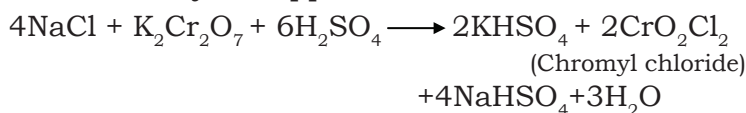


2. TEST FOR Cl^- , Br^- AND I^-

Take four drops of aqueous salt solution/SE in a micro test tube. Acidify with dilute HNO_3 and add four drops of AgNO_3 solution. Observe the colour of the precipitate formed. Cl^- is present if the precipitate is white. Br^- is present if the precipitate is pale yellow and I^- is present if it is yellow. Add 6–7 drops of NH_4OH solution and shake the tube. If white precipitate dissolves completely, Cl^- is confirmed. If pale yellow precipitate dissolves partially, Br^- is confirmed. If yellow precipitate is insoluble, I^- is confirmed.

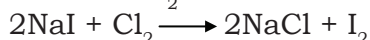
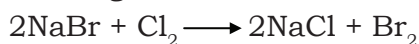
3. CONFIRMATORY TEST FOR Cl^-

Transfer a pinch of salt in one of the arms of the W-tube and add a pinch of solid $\text{K}_2\text{Cr}_2\text{O}_7$, followed by 2–3 drops of concentrated H_2SO_4 . Transfer two drops of NaOH solution in the other arm of the W-tube. Heat carefully the first arm of the W-tube by holding it by the holder and close this end. A reddish orange gas is evolved, which turns the NaOH solution yellow. Add 4–5 drops of Acetic acid to it, followed by one drop of Lead acetate solution. The formation of yellow ppt confirms Cl^- .



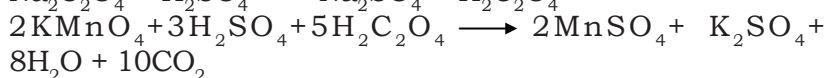
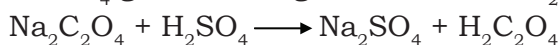
4. CONFIRMATORY TEST FOR Br^- AND I^-

Take five drops of aqueous salt solution/SE in a micro test tube. Add five drops of $\text{CHCl}_3/\text{CCl}_4$ and five drops of concentrated HNO_3 . Shake it vigorously. Observe the colour in the lower organic layer. The appearance of yellow to orange colour confirms Br^- and violet colour confirms I^- .



5. CONFIRMATORY TEST FOR $\text{C}_2\text{O}_4^{2-}$

Take four drops of aqueous salt solution/SE in a micro test tube. Acidify with four drops of dilute H_2SO_4 . Add one drop of KMnO_4 solution and heat. The pink colour of KMnO_4 gets discharged. It confirms $\text{C}_2\text{O}_4^{2-}$.



Anions which do not give positive tests with dilute or concentrated H_2SO_4

1. CONFIRMATORY TEST FOR SO_4^{2-}

Take four drops of aqueous salt solution/SE and acidify with four drops of dilute HCl . Add two drops of BaCl_2 solution. The formation of a white precipitate insoluble in concentrated HCl or concentrated HNO_3 confirms SO_4^{2-} .

2. CONFIRMATORY TEST FOR PO_4^{3-}

Take 4–5 drops of aqueous salt solution/SE in a micro test tube. Acidify with concentrated HNO_3 . Add a pinch of solid Ammonium molybdate and warm the solution. The formation of a yellow precipitate confirms PO_4^{3-} .



Ammonium phosphomolybdate
(Yellow precipitate)

DETECTION OF CATIONS

The cations indicated by the preliminary tests given above are confirmed by a systematic analysis. The first essential step is to prepare a clear and transparent solution of the salt. This is called the 'original solution'. It is prepared as follows:

PREPARATION OF ORIGINAL SOLUTION (OS)

To prepare the original solution, follow the steps one after the other in a systematic order. In case, the salt does not dissolve in a particular solvent even on heating, try the next solvent. Solvents are used in the following order:

- (i) Distilled water
- (ii) Dilute HCl
- (iii) Concentrated HCl
- (iv) Dilute HNO_3
- (v) *Aqua regia* (a mixture of concentrated HCl and concentrated HNO_3 in the ratio of 3:1)

If the salt dissolves in either dilute HNO_3 or *aqua regia*, the solution, thus, obtained is evaporated to dryness in a China dish. The residue so obtained is dissolved in distilled water. Filter if necessary. The clear solution, thus obtained, is used as OS.

GROUP ANALYSIS: The cations are classified into different groups as given in Table 3.7.

Table 3.7
Group Reagents for Precipitating Cations

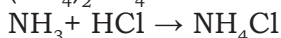
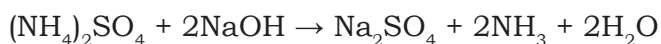
| Group | Cations | Group Reagent |
|------------|---|--|
| Group zero | NH_4^+ | None |
| Group-I | Pb^{2+} | Dilute HCl |
| Group-II | Pb^{2+} , Cu^{2+} , As^{3+} | H_2S gas in presence of dilute HCl |
| Group-III | Al^{3+} , Fe^{3+} | NH_4OH in presence of NH_4Cl |
| Group-IV | Co^{2+} , Ni^{2+} , Mn^{2+} , Zn^{2+} | H_2S in presence of NH_4OH |
| Group-V | Ba^{2+} , Sr^{2+} , Ca^{2+} | $(\text{NH}_4)_2\text{CO}_3$ in presence of NH_4OH |
| Group-VI | Mg^{2+} | None |

ANALYSIS OF ZERO GROUP CATION (NH_4^+ ION)

- Take a pinch of salt in micro test tube. Add 5–6 drops of NaOH solution to it and heat. If there is a smell of Ammonia, it indicates the presence of Ammonium ions. Bring a glass rod dipped in concentrated Hydrochloric acid near the mouth of the test tube. White fumes are observed.
- Repeat the test in a W-tube and pass the gas through Nessler's reagent. The formation of brown precipitate confirms NH_4^+ ion.

CHEMISTRY OF CONFIRMATORY TESTS FOR NH_4^+ ION

Ammonia gas, evolved by the action of Sodium hydroxide on Ammonium salts, reacts with Hydrochloric acid to give Ammonium chloride, which is visible as dense white fume.



On passing the gas through Nessler's reagent, a brown colouration or a brown precipitate of Basic mercury(II) amido-iodine is formed.



Nessler's reagent

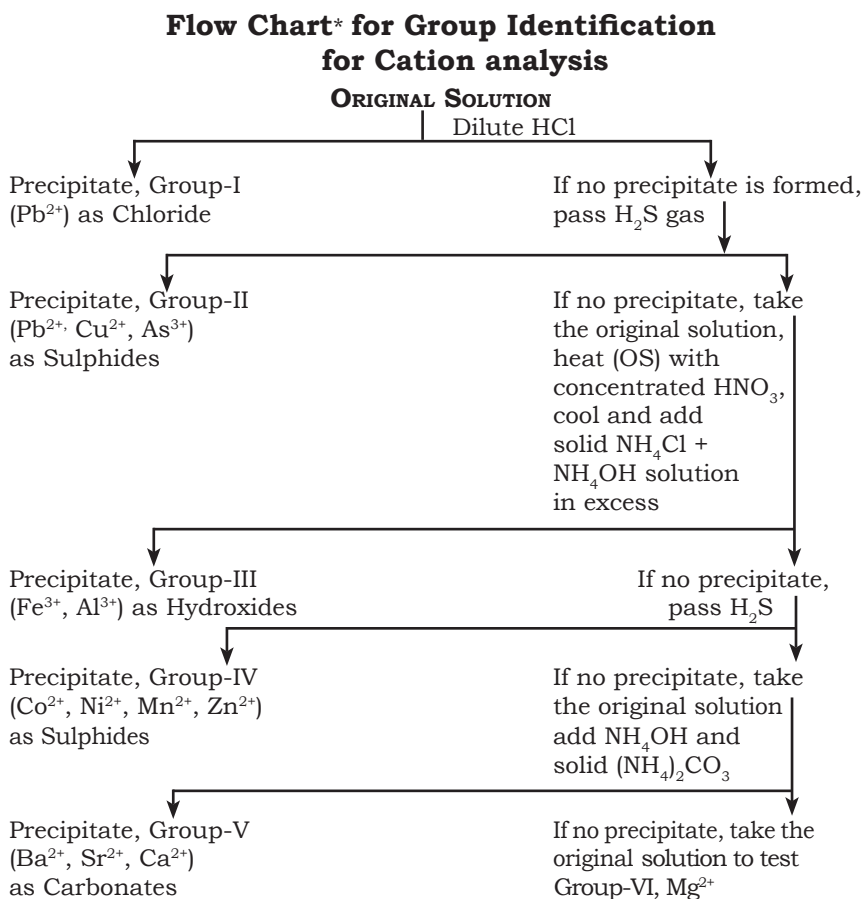
Basic mercury (II)

amido-iodine

(Brown precipitate)

For the analysis of cations belonging to Group I–VI, the cations are precipitated from the original solution by using the group reagents (see Table 3.7), according to the scheme shown in the following flow chart on next page.

The separation of all six groups is represented as shown below:



* The flow chart is for the detection of one cation only. For the detection of more than one cation, modifications will be required.

NOTE

In case, two or more cations are present, use the filtrate of each group to test the presence of the next group.

USE OF H_2S IN GROUP-II AND GROUP-IV

H_2S in acidic medium is the reagent for Group-II and in basic medium is the reagent for Group-IV. For this purpose, H_2S gas has to be passed through the test solution. For analysis at macro scale, H_2S gas is generated in Kipp's apparatus and bubbled through the test solution. However, at microscale, this method is not feasible because the test tubes are small and the gas may push the solution out. Hence, a modified method is used for microscale analysis. A saturated solution of H_2S is prepared in cold distilled water/cold dilute solution of NH_4OH in water. A few drops of this solution are sufficient for the tests. This solution can be stored in a tightly stoppered container kept at a cool place.

ANALYSIS OF GROUP-I CATIONS

Take a small amount of original solution (if prepared in hot concentrated HCl) in a micro test tube and add cold water to it and cool the test tube in tap water. If a white precipitate appears, it indicates the presence of Pb^{2+} ions in Group-I. On the other hand, if the original solution is prepared in water and on the addition of dilute HCl , a white precipitate appears, it may also be Pb^{2+} . If OS is prepared in dilute HCl , Group-I is taken as absent. Confirmatory tests are described below in Table 3.8.

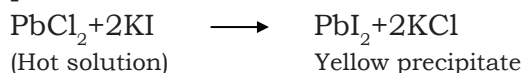
Table 3.8
Confirmatory Test for Cation Pb^{2+}

| | |
|---|---|
| Separate the white ppt and wash with water. Boil the ppt with 8–10 drops of water and transfer two drops each of the resulting clear solution into two wells in the well plate. | |
| To one well, add one drop of Potassium iodide solution. Yellow ppt confirms Pb^{2+} . | To the other well, add one drop of Potassium chromate solution. Yellow ppt confirms Pb^{2+} . |

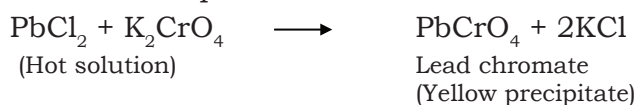
CHEMISTRY OF THE CONFIRMATORY TESTS OF Pb^{2+}

Lead is precipitated as Lead chloride in the first group. The precipitate is soluble in hot water.

- (i) On adding Potassium iodide (KI) solution, a yellow precipitate of Lead iodide is obtained, which confirms the presence of Pb^{2+} ions.



- (ii) On the addition of Potassium chromate (K_2CrO_4) solution, a yellow precipitate of Lead chromate is obtained. This confirms the presence of Pb^{2+} ions.



III. ANALYSIS OF GROUP-II CATIONS

If no precipitate is formed in Group-I, then to the same solution add a few drops of H_2S solution. If a precipitate is formed, then proceed as given below.

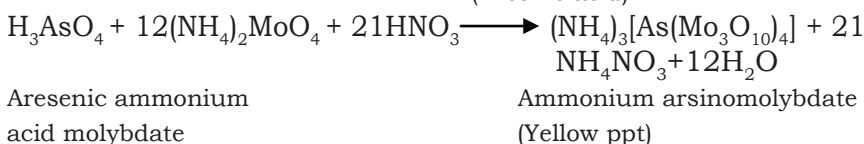
GROUP-II

| | | |
|--|---|--|
| <p>Yellow ppt As^{3+} present</p> <p>Confirmation</p> <p>Dissolve the ppt in minimum quantity of concentrated HNO_3. Boil and then add Ammonium molybdate solution and heat.</p> <p>Canary yellow ppt, As^{3+} confirmed.</p> | <p>Black ppt-Pb^{2+} or Cu^{2+} present</p> <p>Confirmation</p> <p>Transfer one drop of OS in well plate and add one drop of dilute H_2SO_4 and one drop of alcohol. White ppt- Pb^{2+} indicated.</p> <p>Do other tests for Pb^{2+} as given in Group-I table.</p> | <p>To the other part of the solution, add NH_4OH in excess. A blue coloured solution, -Cu^{2+} indicated.</p> <p>Confirmation</p> <p>Transfer one drop of the blue solution to a well in the well plate and two drops to a micro test tube.</p> <p>(i) To the well, add a drop of Acetic acid, followed by a drop of Potassium ferrocyanide solution. Chocolate brown ppt- Cu^{2+} confirmed.</p> <p>(ii) To the micro test tube, add a drop of Acetic acid followed by a drop of Potassium iodide solution. A dirty yellow ppt- Cu^{2+} is confirmed. Add three drops of $\text{CCl}_4/\text{CHCl}_3$ and shake, violet colour in organic layer-Cu^{2+} confirmed.</p> |
|--|---|--|

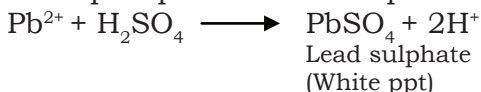
1. TEST FOR ARSENIC ION (As^{3+})

$$3\text{As}_2\text{S}_5 + 10\text{HNO}_3 + 4\text{H}_2\text{O} \longrightarrow 6\text{H}_3\text{AsO}_4 + 10\text{NO} + 15\text{S}$$

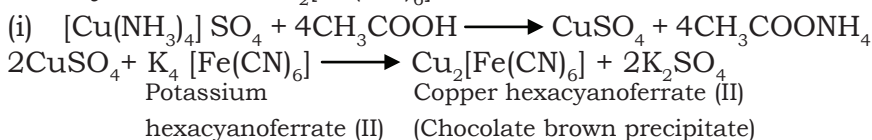
(Arsenic acid)



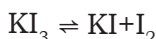
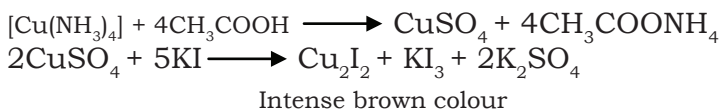
On the addition of dilute H_2SO_4 and a few drops of alcohol, a white precipitate of Lead sulphate confirms the presence of Pb^{2+} .



The blue solution on acidification with Acetic acid, followed by the addition of Potassium ferrocyanide $[K_4Fe(CN)_6]$ solution gives a chocolate colour due to the formation of Copper ferrocyanide i.e., $Cu_2[Fe(CN)_6]$.



(ii) The blue solution on acidification with Acetic acid, followed by the addition of Potassium iodide solution. Dirty yellow ppt- Cu^{2+} confirmed.



On the addition of three drops of $\text{CCl}_4/\text{CHCl}_3$ and on shaking the organic layer, it turns violet which confirms- Cu^{2+} .

IV. ANALYSIS OF GROUP-III CATIONS

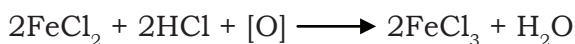
If Group-II is absent, take the original solution in a micro test tube and add 2–3 drops of concentrated HNO_3 to oxidise Fe^{2+} ions to Fe^{3+} ions. Heat the solution for a few minutes. After cooling, add a small amount of solid Ammonium chloride (NH_4Cl) and an excess of Ammonium hydroxide (NH_4OH) solution till it smells of Ammonia. Shake the test tube and observe the colour and the nature of the precipitate. A gelatinous white precipitate indicates the presence of Aluminium ion (Al^{3+}). If the precipitate is brown in colour, it indicates the presence of Ferric ions (Fe^{3+}). If no precipitate is obtained, Group-III is absent. Confirmatory tests of Group-III cations are summarised below.

GROUP-III

| Brown precipitate Fe^{3+} | White precipitate Al^{3+} |
|--|--|
| Dissolve the precipitate in dilute HCl and divide the solution into two parts. (a) To the first part, add Potassium ferrocyanide solution [Potassium hexacyanoferrate (II)]. A blue precipitate/colouration appears. Fe^{3+} confirmed. (b) To the second part, add Potassium thiocyanate solution. A blood red colouration appears. Fe^{3+} confirmed. | Dissolve the white precipitate in dilute HCl and divide into two parts. (a) To the first part, add Sodium hydroxide solution and warm. A white gelatinous precipitate soluble in excess of Sodium hydroxide solution appears. Al^{3+} confirmed. (b) To the second part, first add blue litmus solution, and then, Ammonium hydroxide solution drop-by-drop along the sides of the test tube. A blue floating mass in the colourless solution is obtained. Al^{3+} confirmed. |

CHEMISTRY OF CONFIRMATORY TESTS OF GROUP-III CATIONS

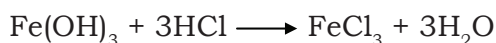
When the original solution is heated with concentrated Nitric acid, Ferrous ions are oxidised to Ferric ions.



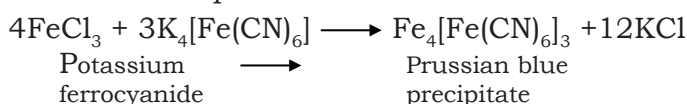
Third group cations are precipitated as their hydroxides, which dissolve in dilute Hydrochloric acid due to the formation of corresponding chlorides.

1. TEST FOR FERRIC IONS (Fe^{3+})

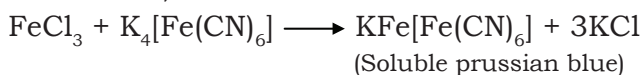
Reddish brown precipitate of Ferric hydroxide dissolves in Hydrochloric acid and Ferric chloride is formed.



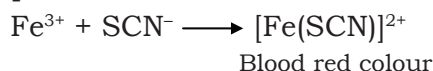
- (a) When the solution containing Ferric chloride is treated with Potassium ferrocyanide solution, a blue precipitate/colouration is obtained. The colour of the precipitate is prussian blue. It is Ferri ferrocyanide. The reaction takes place as follows:



If Potassium hexacyanoferrate (II) (i.e., Potassium ferrocyanide) is added in excess, then a product of composition $\text{KFe}[\text{Fe}(\text{CN})_6]$ is formed. This tends to form a colloidal solution (soluble prussian blue) and cannot be filtered.

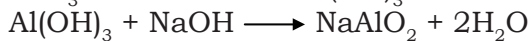
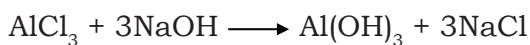


- (b) To the second part of the solution, add Potassium thiocyanate (Potassium sulphocyanide) solution. The appearance of a blood red colouration confirms the presence of Fe^{3+} ions.



2. TEST FOR ALUMINIUM IONS (Al^{3+})

When the solution containing Aluminium chloride is treated with Sodium hydroxide, a white gelatinous precipitate of Aluminium hydroxide is formed, which is soluble in excess of Sodium hydroxide solution due to the formation of Sodium aluminate.



White gelatinous
precipitate

Sodium
aluminate

In the second test, when blue litmus is added to the solution, a red colouration is obtained due to the acidic nature of the

solution. On the addition of NH_4OH solution drop-by-drop, the solution becomes alkaline and Aluminium hydroxide is precipitated. Aluminium hydroxide adsorbs blue colour from the solution and forms insoluble adsorption complex named 'lake'. Thus, a blue mass floating in the colourless solution is obtained. The test is, therefore, called 'lake test'.

V. ANALYSIS OF GROUP-IV CATIONS

If Group-III is absent, add a few drops of H_2S solution to the solution of Group-III. If a precipitate appears (white, black or flesh pink coloured), it indicates the presence of Group-IV cations. The following table gives a summary of confirmatory tests of Group-IV cations.

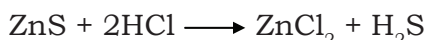
GROUP-IV

| | | | |
|---|--|--|---|
| Black coloured precipitate Ni^{2+} or Co^{2+} indicated. Dissolve black ppt in <i>aqua regia</i> . Heat the solution to dryness and dissolve the residue in water and divide the solution into two parts. | | White ppt indicates Zn^{2+} . | Flesh pink coloured ppt indicates Mn^{2+} . |
| Confirmation of Co^{2+} Take two drops of part-I of the solution or OS in a well plate, add two drops of Acetic acid and a pinch of solid Potassium nitrite. A yellow precipitate confirms Co^{2+} . | Confirmation of Ni^{2+} Take two drops of part-II of the solution or OS in a well of well plate, add a drop of Dimethyl glyoxime solution and NH_4OH red ppt confirms Ni^{2+} . | Confirmation of Zn^{2+} (i) Dissolve ppt in dilute HCl , transfer three drops of the solution to a well and add NaOH dropwise, white ppt soluble in excess of NaOH - Zn^{2+} confirmed. (ii) Transfer two drops of OS to a well and add a drop of Potassium ferrocyanide, a bluish white ppt is obtained. Zn^{2+} confirmed. | Confirmation of Mn^{2+} Dissolve the ppt in dilute HCl by boiling and transfer two drops of it to a well and add NaOH dropwise. A white ppt, which turns brown on standing for sometime, is obtained. Mn^{2+} confirmed. |

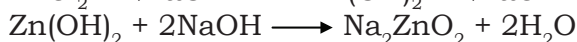
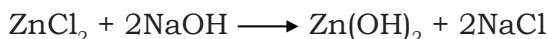
CHEMISTRY OF CONFIRMATORY TESTS OF GROUP-IV CATIONS

1. TEST FOR ZINC ION (Zn^{2+})

Zinc sulphide dissolves in Hydrochloric acid to form Zinc chloride.

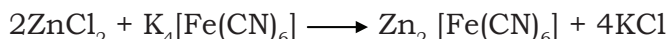


- (a) On the addition of Sodium hydroxide solution, it gives a white precipitate of Zinc hydroxide, which is soluble in excess of NaOH solution on heating. This confirms the presence of Zn^{2+} ions.



Sodium zincate

- (b) When Potassium ferrocyanide $\text{K}_4\text{Fe(CN)}_6$ solution is added to the solution after neutralisation by NH_4OH , a white or a bluish white precipitate of Zinc ferrocyanide appears.

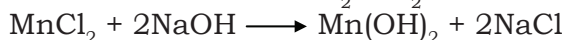
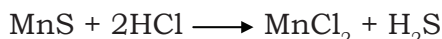


Zinc hexacyanoferrate (II)

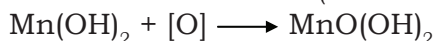
(White or bluish white ppt)

2. TEST FOR MANGANESE ION (Mn^{2+})

Manganese sulphide precipitate dissolves in dilute HCl on boiling. On the addition of NaOH to the solution in excess, a white precipitate of Manganese hydroxide is formed, which turns brown due to atmospheric oxidation into hydrated Manganese dioxide.



(White or bluish white precipitate)

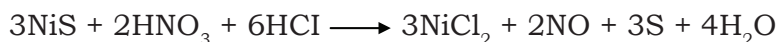


Hydrated Manganese dioxide

(Brown colour)

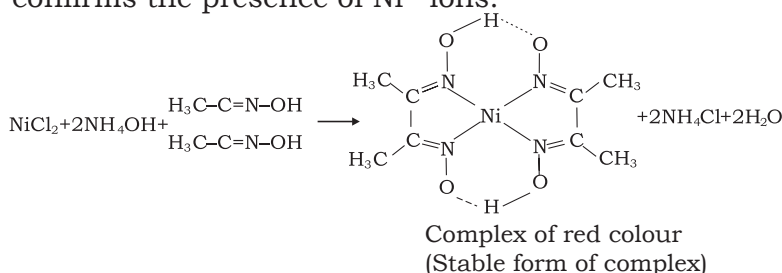
3. TEST FOR NICKEL ION (Ni^{2+})

The black precipitate of Nickel sulphide dissolves in *aqua regia* and the reaction takes place as follows:



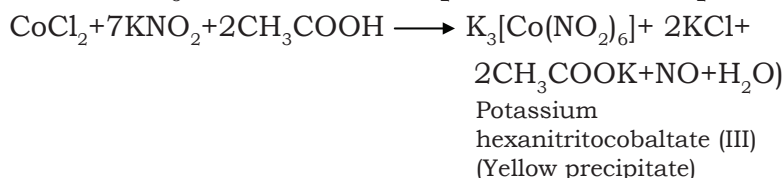
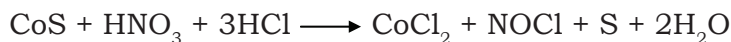
After treatment with *aqua regia*, Nickel chloride is obtained,

which is soluble in water. When Dimethyl glyoxime is added to the solution, prepared by dissolving ppt in *aqua regia* or to OS made alkaline by adding NH_4OH solution, a brilliant red precipitate is obtained, which confirms the presence of Ni^{2+} ions.



4. TEST FOR COBALT ION (Co^{2+})

Cobalt sulphide dissolves in *aqua regia* in the same manner as Nickel sulphide. The solution prepared by dissolving ppt in *aqua regia* or OS is acidified with dilute Acetic acid and on treatment with solid Potassium nitrite a yellow precipitate of the complex of cobalt named Potassium hexanitritocobaltate (III) is formed, which confirms the presence of Co^{2+} ions.



VI. ANALYSIS OF GROUP-V CATIONS

If Group-IV is absent, then take the original solution in a micro test tube and add a small amount of solid NH_4Cl and an excess of NH_4OH solution, followed by solid Ammonium carbonate $(\text{NH}_4)_2\text{CO}_3$. If a white precipitate appears, this indicates the presence of Group-V cations

White precipitate indicates Ba^{2+} or Sr^{2+} or Ca^{2+} .

Separate the precipitate by decantation and wash with water. Dissolve in hot dilute Acetic acid and divide the solution into three parts.

Confirmation to be carried out in the following sequence.

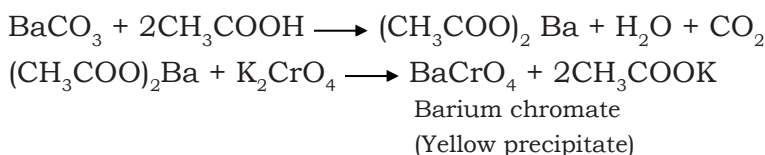
| Confirmation of Ba²⁺ | Confirmation of Sr²⁺ | Confirmation of Ca²⁺ |
|---|---|---|
| (i) Transfer two drops of the solution to a well and add two drops of Potassium chromate solution– Yellow ppt confirms Ba ²⁺ . (ii) Perform flame test with the given salt. Apple green flame confirms Ba ²⁺ . | (i) Transfer two drops of the solution to a well and add two drops of Potassium chromate solution– Yellow ppt confirms Ba ²⁺ . (ii) Perform flame test with the given salt. Apple green flame confirms Ba ²⁺ . | (i) To a part of the solution, add two drops of Ammonium oxalate solution in a micro test tube and shake. White ppt confirms Ca ²⁺ . (ii) Perform flame test with the given salt. Brick red flame confirms Ca ²⁺ . |

CHEMISTRY OF CONFIRMATORY TESTS OF GROUP-V CATIONS

The Group-V cations are precipitated as their carbonates, which dissolve in Acetic acid due to the formation of corresponding acetates.

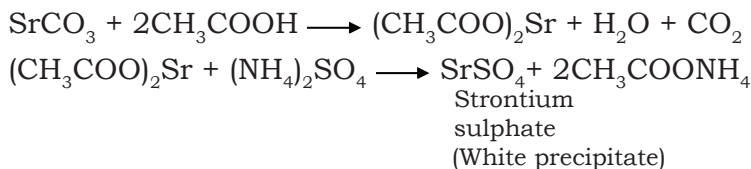
1. TEST FOR BARIUM ION (Ba²⁺)

Potassium chromate (K₂CrO₄) solution gives a yellow precipitate of Barium chromate when the solution of fifth group precipitate in Acetic acid is treated with it.



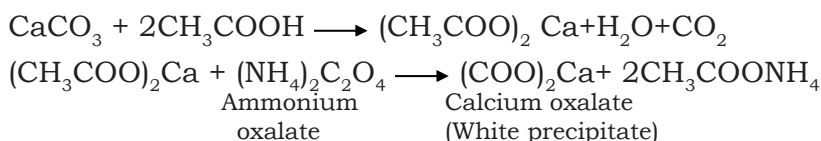
2. TEST FOR STRONTIUM ION (Sr²⁺)

Solution of fifth group precipitate in Acetic acid gives a white precipitate of Strontium sulphate with Ammonium sulphate, (NH₄)₂SO₄, solution on heating and scratching the sides of the test tube with a glass rod.



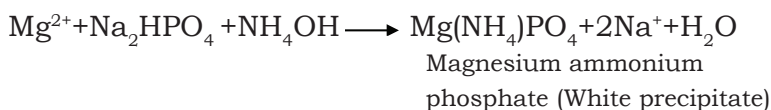
3. TEST FOR CALCIUM ION (Ca²⁺)

Solution of the fifth group precipitate in Acetic acid gives a white precipitate with Ammonium oxalate solution.



VII. ANALYSIS OF GROUP-VI

If precipitate is not formed in group-V then perform the test for Mg²⁺. Transfer 6–7 drops of OS in a micro test tube, add a pinch of solid NH₄Cl, shake and add NH₄OH in slight excess. Add a few drops of Disodium hydrogen phosphate and scratch the side of the test tube with a glass rod. A White PPT Confirms Mg²⁺.



NOTE

In place of Na₂HPO₄, (NH₄)₃PO₄ can also be used.

COMMENTS

The following points should be kept in mind while using the micro Kit for qualitative analysis:

1. The original solution (OS) should be prepared in micro test tube.
2. Whenever heating is required or organic solvents, like CHCl₃ or CCl₄, use the micro test tube.
3. For confirmatory tests, prefer a well plate.
4. For all gas confirmation, use W-tube e.g., CO₃²⁻, NO₃⁻, etc.

NOTE

Watch glass/petridish can be used in place of a well plate for performing tests of cations.

CHEMICALS REQUIRED FOR QUALITATIVE ANALYSIS

Concentrated HCl, concentrated H_2SO_4 , dilute H_2SO_4 , lime water, Lead acetate, $\text{K}_2\text{Cr}_2\text{O}_7$, KI, starch, Na_2CO_3 , Sodium nitroprusside, BaCl_2 , KMnO_4 , Acetic acid, Oxalic acid, NH_4OH , MnO_2 , Cu turnings, FeSO_4 , dilute HNO_3 , concentrated HNO_3 , AgNO_3 , CHCl_3 , Chlorine water, Ammonium molybdate, NaOH, Nessler's reagent, H_2S gas, K_2CrO_4 , alcohol, Potassium ferrocyanide, Ammonium chloride, Potassium thiocyanate, Potassium nitrite, Ammonium thiocyanate, DMG, Ammonium carbonate, Ammonium sulphate, Ammonium oxalate and Disodium hydrogen phosphate.

CHAPTER 4

QUALITATIVE ANALYSIS (ORGANIC)

EXPERIMENT 4.1

To detect extra elements in the given organic compound.
For the detection of extra elements, it is essential to prepare Lassaigue's Extract to convert the extra elements into water soluble anions.

APPARATUS REQUIRED

Three ignition tubes, a pair of tongs, spirit lamp/burner, micro spatula, dropper, China dish, glass rod, tripod stand, wire gauze, funnel and filter paper.

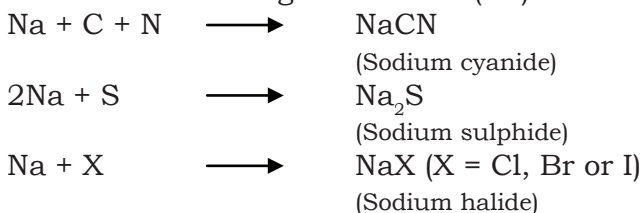
CHEMICALS REQUIRED

Sodium metal, Ferrous sulphate (solid), dilute Sulphuric acid, Sodium nitroprusside, Lead acetate, Acetic acid, dilute Nitric acid, Silver nitrate, Ammonium hydroxide, Chloroform/Carbon tetrachloride and Chlorine water.

PREPARATION OF LASSAIGNE'S EXTRACT (LE)

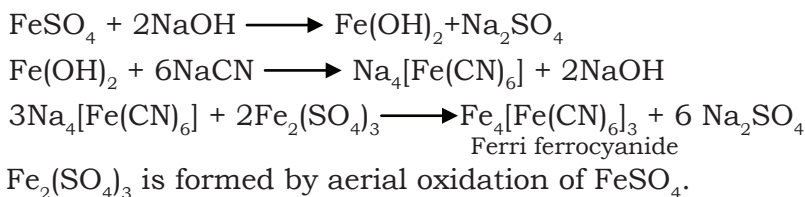
1. Take a small piece of freshly cut Sodium metal and dry it between the folds of a filter paper. Take it in an ignition tube.
2. Hold the ignition tube with a pair of tongs and heat it till the Sodium melts into a shining globule.
3. Drop a pinch of solid organic compound with the help of a micro spatula into the hot ignition tube. If the compound is liquid, first drop a pinch of solid Sodium carbonate into the hot ignition tube, and then, add 2–3 drops of the liquid organic compound.
4. Heat the ignition tube at the tip of the flame till it becomes red hot.
5. Put the red hot ignition tube in a China dish containing 5 mL distilled water and cover it immediately with a wire gauze. Break the bottom of the ignition tube with the help of a glass rod, if it does not break itself.

- Repeat the process with two more ignition tubes.
- Boil the contents of the China dish on a tripod stand with a wire gauze for two minutes and filter. The filtrate is known as Lassaigne's Extract (LE)



TEST FOR NITROGEN

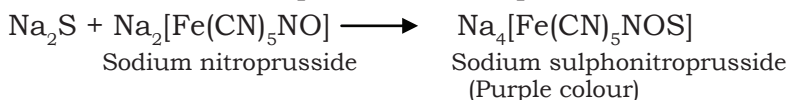
Take five drops of LE in a micro test tube and add a few crystals of Ferrous sulphate. Warm it and add five drops of dilute Sulphuric acid. A blue colour or precipitate confirms the presence of Nitrogen.



TESTS FOR SULPHUR

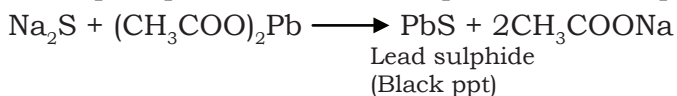
1. SODIUM NITROPRUSSIDE TEST

Take two drops of LE in a well plate and add two drops of Sodium nitroprusside solution. The formation of purple colour confirms the presence of Sulphur.



2. LEAD ACETATE TEST

Take two drops of LE in a well plate. Add two drops of Acetic acid, and then, one drop of Lead acetate. The formation of black precipitate confirms the presence of Sulphur.

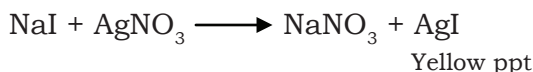
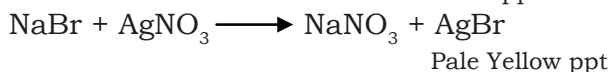
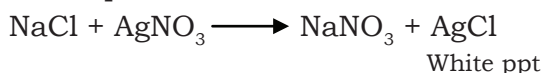


TESTS FOR HALOGENS

1. AgNO_3 TEST

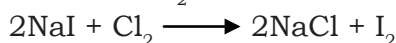
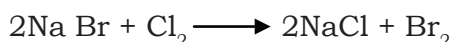
Take five drops of LE in a micro test tube. Acidify with dilute HNO_3 and add two drops of AgNO_3 solution. The formation of a white or yellow precipitate indicates the presence of a halogen. Check its solubility in NH_4OH .

- (a) White precipitate, which is completely soluble in NH_4OH , confirms the presence of Cl.
- (b) Pale yellow precipitate, which is partially soluble in NH_4OH , confirms the presence of Br.
- (c) Yellow precipitate, which is insoluble in NH_4OH , confirms the presence of I.



2. LAYER TEST

Take 4–5 drops of LE in a micro test tube. Acidify with dilute HNO_3 . Add five drops of CCl_4 or CHCl_3 , which form the lower layer. Now, add five drops of freshly prepared Chlorine water and shake the tube. The appearance of yellow to orange colour in the lower layer confirms the presence of Br^- and purple colour confirms the presence of I^- .



EXPERIMENT 4.2

To identify one functional group present in an organic compound from the following:

1. Unsaturation
2. Alcoholic
3. Phenolic
4. Aldehydic and Ketonic
5. Carboxylic acid
6. Amino group (primary)

APPARATUS REQUIRED

Well plate, micro test tubes, burner, W-tube, droppers, ignition tubes and water bath (use 50 mL glass beaker as water bath).

CHEMICALS REQUIRED

Calcium carbide, alcoholic Ferric chloride, Sodium hydrogen carbonate, Sodium nitrite, β -naphthol, Ferrous sulphate, alkaline solution of KMnO_4 , Bromine water, ceric Ammonium nitrate reagent, 2,4-DNP reagent, Fehling's solution A, Fehling's solution B, Schiff's reagent, Silver nitrate, dilute HCl , alcohol, Sodium hydroxide and Acetone.

PROCEDURE

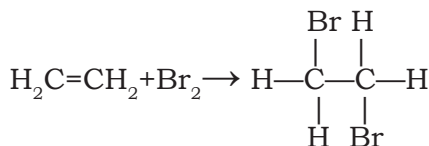
1. TEST OF UNSATURATION

(a) BAEYER'S TEST

- (i) Take a micro spatula of CaC_2 on one side of the W-tube and on the other side, dilute alkaline KMnO_4 solution.
- (ii) Add a few drops of water to the side containing CaC_2 and close the mouth with your thumb. The produced gas, Acetylene, goes to the other side of the W-tube and reacts with KMnO_4 solution, which gets decolourised within a few seconds.

(b) BROMINE WATER TEST

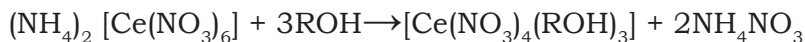
Repeat the above experiment by taking a few drops of Bromine water in place of alkaline KMnO_4 solution. The yellow Bromine water gets decolourised.



2. TEST FOR ALCOHOL

CERIC AMMONIUM NITRATE TEST

Take 3–4 drops of the organic compound (if liquid) or 3–4 drops of its solution in Acetone (if solid) in a micro test tube and add one drop of ceric Ammonium nitrate reagent. The appearance of wine-red colour indicates the presence of alcoholic group.



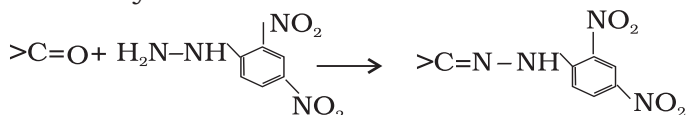
3. TEST FOR PHENOL

Take 3–4 drops of the solution of the compound in alcohol and add one drop of alcoholic FeCl_3 solution. The appearance of green, blue or purple colour indicates the presence of phenolic group.



4. TESTS FOR ALDEHYDES AND KETONES

Take 3–4 drops of 2,4-DNP reagent and add one drop of the compound (if liquid) or its solution in alcohol (if solid). The formation of yellow to red precipitate indicates the presence of carbonyl group ($>\text{C}=\text{O}$). This test is common to both aldehydes and ketones.



TESTS TO DISTINGUISH BETWEEN ALDEHYDES AND KETONES

The following tests are given by aldehydes only. If these tests are negative, the compound is taken to be a ketone.

(i) SCHIFF'S REAGENT TEST

Take 3–4 drops of the compound (if liquid) or its alcoholic solution, if solid, in a micro test tube and add 2–3 drops of the Schiff's reagent. The appearance of pink colour indicates the presence of an aldehyde.

(ii) FEHLING'S SOLUTION TEST

Take five drops each of Fehling's solution A and Fehling's solution B in a micro test tube. Add 2–3 drops of organic compound (if liquid) or its alcoholic solution to it. Heat the contents for about two minutes in a water bath. The formation of a brick-red precipitate of Cuprous oxide indicates the presence of an aldehyde.

NOTE

This test is not given by aromatic aldehydes.

(iii) TOLLEN'S REAGENT TEST (SILVER MIRROR TEST)

Take 1 mL of Silver nitrate solution in a micro test tube. Add one drop of Sodium hydroxide solution to it and shake. A dark brown precipitate of Silver oxide appears. Dissolve the precipitate by adding Ammonium hydroxide solution dropwise. Now, add one drop of aqueous or alcoholic solution of organic compound. Heat the reaction mixture on a water bath for five minutes. The formation of a silver mirror on the inner side of the test tube indicates the presence of an aldehyde. Sometimes, in place of silver mirror, a black precipitate is formed.

5. TEST FOR CARBOXYLIC ACID GROUP

SODIUM HYDROGENCARBONATE TEST

Take 2 mL of approximately 5% aqueous solution of Sodium hydrogencarbonate in a micro test tube. Add a few drops/a pinch of organic compound. The brisk effervescence indicates the presence of Carboxylic group.

6. TEST FOR AMINO GROUP (PRIMARY AROMATIC AMINE)

AZO DYE TEST

Dissolve one drop/a pinch of the organic compound in minimum amount of dilute HCl in a micro test tube. Cool it

to 0–5°C and add 10 drops of cold Sodium nitrite solution. A solution of Diazonium chloride salt is formed. In another micro test tube, take a small crystal of β -naphthol (2-naphthol) and dissolve it in minimum amount of dilute NaOH solution and add it to the Diazonium chloride solution obtained earlier, slowly with shaking. The formation of an orange or red dye confirms the presence of an aromatic primary amino compound.

Note for the Teacher
Suggested List of Organic Compounds for
Functional Group Analysis

| | |
|-----------------------------|---------------------------------|
| Methanol | 2-Naphthol (β -Naphthol) |
| Ethanol | Methanal (formaldehyde) |
| Propan-2-ol | Ethanal (Acetaldehyde) |
| Phenol | Propanone (Acetone) |
| Resorcinol | Butan-2-one (Ethylmethylketone) |
| Ethanoic acid (Acetic acid) | Aniline |
| Benzoic acid | |
| Oxalic acid | |
| Citric acid | |

EXPERIMENT 4.3

To characterise carbohydrates, fats and proteins in pure form and detect their presence in the given food stuff.

APPARATUS REQUIRED

Well plate, 10 micro test tubes, test tube stand, pasteur pipette, dropper, micro spatula, spirit lamp/burner, tripod stand and China dish.

CHEMICALS REQUIRED

Molisch's reagent, concentrated H_2SO_4 , concentrated HNO_3 , Benedict's solution, Fehling's solution A, Fehling's solution B, Silver nitrate, NH_4OH , NaOH , Iodine solution, starch solution, Potassium hydrogensulphate, Copper sulphate, glucose and α -naphthol.

FOOD STUFF REQUIRED

Gram, groundnut/mustard oil, wheat flour, milk, grape juice and egg.

PROCEDURE

1. TEST FOR CARBOHYDRATES

MOLISCH'S TEST

Dissolve a micro spatula of glucose in 2 mL water to form a clear solution. In a micro test tube, take five drops of the above glucose solution, add 2–3 drops of α -naphthol solution in alcohol, and then, add 2–3 drops of concentrated H_2SO_4 along the side of the micro test tube. The formation of purple ring at the interface of two layers confirms the presence of carbohydrates.

2. TEST FOR REDUCING SUGARS

(a) FEHLING'S TEST

Take 1 mL each of Fehling's solution A and B in a micro test tube and add 2–3 drops of the above solution. Heat the contents for about two minutes on a water bath. The formation of a brick-red ppt of Cuprous oxide indicates the presence of carbohydrates with reducing property, such as glucose and fructose.

(b) BENEDICT'S TEST

Take 1 mL of Benedict's reagent in a micro test tube. Add 2–3 drops of the glucose solution prepared above and heat it. The formation of red ppt due to the formation of Copper oxide indicates the presence of carbohydrates with reducing property e.g., glucose and fructose.

(c) TOLLEN'S REAGENT TEST

Take 1 mL of freshly prepared Silver nitrate (2%) in a test tube. Add one drop of Sodium hydroxide solution to it and shake it well. A dark brown ppt of Silver oxide appears. Dissolve the ppt by adding Ammonium hydroxide solution dropwise. Now, add 2–3 drops of the glucose solution prepared above and heat the reaction mixture (without shaking) in a water bath for five minutes. The formation of silver mirror on the inner side of the test tube or black ppt indicates the presence of reducing sugar. For the above tests in food stuff, grape juice can be used in place of glucose solution.

3. TEST FOR STARCH

IODINE TEST

Heat a small amount of substance (wheat flour) with water to get an aqueous colloidal solution of starch. Add a few drops of Iodine solution. The appearance of blue colour indicates the presence of starch.

4. TEST FOR OILS AND FATS

Add a few crystals of dry Potassium hydrogensulphate to 3 mL of groundnut oil/mustard oil/ghee taken in a test tube and heat it gently. A pungent smell confirms the presence of oil or fat. The presence of oil or fat in food stuff can be determined in the following way. Crush 1 g of the given food stuff with a pestle and mortar and transfer it on a clean white paper (don't use filter paper). Fold the paper and press it. Open the folds of the paper, remove the food stuff and observe the paper against light. Translucent spots confirm the presence of oils/fats.

5. TEST FOR PROTEINS

(a) BIURET TEST

Warm 2–3 drops of egg albumin (white portion of egg)/milk in 1 mL water with two drops of Sodium hydroxide solution. Then, add a few drops of Copper sulphate solution. The appearance of violet colour indicates the presence of proteins.

(b) XANTHOPROTEIC TEST

Take egg albumin dispersion in 1 mL water and add 2–3 drops of concentrated HNO_3 and heat. A yellow precipitate indicates the presence of protein.

CHROMATOGRAPHY

The technique of chromatography is vastly used for the separation, purification and identification of compounds. Chromatography is a physical method of separation, in which the components to be separated are distributed between two phases, one of which is stationary, while the other, known as the mobile phase, moves in a definite direction. The distribution of the component may take place either by adsorption or by partition.

1. ADSORPTION CHROMATOGRAPHY

In adsorption chromatography, the substances are selectively adsorbed in the stationary phase. Stronger the adsorption, slower is the movement of the substance with the mobile phase. Column and thin layer chromatography are the common examples of adsorption chromatography.

(a) COLUMN CHROMATOGRAPHY

In it, the stationary phase is a suitable adsorbent packed in a glass column. The mobile phase (a suitable solvent) flows down through the packed column. The weakly adsorbed substance is taken away by it initially, followed by the strongly adsorbed substance. This results in their separation.

(b) THIN LAYER CHROMATOGRAPHY

The stationary phase in it is a thin layer of a suitable adsorbent adhering to a glass plate. The mobile phase (a suitable solvent) moves up due to capillary action. A weakly adsorbed substance moves up faster with the mobile phase than a strongly adsorbed substance. This results in their separation.

2. PARTITION CHROMATOGRAPHY

Paper chromatography is the common example of partition chromatography, in which water present in the pores of a

paper is the stationary phase and a suitable solvent is the mobile phase. The separation depends upon the partition of substances between the two phases. The substance, which is more soluble in the mobile phase, moves faster with it. This results in the separation of different substances. Using a particular solvent (mobile phase) and at a given temperature, different substances move at different rates. These different rates are expressed in terms of a property called ' R_f ', known as 'retention factor'. It expressed as:

$$R_f = \frac{\text{Distance travelled by the substance from the reference line}}{\text{Distance travelled by the solvent front from the reference line}}$$

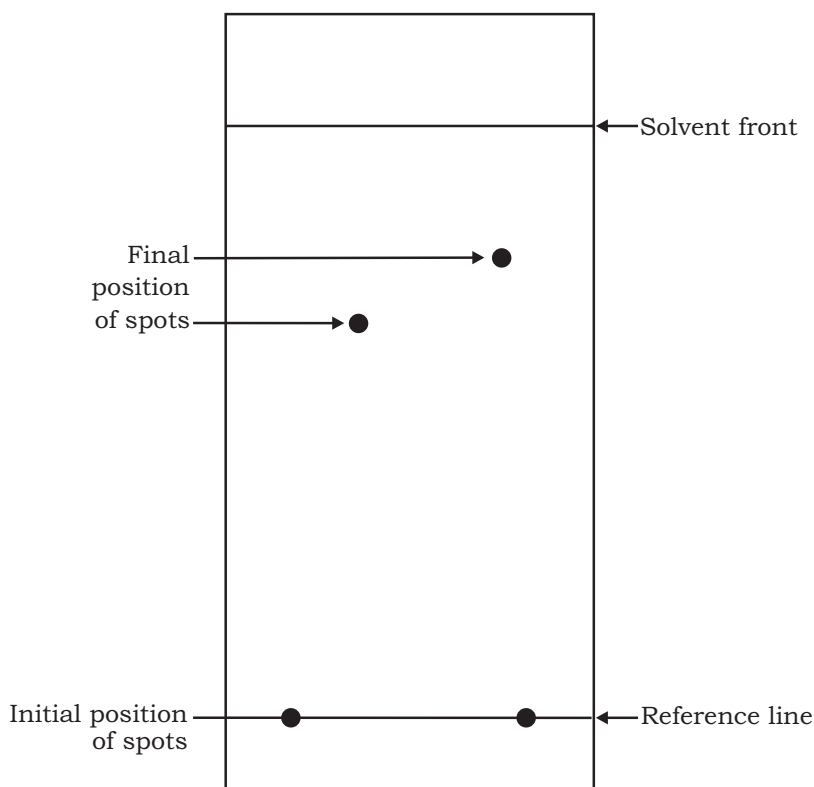


Fig. 5.1: Paper chromatography showing the reference line and the solvent front

Reference line is the horizontal line passing through the points where the spots of different substances are put.

Since the solvent front moves faster than the compounds, the R_f value of a substance will always be less than one. Also note that the R_f value has no unit. If the compound is coloured, then its position on the chromatographic paper may be easily located. However, if the substance is colourless, it may be treated with a suitable reagent, which imparts it a characteristic colour. This reagent is given the name 'developer'. Iodine is the most commonly used developer in paper chromatography. Several other techniques are also available for locating the spots.

EXPERIMENT 5.1

To study the separation of pigments from extracts of leaves and flowers by paper chromatography and determination of their R_f values.

APPARATUS REQUIRED

Chromatography jar with cork and hook, strip of filter paper–6mm×15 cm, capillary tube and black pencil.

CHEMICALS REQUIRED

Ethanol and extract of leaves/flowers/ink.

PROCEDURE

1. Draw a horizontal reference line with the help of a pencil about 3 cm away from one edge of the 15-cm-long strip of filter paper.
2. Put a spot of extract of leaves, flower, etc., with the help of a capillary tube on the reference line.
3. Transfer 5–6 mL of Ethanol into the jar.
4. Mount the paper strip on the hook fixed to a cork.
5. Carefully insert the filter paper into the jar so that the lower end dips in Ethanol. The spot should be above the level of the Ethanol.
6. Wait for the Ethanol to rise up to three-fourth length of the paper strip.
7. Remove the paper strip from the jar and mark the spots and solvent front on the filter paper.

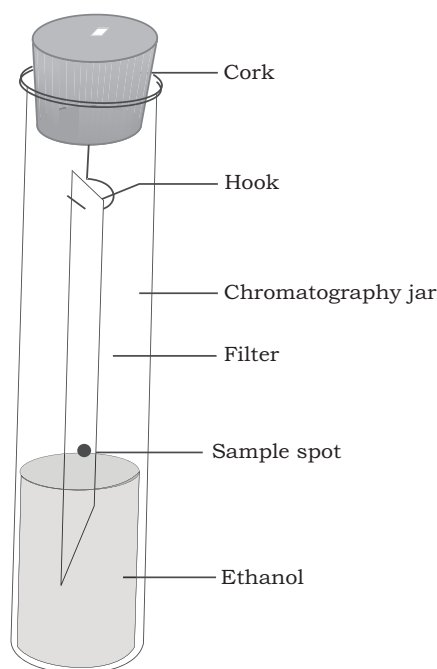


Fig. 5.2: Arrangement for vertical chromatography

- Measure the distances travelled by different spots and the solvent from the reference line.
- Repeat the procedure by using a new strip of filter paper and water as solvent.

Observations

| S. No. | Name of the solvent | Colour of the separated components | | Distance travelled by the components | | Distance travelled by the solvent | R _f Values | |
|--------|---------------------|------------------------------------|---|--------------------------------------|---|-----------------------------------|-----------------------|---|
| | | A | B | A | B | | A | B |
| 1. | Alcohol | | | | | | | |
| 2. | Water | | | | | | | |

RESULT

.....

PRECAUTIONS

- Use a fine capillary to put a spot on the paper to avoid its spreading.
- After putting the spot on the filter strip, allow it to dry completely before fixing the paper in the jar.
- Dip the paper strip in the solvent in such a way that the spot of the mixture is above the solvent level.
- Ensure that the filter paper strip hangs freely in the jar without touching the sides.
- Keep the jar covered with a lid when the chromatogram is being developed.
- Once the experiment is set, do not disturb the jar as long as the chromatogram is being developed.

EXPERIMENT 5.2

To separate the constituents present in an inorganic mixture containing two cations only (Pb^{2+} and Cd^{2+}) using the technique of paper chromatography.

THEORY

The principle for the separation of cations is the same as has been explained in Experiment 5.1. In this case, the two cations to be separated are colourless. Therefore, a developer is needed. In the present case, Ammonium sulphide ($(\text{NH}_4)_2\text{S}$) can be used to locate the position of these ions on chromatographic paper or plate.

APPARATUS REQUIRED

Two petridish, cotton and circular Whatman paper

CHEMICALS REQUIRED

Solution of mixture of the above two cations (Pb^{2+} and Cd^{2+}), Ammonium sulphide solution and developing solvent (Ethanol, 6M HNO_3 and water in the ratio of 8:1:1).

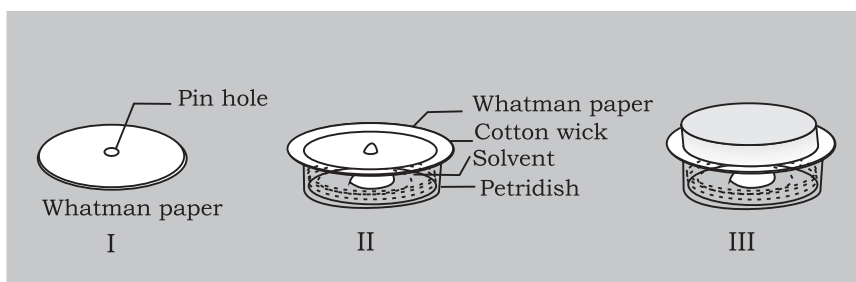


Fig. 5.3 Arrangement for horizontal chromatography

PROCEDURE

1. Put a spot of the mixture on the marked centre of the circular paper.
2. Make a pointed wick with cotton.
3. Insert the tip of the wick in the centre of the paper with the help of a pin where the spot is put.
4. Keep the base of the wick in the solvent in the petridish and let it stand for a while. Cover the paper with another petridish.

- Take the paper (chromatogram) out, mark the solvent front and dry it. Spray Ammonium sulphide solution on it.
- Calculate R_f value of both the components (cations).

Observation

| S. No. | Colour of the spots of cations | Distance travelled by the components of the spots from the reference line | Distance travelled by the solvent from the reference line | R_f value |
|--------|--------------------------------|---|---|-------------|
| 1. | | | | |
| 2. | | | | |

RESULT

R_f value of Pb^{2+} ring (black ring due to the formation of PbS).

R_f value of Cd^{2+} ring (yellow ring due to the formation of Cd).

NOTE

Ammonium sulphide is prepared by passing H_2S gas through a mixture containing 100 mL H_2O and 10 mL liquor Ammonia for about 45 minutes.

PRECAUTIONS

- Use a fine capillary to put the spot on the filter paper to avoid its spreading.
- After putting the spots on the filter paper, allow them to dry completely.
- The tip of the wick should be fine.
- The filter paper should be slightly bigger than the petridish.
- Keep the filter paper covered with the petridish when the chromatogram is being developed.
- Once the experiment is set, do not disturb the petridish as long as the chromatogram is being developed.
- Spray the chromatogram with the developer only after it is completely dry.

CHAPTER 6

PREPARATION OF COMPOUNDS

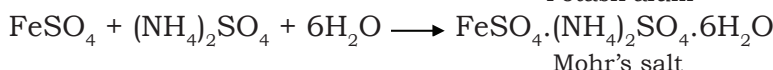
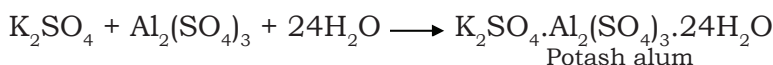
I. PREPARATION OF INORGANIC COMPOUNDS

EXPERIMENT 6.1

To prepare double salts (i) Potash alum (ii) Ferrous ammonium sulphate (Mohr's salt).

THEORY

When a mixture containing equimolar proportions of the two constituents of double salt is crystallised from its solution, a double salt is formed as follows.



Dilute Sulphuric acid is added to the solution to prevent the hydrolysis of Al^{3+} ions or Fe^{+2} ions in the corresponding mixtures.

APPARATUS REQUIRED

Two beakers (50 mL), glass rod, filtration apparatus, tripod stand and spirit lamp.

CHEMICALS REQUIRED

Potassium sulphate (K_2SO_4), Aluminium sulphate ($\text{Al}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$), Ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), Ammonium sulphate [$(\text{NH}_4)_2\text{SO}_4$] and dilute Sulphuric acid.

PROCEDURE

(i) POTASH ALUM

1. Dissolve 1.7 g Potassium sulphate in 10 mL distilled water taken in a 50 mL beaker.
2. Dissolve 4.5 g Aluminium sulphate in 10 mL distilled water in another 50 mL beaker. Add 4–5 drops of dilute H_2SO_4 .

- Mix the two solutions.
- Concentrate the resulting solution by heating, till the saturation point is reached. Check it by dipping the tip of a glass rod in the solution, take it out and blow this end of the glass rod with air. Small crystals appear on the tip of the glass rod at saturation point.
- Allow the solution to cool to room temperature slowly and leave it for a few hours. Gradually, the crystals of Potash alum will separate out.
- Separate the crystals by filtration, wash with cold water, dry between the folds of a filter paper and record the yield.

RESULT

Yield=..... g.

Colour=.....

Shape=.....

(ii) MOHR'S SALT

Dissolve 2.8 g Ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and 1.3 g Ammonium sulphate ($(\text{NH}_4)_2\text{SO}_4$) in 15 mL water taken in a 50 mL beaker.

- Add about 5–6 drops of dilute H_2SO_4 .
- Concentrate the solution by heating till the saturation point is reached.
- Allow the solution to cool to room temperature slowly and leave it for a few hours. Gradually, the crystals of Mohr's salt separate out.
- Filter the crystals, dry them between the folds of a filter paper and weigh.

RESULT

Yield=..... g.

Colour=.....

Shape=.....

PRECAUTIONS

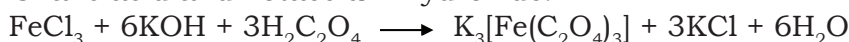
The crystals may not appear immediately on cooling. If so, leave the solution overnight.

EXPERIMENT 6.2

To prepare Potassium trioxalatoferrate (III).

THEORY

Green coloured crystals of Potassium trioxalatoferrate (III) are obtained from an aqueous solution containing Ferric chloride, Oxalic acid and Potassium hydroxide.



APPARATUS REQUIRED

Beaker (50 mL), China dish, filtration unit, burner/spirit lamp, tripod stand and glass rod.

CHEMICALS REQUIRED

Oxalic acid $[(\text{COOH})_2 \cdot 2\text{H}_2\text{O}]$, Potassium hydroxide (KOH) and Ferric chloride (FeCl_3).

PROCEDURE

1. Prepare Oxalic acid solution by taking 1.5 g Oxalic acid in 10 mL hot water in a 50 mL clean beaker.
2. To the above solution, add 1.9 g Potassium hydroxide gradually in lots with stirring so that it dissolves completely.
3. Now, add 1.2 g Ferric chloride to the reaction mixture with constant stirring till it completely dissolves.
4. Heat the solution and concentrate the green solution in a China dish over the burner till the saturation point is reached. Cool the solution slowly and leave it for a few hours.
5. Filter the crystals formed, dry them by pressing between the folds of a filter paper.
6. Record the yield.

RESULT

The yield of Potassium trioxalatoferrate (III) is g.

PRECAUTIONS

Crystals of Potassium trioxalatoferrate (III) may not appear immediately on cooling. The solution may have to be left overnight.

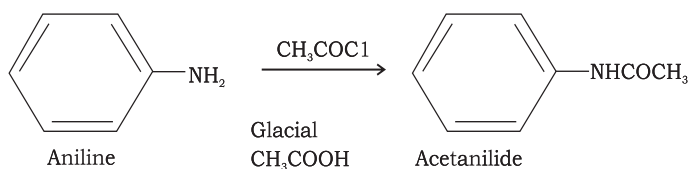
II. PREPARATION OF ORGANIC COMPOUNDS

EXPERIMENT 6.3

To prepare Acetanilide.

THEORY

The replacement of one hydrogen atom of the NH_2 group of aniline by $\text{CH}_3\text{CO}-$ group in the presence of glacial Acetic acid gives acetanilide. Acetylation with Acetyl chloride is carried out in the presence of glacial Acetic acid.



APPARATUS REQUIRED

Boiling tube, water bath, melting point assembly, micro filtration unit and beaker (50 mL)

CHEMICALS REQUIRED

Aniline, glacial Acetic acid, Acetyl chloride and blue litmus paper.

PROCEDURE

1. Take 1 mL of aniline in a dry boiling tube, add 1 mL glacial Acetic acid to it and mix the two.
2. To the above mixture, add 1 mL Acetyl chloride in lots of 4–5 drops at a time with shaking. The mixture becomes warm. If the boiling tube becomes unbearable to touch, cool it under tap water.
3. After the addition of the whole amount of Acetyl chloride, heat the mixture occasionally over flame with shaking for about 15 minutes. The reaction mixture should not boil.
4. Cool the boiling tube and add the mixture to ice-cold water (10 mL) taken in a beaker (50 mL) with constant stirring.
5. Filter acetanilide separated as white solid and wash with water till the filtrate is neutral to litmus.

6. Crystallise a small quantity of crude acetanilide with hot water. White shining needle-shaped crystals are obtained.
7. Dry the crystals, weigh and determine the melting point. Report the yield of crude compound and melting point of crystallised sample.

RESULT

Yield=.....g

Melting point=.....°C

PRECAUTIONS

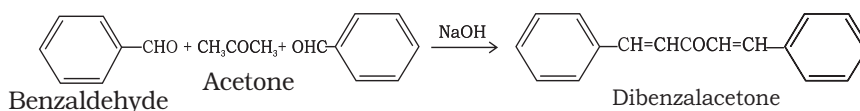
1. Use dry boiling tube for the preparation.
2. Do not inhale the vapours during the addition of Acetyl chloride and during heating.
3. Determine the melting point of dried and crystallised sample.

EXPERIMENT 6.4

To prepare Dibenzalacetone (Dibenzylidene acetone).

THEORY

The α -hydrogen atom of aliphatic aldehydes and ketones is acidic in nature. In the presence of dilute alkali, an aldehyde or ketone containing α -hydrogen condenses with an aromatic aldehyde to give β -unsaturated aldehyde or ketone. This reaction is called 'Claisen-Schmidt reaction'. For example, benzaldehyde undergoes condensation with Acetone in the presence of aqueous Sodium hydroxide to give Dibenzalacetone.



APPARATUS REQUIRED

Micro conical flask, beaker (50 mL), funnel, melting point assembly, thermometer and micro filtration unit.

CHEMICALS REQUIRED

Ethanol, NaOH, Benzaldehyde, Acetone and ice.

PROCEDURE

1. Prepare a solution of 1 g Sodium hydroxide in a mixture of 8 mL Ethanol and 10 mL water taken in a 50 mL beaker. Cool the beaker in a water bath maintained at a temperature of about 10–15°C.
2. Prepare a mixture of 1 mL Benzaldehyde and 0.5 mL Acetone in a micro conical flask. Cool it in ice bath. Add half of this mixture slowly in ice cooled NaOH solution prepared in Step 1 with vigorous stirring. A fluffy precipitate is formed within 1–2 minutes. Stir the mixture gently for about five minutes.
3. After five minutes, add the remaining mixture of Benzaldehyde and Acetone and occasionally stir for 30 minutes.
4. Filter the pale yellow solid obtained and wash with cold water. Dry it and crystallise its small amount using alcohol.

5. Report the yield of the crude product and melting point of the crystallised compound.

RESULT

Yield=.....g

Melting point=.....°C

PRECAUTIONS

1. Ensure proper cooling of the reactants before mixing them.
2. Handle Ethanol carefully as it is inflammable.

EXPERIMENT 6.5

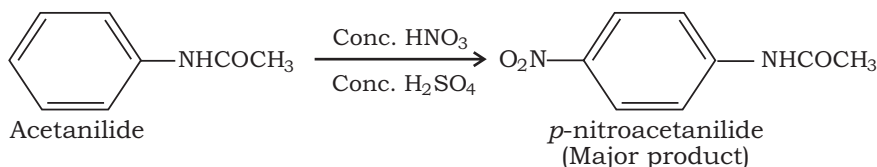
To prepare *p*-nitroacetanilide.

THEORY

p-Nitroacetanilide is prepared by the nitration of acetanilide by using a mixture of concentrated Nitric acid and concentrated Sulphuric acid as nitrating reagent. The mixture of the two acids releases Nitronium ion (NO_2^+), which acts as an electrophile in the reaction.



Nitronium ion attacks the Benzene ring, containing anilide group, mainly at the para position to give *p*-nitroacetanilide as a major product. This is an example of aromatic electrophilic substitution reaction.



APPARATUS REQUIRED

Micro conical flask, micro funnel, glass rod, ice bath, micro filtration unit and melting point assembly.

CHEMICALS REQUIRED

Acetanilide, glacial Acetic acid, concentrated H_2SO_4 , concentrated HNO_3 and ice Ethanol/Methanol.

PROCEDURE

1. Dissolve 1 g of acetanilide in 1 mL glacial Acetic acid taken in a micro conical flask.
2. Add 2 mL of concentrated H_2SO_4 to the above mixture gradually with stirring. The mixture becomes hot and a clear solution is obtained. Cool the reaction mixture in an ice bath maintained at $0-5^\circ\text{C}$.
3. Add a cold mixture of 0.5 mL concentrated HNO_3 and 0.3 mL concentrated H_2SO_4 to the viscous reaction mixture drop-by-drop with constant stirring/shaking, so that the temperature of the mixture does not rise above 10°C .

4. Remove the conical flask from the ice bath and allow the reaction mixture to attain room temperature. Let it stand at room temperature for about 30 minutes with occasional stirring. Then, pour it on about 30 g crushed ice.
5. Stir the mixture well and filter the compound so obtained.
6. Wash the compound with cold water and dry it.
7. Crystallise a small amount of the pale yellow solid from alcohol. Colourless crystals of *p*-nitroacetanilide are obtained. Yellow ortho-nitroacetanilide formed in small amount remains dissolved in the mother liquor.
8. Record the yield of the crude product and melting point of the pure compound.

RESULT

Yield=.....g

Melting point=.....°C

PRECAUTIONS

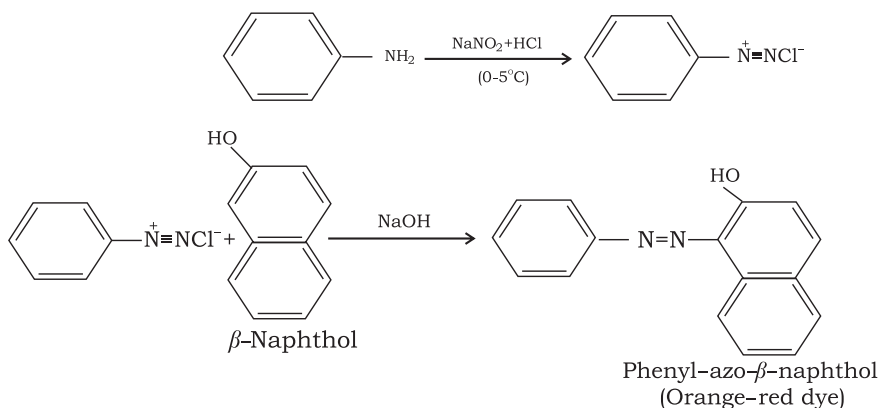
1. Handle the nitrating mixture of concentrated HNO_3 and concentrated H_2SO_4 carefully.
2. Do not inhale the fumes of the reaction mixture.

EXPERIMENT 6.6

To prepare Phenyl-azo- β -naphthol (an azo dye).

THEORY

Aniline is an aromatic primary amine. It forms Diazonium salt when treated with Nitrous acid at 0–5°C. Nitrous acid is generated in-situ by the reaction of Sodium nitrite with Hydrochloric acid. The process is called 'diazotisation'. Diazonium salt is coupled with an alkaline solution of β -naphthol to form an orange-red azo dye.



APPARATUS REQUIRED

Beaker (50 mL), conical flask (10 mL), glass rod, thermometer, filter paper, funnel, melting point assembly and micro filtration unit.

CHEMICALS REQUIRED

Aniline, concentrated HCl , Sodium nitrite, β -naphthol, Sodium hydroxide, glacial Acetic acid and ice.

PROCEDURE

1. Take 1.6 mL concentrated Hydrochloric acid in a 50 mL beaker. Dilute it with 1.6 mL water and dissolve 0.5 mL aniline in it.
2. Cool the above mixture by placing the beaker in an ice bath maintained at 0–5°C.
3. Diazotise the above mixture by adding a cool solution of 0.4 g Sodium nitrite in 2 mL water.

4. Dissolve 0.8 g β -naphthol in 4.5 mL of 10% Sodium hydroxide solution. Add about 10 g of crushed ice to it.
5. Stir the β -naphthol solution well and add chilled Diazonium chloride solution slowly to it with constant stirring.
6. An orange-red dye of Phenyl-azo- β -naphthol is formed.
7. Allow the mixture to stand in the bath for 10 minutes with occasional shaking.
8. Filter the crystals obtained and wash well with cold water.
9. Crystallise a small amount of the crude product from glacial Acetic acid.
10. Filter the crystallised sample. Wash with a little alcohol to remove Acetic acid. Dry the crystallised sample.
11. Record the yield of the crude product and the melting point of the crystallised compound.

RESULT

Yield=.....g

Melting point=.....°C

PRECAUTIONS

1. Maintain the temperature of the reaction mixture in the range of 0–5°C during diazotisation.
2. Always add Diazonium chloride solution in alkaline β -naphthol for dye formation and not vice versa.

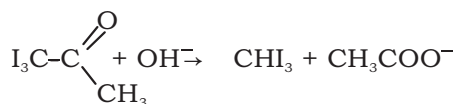
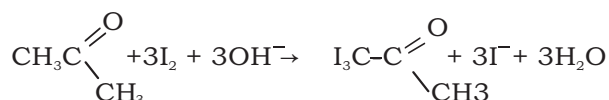
EXPERIMENT 6.7

To prepare Iodoform.

THEORY

Iodoform is prepared by the reaction of a compound containing $\text{CH}_3\text{CO}-$ group (for example in aldehydes and ketone) or any compound, which would produce this group during the reaction, such as Ethanol or 2-propanol. Traditionally, Iodoform is prepared by the reaction of Iodine in alkaline medium with an organic compound.

Alternatively, the reaction can be carried out using Sodium hypochlorite and Potassium iodide. Sodium hypochlorite oxidises the I^- ions from KI to iodine, I_2 , which reacts with the organic compound.



APPARATUS REQUIRED

Beaker (50 mL), measuring cylinder (10 mL), funnel, glass rod, melting point assembly and micro filtration unit.

CHEMICALS REQUIRED

Acetone, Potassium iodide, Sodium hypochlorite solution (4%) and Ethanol.

PROCEDURE

1. Dissolve 0.15 g KI in 20 mL water taken in a 50 mL beaker. Add 0.5 mL acetone to it.
2. Add 20 mL 4% Sodium hypochlorite solution to the above mixture with gentle stirring. Continue stirring the reaction mixture for 10 minutes. A yellow solid separates out.
3. Filter the yellow solid and wash it with water.

4. Crystallise the compound with alcohol.
5. Record the yield and the melting point of the pure compound.

RESULT

Yield=.....g

Melting point=.....°C

PRECAUTIONS

Sodium hypochlorite is commercially available in solutions of different concentrations. Use the appropriate quantity of the solution according to its concentration.

NOTE

Crystallised sample of Acetanilide, *p*-nitroacetanilide and Iodoform may be used for detection of extra elements (N and I).

CHAPTER 7

CHEMICAL EQUILIBRIUM

EXPERIMENT 7.1

To study the pH change by common ion effect in case of weak acids and bases.

APPARATUS REQUIRED

Six micro test tubes and dropper/pasteur pipette.

I. USING A WEAK BASE AND ITS SALT

CHEMICALS REQUIRED

1M NH_4OH , 1M NH_4Cl and universal indicator solution/paper.

PROCEDURE

Mix a number of drops of solutions of NH_4OH and NH_4Cl and water in micro test tubes as given in Table 7.1. Test each solution with universal pH solution or paper and record its colour. Find out the corresponding pH value from the chart given with the universal indicator and record it in Table 7.1.

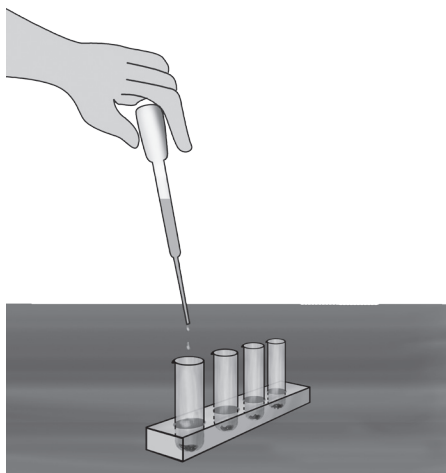


Table 7.1
Observation Table for Common Ion Effect
(NH_4OH 1M and NH_4Cl 1M)

| S. No. | Volume (Drops) of | | | Colour Produced | pH Value |
|--------|------------------------|------------------------|-------|-----------------|----------|
| | NH_4OH | NH_4Cl | Water | | |
| 1. | 3 | – | 22 | | |
| 2. | 3 | 3 | 19 | | |
| 3. | 3 | 1 | 21 | | |
| 4. | 1 | 3 | 21 | | |
| 5. | 2 | 20 | 3 | | |
| 6. | 20 | 2 | 3 | | |

II. USING A WEAK ACID AND ITS SALT

CHEMICALS REQUIRED

1M CH_3COOH , 1M CH_3COONa and universal indicator/solution.

PROCEDURE

Mix the solutions of CH_3COOH and CH_3COONa in the ratios given in Table 7.2 and find out the pH using universal indicator/paper.

Table 7.2
Observation Table for Common Ion
Effect (CH_3COOH 1M and CH_3COONa 1M)

| S. No. | Volume (Drops) of | | | Colour Produced | pH Value |
|--------|--------------------------|---------------------------|-------|-----------------|----------|
| | CH_3COOH | CH_3COONa | Water | | |
| 1. | 3 | – | 22 | | |
| 2. | 3 | 3 | 19 | | |
| 3. | 3 | 1 | 21 | | |
| 4. | 1 | 3 | 21 | | |
| 5. | 2 | 20 | 3 | | |
| 6. | 20 | 2 | 3 | | |

CONCLUSION

Interpret your observations regarding the change in pH as a result of common ion effect.

EXPERIMENT 7.2

To study the shift in equilibrium between the chemical systems by either increasing the concentration of ions or the temperature.

APPARATUS REQUIRED

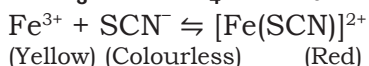
Glass rod, micro test tubes, spatula, test tube stand, micro beakers, droppers, well plate, pasteur pipette and spirit lamp/ bunsen burner, beaker (50 mL) and thermometer.

CHEMICALS REQUIRED

1% FeCl_3 , 1% NH_4SCN , CoCl_2 and dilute and concentrated HCl

PROCEDURE

(i) FeCl_3 and NH_4SCN System



(a) EFFECT OF CHANGE IN CONCENTRATION

1. Take 10 mL water in a micro beaker and add two drops of Ferric chloride solution and two drops of NH_4SCN . It is an almost colourless solution with a light yellow tinge. Record the colour of the solution. Transfer five drops of this solution into each of the four micro test tubes of well plate.
2. To well-1, add two drops of water and use it as control for comparison in the next steps.
3. Increase the concentration of FeCl_3 in micro test tube-2 by adding one drop of water and one drop of FeCl_3 solution and two drops in micro test tube-3. Mix, observe and record whether the solution is less or more intense red as compared to the control solution (well-1).
4. Add two drops of NH_4SCN solution in the micro test tube-4. Mix, observe and record the colour of the solution as compared to the solution in the micro test tube in the given table.

Table 7.3
Effect of Concentration

| S. No. | Solution | Colour |
|--------|---|--------|
| 1. | Mixture solution + 2 drops of water | |
| 2. | Mixture solution + 1 drop of water + 1 drop of FeCl_3 | |
| 3. | Mixture solution + 2 drops of NH_4SCN | |

(b) EFFECT OF CHANGE IN TEMPERATURE

Take five drops of each of the mixture.

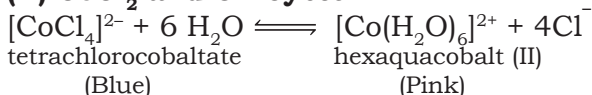
1. Pass the glass rod through the flame of the burner 3–4 times. Put the rod in the well-2. Note the colour change at high temperature.
2. Remove the rod, clean it with water and dip in cold water and put it in well-2. Note the colour change at low temperature.

Table 7.4
Effect of Temperature

| S. No. | Temperature | Colour |
|--------|------------------|--------|
| 1. | Room temperature | |
| 2. | High temperature | |
| 3. | Low temperature | |

PROCEDURE

(ii) CoCl_2 and Cl^- System



(a) EFFECT OF CHANGE IN CONCENTRATION

1. Take one micro spatula of solid Cobalt chloride in each of the two micro beakers/test tubes.
2. Using a dropper, add six drops of water in each of the test tubes —1 and 2. Use the tip of spatula to stir until all crystals have dissolved. Note the colour of the solution. Record in Table 7.5.
3. One test tube is kept as standard for comparison.

- Using another dropper, add 2–3 drops of dilute HCl in test tube-2 and record the colour.
- Wait for about 30 seconds, and then add 5–6 drops of water in test tube-2. Note the colour.

Table 7.5
Effect of Concentration

| S. No. | Solution | Colour |
|--------|--|--------|
| 1. | Concentrated aq. solution of CoCl_2 | |
| 2. | Above solution + concentrated HCl | |
| 3. | Above solution + H_2O | |

CONCLUSION

Interpret your observations regarding the shift in equilibrium as a result of increasing the concentration of concentrated HCl and H_2O .

(b) EFFECT OF CHANGE IN TEMPERATURE

- Take one micro spatula of solid Cobalt chloride into each of the two micro test tubes.
- Using a dropper, add six drops of water in each of the test tubes.
- Using another dropper, add 2–3 drops of dilute HCl in each of the test tubes.
- One test tube is kept as standard for comparison.
- Heat the other test tube in a water bath and record the colour of the solution at 4–5 different temperatures in Table 7.6.

Table 7.6
Effect of Temperature

| S. No. | Temperature | Colour of the Solution |
|--------|-------------|------------------------|
| | | |
| | | |
| | | |

CONCLUSION

Interpret your observations regarding the shift in equilibrium as a result of increasing temperature.

SURFACE CHEMISTRY

In a true solution, solute particles mix homogeneously with the molecules of a solvent, and thus, form a single phase. However, a colloidal solution is a heterogeneous system, in which very fine particles of one substance disperse (dispersed phase) in another substance called dispersion medium. Particles of the dispersed phase do not form a single phase with particles of the dispersion medium as they are either very large molecules or essentially aggregates of small molecules. Colloidal particles are larger in size than simple molecules but small enough to remain suspended in the dispersion medium (1–100 nm). Some examples of very large molecules which form colloidal dispersion are starch, gum and proteins, whereas, colloidal Sulphur is an example of aggregate of small molecules. Further, a heterogeneous system of a solid as dispersed phase and a liquid as dispersion medium is called a 'sol'. Depending upon the nature of interaction between the dispersed phase and the dispersion medium, colloidal sols are divided into two categories, namely, lyophilic (solvent attracting) and lyophobic (solvent repelling). If water is the dispersion medium, the terms used are 'hydrophilic' and 'hydrophobic'. Egg albumin, starch and gum are lyophilic sols. Freshly prepared Ferric hydroxide, Aluminium hydroxide and Arsenic sulphide sols are examples of lyophobic sols. A few methods of preparation of colloids are — chemical methods, electrical disintegration, mechanical methods, like grinding, use of a colloid mill and peptisation. In this unit, you will learn to prepare both types of sols. Also, you will learn a method for purification of sols.

EXPERIMENT 8.1

To prepare lyophilic and lyophobic sols for the following:

- (1) Egg albumin (2) Starch/gum (3) $\text{Fe}(\text{OH})_3/\text{Al}(\text{OH})_3$
- (4) As_2S_3

APPARATUS REQUIRED

Beakers (10 mL and 50 mL), measuring cylinder, spirit lamp/burner with tripod stand, glass rod, pasteur pipette/dropper, micro test tubes with stand and China dish.

CHEMICALS REQUIRED

FeCl_3 , AlCl_3 , As_2O_3 , starch, gum, egg albumin, H_2S , $\text{Al}_2(\text{SO}_4)_3$, NaCl and parchment paper.

PROCEDURE

I. PREPARATION OF LYOPHILIC SOL

1. EGG ALBUMIN SOL

Prepare 10 mL 15 per cent (w/v) solution of NaCl in a 50 mL beaker. Break one egg in a China dish and suck 10 mL of the albumin with the help of pasteur pipette and pour it in Sodium chloride solution. Stir it well to ensure that the sol has been well prepared.

2. STARCH/GUM SOL

- (i) Measure 20 mL distilled water with the help of a measuring cylinder and transfer it to a 50 mL beaker and boil it.
- (ii) Make a paste of 0.1 g starch or gum in water and transfer this paste in boiling water with constant stirring.

II. PREPARATION OF LYOPHOBIC SOL

1. FERRIC HYDROXIDE/ALUMINIUM HYDROXIDE SOL

- (i) Take 5 mL distilled water in a 10 mL beaker and boil it.
- (ii) Add 5–6 drops of 3% Ferric chloride/Aluminium chloride solution to the boiling water and stir it well. Keep the water boiling till brown sol of $\text{Fe}(\text{OH})_3$ or white sol of $\text{Al}(\text{OH})_3$ is formed.

2. ARSENIOSULPHIDE SOL

- (i) Take 25 mL distilled water in a 50-mL beaker and boil.
- (ii) Add 0.2 g Arsenious oxide to water and continue boiling the contents for 2–3 minutes.
- (iii) Cool and filter the solution.
- (iv) Pass H_2S gas through the filtered solution till the solution gives the smell of H_2S .
- (v) Expel H_2S gas from the solution by slow heating and filter it.
- (vi) Arsenious sulphide sol is formed.

PRECAUTIONS

1. While preparing colloidal solutions of starch, gum, Ferric chloride, Aluminium chloride, etc., pour the paste or solution gradually into the boiling water with constant stirring. Addition of these substances in excess may cause precipitation.
2. Arsenious oxide is poisonous in nature, so wash your hands immediately every time after handling this chemical.

EXPERIMENT 8.2

To purify prepared sol by dialysis.

APPARATUS REQUIRED

Parchment/cellophane paper (30cm × 30cm) — one sheet, trough-one, thread and two test tubes.

CHEMICALS REQUIRED

Colloidal dispersion of egg albumin (prepared in Experiment 8.1), distilled water and Silver nitrate.

PROCEDURE

1. Take a square sheet (30cm × 30cm) of parchment/cellophane paper.
2. Soak the sheet in water and give it a conical shape.
3. Pour the colloidal dispersion of egg albumin in the cone of parchment/cellophane paper.
4. Tie the cone with a thread and suspend it in a trough containing distilled water.
5. After about half-an-hour, test for the presence of ions in the trough water as mentioned below.
6. Change the water present in the trough after every half-an-hour till it is free of the impurities of Na^+ and Cl^- ions. To check the presence of Cl^- ions, take water from the trough in a test tube and add Silver nitrate solution. A white precipitate of Silver chloride indicates the presence of chloride ions.
7. Note the time required for the purification of colloidal dispersion.

NOTE

In some cases, dialysis may be a slow process. Therefore, in such cases, it is advisable to change the water of the trough twice or thrice till the colloidal dispersion is free of ions.

PRECAUTIONS

1. For dialysis, keep the neck of the parchment bag above the water surface.
2. Change the water in the trough from time to time during dialysis.

EXPERIMENT 8.3

To study the role of emulsifying agents in stabilising the emulsion of different oils.

THEORY

Emulsion is a type of colloid, in which both the dispersed phase and the dispersion medium are liquids. Here, the 'dispersed phase' and the 'dispersion medium' are distinguished by their relative amounts. The one, which is present in smaller proportion, is called 'dispersed phase', while the other, which is present in relatively large quantity, is known as the 'dispersion medium'.

When oil is shaken with water, a faint milky mixture is often observed, which is unstable and is called an emulsion of oil in water (o/w). On standing, it gets separated into two layers, i.e., oil and water. The mixing capacity of different oils with water is different. This mixing capacity of an oil, in addition to its nature, depends on the method of shaking also (i.e., vigorous shaking or swirling). The stability of an oil in water emulsion is increased by the addition of a suitable emulsifying agent, such as soap solution. Soap contains Sodium salt of long chain Aliphatic Carboxylic acids with Carboxyl group as the polar group, which decreases the inter-facial tension between oil and water. Hence, oil mixes with water in greater quantity and emulsification takes place. The minimum concentration of soap required for complete emulsification is called 'optimum concentration'. Any other amount, less or more than this optimum amount, does not cause effective stabilisation.

APPARATUS REQUIRED

Six test tubes, five droppers, test tube stand, one glass rod and one stopwatch.

CHEMICALS REQUIRED

Soap/detergent — 0.1g, mustard oil, linseed oil, castor oil and machine oil — 1 mL each.

PROCEDURE

1. Dissolve about 0.2 g of soap/detergent in 2 mL distilled water in a micro test tube with vigorous shaking. Heat the contents of the micro test tube if needed. Label it as 'A'.
2. Take four micro test tubes. Mark these as B, C, D and E and to each of the test tube, add 0.5 mL distilled water, followed by one drop of mustard oil in test tube B, linseed oil in test tube C, castor oil in test tube D and machine oil in test tube E.
3. Shake test tube B vigorously for five minutes, keep it in a test tube stand, and simultaneously, start the stopwatch. Record the time taken for the separation of the two layers.
4. Repeat the same procedure with test tubes C, D and E and record the time taken for the separation of the layers in each case.
5. Now, add two drops of soap/detergent solution from test tube 'A' into each test tube (B, C, D and E). Shake each test tube for five minutes and record the time of separation of the layers in each case again.
6. Record your observations in Table 8.1 as given below.

Table 8.1

Emulsification of Different Oils by Soap/Detergent

| Test tube | Name of oil used for emulsification | Time taken for the separation of layers | |
|-----------|-------------------------------------|---|---------------------|
| | | Without soap/detergent | With soap/detergent |
| B | | | |
| C | | | |
| D | | | |
| E | | | |

PRECAUTIONS

1. Add equal drops of soap/detergent solution to all test tubes.
2. To minimise the error in recording the time required for the separation of layers in different systems, shake all the test tubes for identical time span.
3. Start the stopwatch immediately after shaking the emulsion and stop it immediately when the two layers separate.

CHEMICAL KINETICS

Chemical kinetics is the study of the rates of the chemical reactions and the factors which affect these rates.

RATE OF REACTION

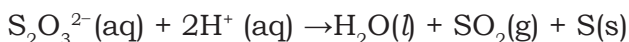
Rate of reaction can be measured in terms of either decrease in concentration of any one of the reactants or increase in concentration of any one of the products with time.

Factors, such as concentration, temperature and catalyst affect the rate of reaction.

Sodium thiosulphate reacts with Hydrochloric acid and produces a colloidal solution of Sulphur, which makes the solution translucent. The reaction occurs as follows:



Ionic form of the above reaction is written as:



The property of the colloidal solution of Sulphur to make the solution translucent is used to study the rate of precipitation of Sulphur. The rate of precipitation of Sulphur increases with an increase in the concentration of the reacting species or with an increase in the temperature of the system. With an increase in the concentration of reactants, the number of molecular collisions per unit time between the reacting species increases and consequently, the chances of product formation increase. This results in an increase in the rate of precipitation of Sulphur. Similarly, on increasing the temperature, the kinetic energy of the reacting species increases, so the number of effective collisions that result in the formation of products increase leading to a faster rate of reaction.

EXPERIMENT 9.1

To study the effect of change in concentration on the rate of reaction between Sodium thiosulphate and Hydrochloric acid.

APPARATUS REQUIRED

Seven micro test tubes, micro beakers, test tube stand, two volumetric flasks (25 mL), two pasteur pipettes and stopwatch.

CHEMICALS REQUIRED

0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ and 1 M HCl

PROCEDURE

1. Prepare 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ solution and 1 M HCl solution in two 25 mL volumetric flasks.
2. Take seven micro test tubes marked as 1, 2, 3, 4, 5, 6 and 7 in a test tube stand.
3. In micro test tube 1, transfer seven drops of 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$, six drops in test tube 2, five in test tube 3, and so on.
4. Make up the volume of each test tube equal to that in test tube 1 by transferring one drop of water to test tube 2, two drops of water to test tube 3, and so on, as indicated in Table 1.
5. Take a white paper and mark X on it.
6. Take test tube 1 and transfer two drops of 1M HCl in it and start the stop water. Hold it over the mark 'X' on the white paper. Swirl it gently.
7. When the visibility of the X mark disappears due to the formation of colloidal Sulphur, stop the water and record the time.
8. Repeat the steps 5, 6 and 7 with the remaining test tubes marked 2 to 7.

NOTE

You may use micro beakers instead of test tubes, using three times the quantities mentioned above.

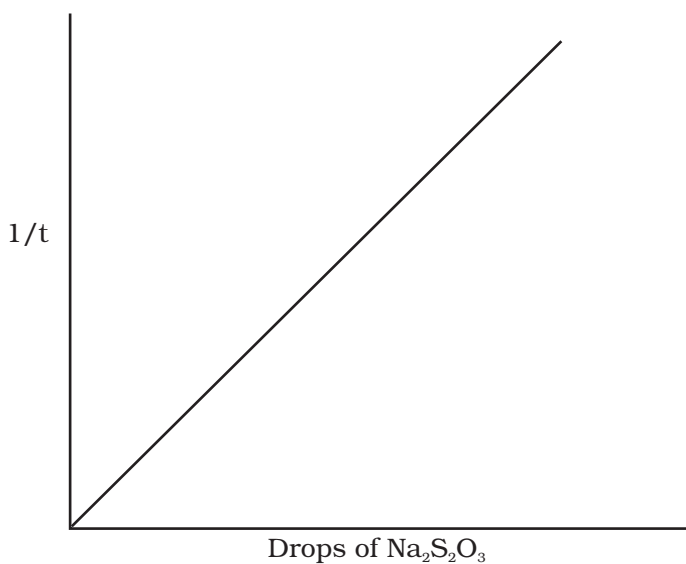
Table 9.1
Observation Table

| Test tube No. | Drops of 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ | Drops of H_2O | Drops of 1M HCl | Time taken for the disappearance of mark 'X' |
|---------------|--|-------------------------------|-----------------|--|
| 1. | 7 | — | 2 | |
| 2. | 6 | 1 | 2 | |
| 3. | 5 | 2 | 2 | |
| 4. | 4 | 3 | 2 | |
| 5. | 3 | 4 | 2 | |
| 6. | 2 | 5 | 2 | |
| 7. | 1 | 6 | 2 | |

Plot a graph between volume of $\text{Na}_2\text{S}_2\text{O}_3$ added (x-axis) and inverse of time ($1/t$) required for the mark to disappear (y-axis).

RESULT

Draw conclusions from the graph.



EXPERIMENT 9.2

To study the effect of temperature on the rate of the reaction between Sodium thiosulphate and Hydrochloric acid.

APPARATUS REQUIRED

Stopwatch, thermometer, two volumetric flasks (25 mL), micro beakers (10 mL), spirit lamp/bunsen burner, China dish and tripod stand.

CHEMICALS REQUIRED

0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ and 1 M HCl

PROCEDURE

1. Take 3 mL 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ in a 10 mL beaker and add 1 mL 1.0 M HCl to it.
2. Record the time taken for the disappearance of mark 'X' at room temperature as in Experiment 9.1.
3. Heat the stock solutions of 0.2 M $\text{Na}_2\text{S}_2\text{O}_3$ and 1.0 M HCl to 30°C in a water bath. Mix the quantities of these solutions as mentioned in Step 1 and record the time taken for the disappearance of mark 'X'.
4. Repeat the experiment with stock solutions heated to 40°C and 50°C.

Table 9.2
Observation Table

| S. No. | Temperature of the reaction mixture (°C) | Volume of $\text{Na}_2\text{S}_2\text{O}_3$ (mL) | Volume of HCl (mL) | Time taken for disappearance of mark 'X' in seconds (s) |
|--------|--|--|--------------------|---|
| 1. | Room temp. (°C) | 3.0 | 1.0 | |
| 2. | 30°C | 3.0 | 1.0 | |
| 3. | 40°C | 3.0 | 1.0 | |
| 4. | 50°C | 3.0 | 1.0 | |

Plot a graph between temperature (x-axis) and inverse of time for the mark to become invisible ($1/t$) (y-axis)

RESULT

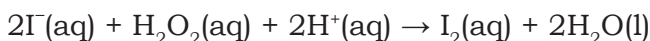
Draw conclusions from the graph regarding the effect of temperature on the rate of the reaction.

EXPERIMENT 9.3

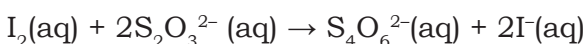
To study the effect of variation of concentration on the rate of reaction of Iodide ions with Hydrogen peroxide at room temperature (clock reaction).

THEORY

The reaction between Iodide ions and Hydrogen peroxide occurs in the acidic medium and can be represented in the following manner:



In this reaction, Hydrogen peroxide oxidises Iodide ions (I^{-}) to molecular Iodine. If calculated amount of Sodium thiosulphate is added in the presence of starch solution as an indicator to the above reaction mixture, the liberated Iodine reacts with Thiosulphate ions as fast as it is formed and is reduced back to Iodide ions till all Thiosulphate ions are oxidised to Tetrathionate ions.



After the complete consumption of Thiosulphate ions, the concentration of liberated Iodine increases rapidly and forms intense blue complex with starch. The time required to consume a fixed amount of the Thiosulphate ions is reproducible. Since the time for the appearance of colour is recorded, the reaction is called 'clock reaction'.

APPARATUS REQUIRED

Three volumetric flasks (25 mL), five micro beakers (10 mL), four droppers and stopwatch.

CHEMICALS REQUIRED

1.0 M H_2SO_4 , 0.1 M KI, 0.04 M $\text{Na}_2\text{S}_2\text{O}_3$, 3% H_2O_2 and 1% starch solution.

PROCEDURE

1. Label 0.04 M $\text{Na}_2\text{S}_2\text{O}_3$ as solution A.
2. Label 0.1 M KI as solution B.
3. Transfer 5 mL of 3% H_2O_2 , 5 mL of 1.0 M H_2SO_4 and two drops of freshly prepared 1% starch solution in a 25 mL volumetric flask. Label it as solution C.

- Take five micro beakers (10 mL) and label them as 1, 2, 3, 4 and 5.
- Make the additions in each beaker according to the table given below by following the sequence of addition.
- Start the stopwatch when the first drop of solution C is added to the contents in the flask.
- Note the time when blue colour appears.

Table 9.3
Observation Table

| Beaker No. | No. of drops of | | | | |
|------------|--|-----------------|----------------------|---------------------------------------|----------|
| | Solution A ($\text{Na}_2\text{S}_2\text{O}_3$) | Solution B (KI) | H_2O | Solution C (H_2O_2) | Time (s) |
| 1. | 1 | 10 | 0 | 10 | |
| 2. | 1 | 8 | 2 | 10 | |
| 3. | 1 | 6 | 4 | 10 | |
| 4. | 1 | 4 | 6 | 10 | |
| 5. | 1 | 2 | 8 | 10 | |

Plot a graph between the inverse of time ($1/t$) (y axis) and number of drops of KI (concentration of KI) (x axis).

RESULT

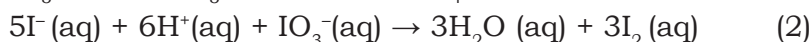
Draw conclusions from the graph regarding the effect of increasing the concentration of KI on the rate of reaction.

EXPERIMENT 9.4

To study the rate of reaction between Potassium iodate (KIO_3) and Sodium sulphite (Na_2SO_3) using starch solution as indicator.

THEORY

The reaction between KIO_3 and Na_2SO_3 involves the formation of Iodide ions, which are oxidised in acidic medium by IO_3^- ions to Iodine. The overall reaction proceeds in the following two steps.



The evolved Iodine produces blue colour with the starch solution. This reaction, like the earlier reaction, is also known as 'clock reaction'.

APPARATUS REQUIRED

Pasteur pipette, five micro beakers (10 mL), two beakers (50 mL), spirit lamp/bunsen burner, tripod stand and stopwatch.

CHEMICALS REQUIRED

1% starch solution, 0.2% Na_2SO_3 solution, 0.2% KIO_3 and concentrated H_2SO_4

PROCEDURE

- For 0.2% KIO_3 solution, dissolve 0.1 g of KIO_3 in 10 mL water containing 0.6 mL of 0.5 M H_2SO_4 and make up to 50 mL by adding water.
 - For 0.2% Na_2SO_3 solution, dissolve 0.05 g of Na_2SO_3 in 25 mL water.
 - For 1% starch, dissolve 0.25 g starch (soluble) in 25 mL boiled water.
- In a 10 mL beaker, transfer 1 mL of KIO_3 solution, 1 mL of 1% starch solution and 3 mL of water to make 5 mL solution. Mix it well.

- Transfer 5 mL 0.2% Na_2SO_3 solution in the above beaker and record the time required for the appearance of blue colour.
- Wash the beaker and repeat Step 2 and 3 as per the details given in the following table.

Table 9.4
Observation Table

| S. No. | KIO_3 (mL) | Starch (mL) | Water (mL) | Na_2SO_3 (mL) | Time taken (s) |
|--------|---------------------|-------------|------------|-------------------------------|----------------|
| 1. | 1.0 | 1.0 | 3.0 | 5.0 | |
| 2. | 2.0 | 1.0 | 2.0 | 5.0 | |
| 3. | 3.0 | 1.0 | 1.0 | 5.0 | |
| 4. | 4.0 | 1.0 | — | 5.0 | |
| 5. | 4.5 | 0.5 | — | 5.0 | |

Plot a graph between the volume of KIO_3 solution (concentration of KIO_3) on x -axis and inverse of the time taken for the appearance of blue colour (rate of the reaction) on y -axis.

RESULT

Draw conclusions from the graph.

PRECAUTIONS

- As Sodium sulphite is likely to be easily oxidised in air, therefore, always use its fresh solution.
- Use a freshly prepared starch solution.

CHAPTER 10

THERMOCHEMISTRY

Most of the reactions are carried out at atmospheric pressure, which remains nearly constant. Heat changes measured for reactions carried out under a constant pressure (which may or may not be equal to the atmospheric pressure) are enthalpy changes. Enthalpy changes are directly related to temperature changes by the relation:

$$\begin{aligned}\Delta H &= q_p \\ &= M \cdot c_p \cdot \Delta T \quad \dots\dots\dots (1)\end{aligned}$$

Where, m = Mass

c_p = Specific heat capacity at constant pressure

ΔT = Change in temperature

Measurement of heat changes is generally carried out in metallic vessels called 'calorimeters'. Metallic calorimeters are not used for studying thermochemical changes because metals may react with the substances used. Stainless steel or gold-plated Copper calorimeters may be used. Reactions may also be carried out in glass beakers placed in thermos flask or in a thermally-insulated box or in a styrofoam cup.

During the measurement of heat changes, calorimeter, thermometer and stirrer also absorb some heat. This amount of heat should also be known. It may be calculated from the heat capacity of calorimeter C , which may be determined experimentally. In case of a glass vessel (e.g., beaker), the heat capacity for that part of the beaker, which is actually in contact with the reaction mixture, is found. This is so because the thermal conductivity of glass is low and only the area of glass vessel in contact with the liquid absorbs the maximum heat. The heat capacity of the calorimeter, therefore, varies with the volume of the liquid taken in it.

Method of mixtures is used to determine the heat capacity of calorimeter. For this purpose, a known volume of hot water at a known temperature is added to a known volume of water contained in the calorimeter at room temperature. Since energy is conserved, the heat gained by the calorimeter and cold water should be equal to the heat lost by the hot water. Thus, we can write the following equation:

Heat gained by cold water and calorimeter = Heat lost by hot water

$$C(t_m - t_c) + m_c c_p (t_m - t_c) = m_h c_p (t_h - t_m) \quad (2).$$

Where:

t_c , t_h and t_m = Temperature of the cold water, hot water and the mixture, respectively

C = Heat capacity of the calorimeter

c_p = Specific heat capacity of water

m_c and m_h = Masses of the cold and hot water, respectively

From equation (2)

$$C = \frac{(m_h c_p t_h - t_m) - (m_c c_p t_m - t_c)}{(t_m - t_c)} \dots\dots\dots (3)$$

For water, $c_p = 4.184 \text{ J } ^\circ\text{C}^{-1} \text{ g}^{-1}$

Since, the density of water is quite close to 1 g mL^{-1} , its mass in grams can be taken to be equal to its volume in millilitre. If V_1 and V_2 are the volumes of cold water and hot water, respectively, the equation (3) may be written as:

$$C = \frac{V_h \times 4.184(t_h - t_m) - V_c \times 4.184(t_m - t_c)}{(t_m - t_c)} \dots\dots\dots (4)$$

EXPERIMENT 10.1

To determine the enthalpy of the solution of Potassium nitrate (1:200 molar ratio).

THEORY

Solution formation is often accompanied by enthalpy change. Enthalpy of a solution is the amount of heat liberated or absorbed when one mole of a solute is dissolved in a specified amount of solvent (usually water) under a constant pressure. The amount of solvent (water) is expressed in terms of its number of moles (n). The ratio of the amount of solute and solvent is expressed as 1: n . For example, the ratio 1:200 means that 1 mole solute is dissolved in 200 mole solvent.

APPARATUS REQUIRED

Calorimeter (50 mL beaker inserted in LDPE foam case), digital thermometer (0.1°C), stirrer (glass), beaker (50 mL), tripod stand, wire gauge, spirit lamp/burner and pipette (5 mL).

CHEMICALS REQUIRED

Potassium nitrate (KNO_3)

PROCEDURE

I. DETERMINATION OF HEAT CAPACITY OF CALORIMETER

1. Take a beaker of 50 mL capacity. Clean and dry it. To the above beaker, add 10 mL water with the help of a 5 mL pipette provided in the Kit.
2. Place the beaker in an LDPE foam case. Insert the stirrer and thermometer in the lid. Adjust the position of the thermometer in a way that it passes through the loop of the stirrer. Adjust the height of the thermometer to fit the case.
3. Record the temperature of water in the calorimeter and check after two minutes for its constant value. Let it be t_c .
4. In another 50 mL beaker, take a small amount of distilled water and rinse it. Add 10 mL distilled water with the help of a 5 mL pipette.
5. Heat the above beaker on a wire gauge placed on a tripod stand by 10°C as compared to the water in the calorimeter.

- Remove the beaker from the tripod stand. Wait for one minute. Note the exact temperature of the hot water (t_h) and quickly transfer it to the beaker in the calorimeter. Place the lid along with the stirrer and thermometer, stir the mixed water and note its constant temperature (t_m).

CALCULATIONS

Calculate the heat capacity of the calorimeter by using the equation.

$$(m_c \times 4.184 + C) (t_m - t_c) = m_h \times 4.184 \times (t_h - t_m)$$

Where m_c and m_h are masses of cold and hot water, respectively.

Taking the density of water as 1.0 g mL^{-1} , the above relation may be written as:

$$(V_c \times 4.184 + C) (t_m - t_c) = V_h \times 4.184 \times (t_h - t_m)$$

Where,

C = Heat capacity of the calorimeter

t_c = Temperature of cold water

t_h = Temperature of hot water

t_m = Temperature of mixed water

V_h = Volume of hot water in mL

V_c = Volume of cold water in mL

II. DETERMINATION OF ENTHALPY OF SOLUTION

Transfer 20 mL distilled water in a clean and dry 50 mL beaker placed in the cavity of an LDPE foam case and cover it with a lid, in which a thermometer and a stirrer have been fixed. Record the constant temperature. Let the temperature be t_1 °C. Finely powder Potassium nitrate with the help of mortar and pestle and weigh exactly 0.56 g.

Open the lid of the LDPE foam case and quickly transfer the weighed quantity of Potassium nitrate and replace the lid.

Stir the contents of the calorimeter, till the temperature becomes constant. Record this temperature and let it be t_2 °C.

OBSERVATIONS

Mass of water $m_w = 20 \text{ g}$.

Mass of Potassium nitrate = 0.56 g.

Temperature of water = t_1 °C.

Temperature of the solution = t_2 °C.

CALCULATIONS

Change in temperature $|t_2 - t_1|$

(Absolute value)

ΔH is positive, if t_2 is less than t_1 (Endothermic)

ΔH is negative, if t_2 is more than t_1 (Exothermic)

$$q = (m_w \times 4.184 +) \times |\Delta t|$$

By dissolving 0.56 g KNO_3 , heat absorbed = q

$$\text{On dissolving 101g (1 mol KNO}_3\text{) } \Delta_{\text{sol.}} H = \frac{q \text{ J} \times 101 \text{ g}}{0.56 \text{ g}} \text{ mol}^{-1}$$

PRECAUTIONS

1. Use clean and dry beaker as calorimeter.
2. The beaker which is used for heating water should be first rinsed with water.
3. Transfer the hot water into the calorimeter quickly.
4. Potassium nitrate should be finely powdered before weighing, so that it dissolves in water completely and quickly.

NOTE

The above experiment can be performed by taking 0.82 g Potassium dichromate (1:400) or 0.30 g Ammonium chloride (1:200).

EXPERIMENT 10.2

To determine the enthalpy of neutralisation of HCl and NaOH.

APPARATUS REQUIRED

Calorimeter, two beakers (50 mL), thermometer (0.1°C or digital) stirrer, burner/spirit lamp and tripod stand with wire gauge and pipette (5 mL).

CHEMICALS REQUIRED

1M HCl solution and 1M NaOH solution.

PROCEDURE

I. DETERMINATION OF HEAT CAPACITY OF THE CALORIMETER

This may be determined by following the procedure as given in Experiment 10.1.

II. DETERMINATION OF ENTHALPY OF NEUTRALISATION

1. Take 10 mL 1M HCl solution in a clean and dry 50 mL beaker kept in an LDPE foam case. Take another 50 mL clean beaker. Rinse it with 1 M NaOH solution and then take 10 mL 1 M NaOH solution in it.
2. Note the temperatures of both the solutions. Let these be $t_1^\circ\text{C}$ and $t_2^\circ\text{C}$.
3. Transfer the NaOH solution to the calorimeter. Put the lid with stirrer and thermometer in place.
4. Stir the solutions rapidly and note the final temperature of the mixture when it becomes constant. Let it be t_f .

CALCULATIONS

Calculation of the heat capacity (C) of the calorimeter as done in Experiment 10.1.

CALCULATIONS OF ENTHALPY OF NEUTRALISATION

$$\text{Initial temperature} = \frac{t_1 + t_2}{2} = t_i$$

$$\text{Final temperature of the solution} = t_f$$

$$\text{Rise in temperature} = \Delta t = (t_f - t_i) ^\circ\text{C}.$$

Heat produced during neutralisation = $(m_w \times 4.184 + C) \Delta t$
(Taking the density of water as 1 g mL^{-1}) = $(20 \times 4.184 + C) \Delta t$
This is the quantity of heat produced when 10 mL of 1 M HCl is neutralised by 10 mL of 1 M NaOH.

Determine the heat produced when 1000 mL of 1M HCl is allowed to neutralise 1000 mL of 1M NaOH. This quantity would be 100 times the quantity obtained above.

Express the quantity of heat evolved in kJ/mol.

If the solutions used have molarity M each, the enthalpy of neutralisation in J/mol can be calculated from the relation.

$$\Delta_{\text{neut}} H = \frac{(20 \times 4.184 + C) \Delta t}{M} \times 100$$

RESULT

The enthalpy of neutralisation of HCl and NaOH is..... kJ mol⁻¹.

PRECAUTIONS

1. The molarities of HCl and NaOH solutions should be exactly equal.
2. The molarities of HCl and NaOH solutions may be between 1 M to 0.5 M, but must be known exactly.
3. The bulb of the thermometer should dip in the solution completely.
4. After transferring the hot water in determination of heat capacity, the lid of the calorimeter should be immediately replaced.

CHAPTER 11

ELECTROCHEMISTRY

EXPERIMENT 11.1

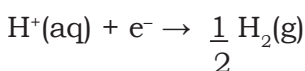
To study the variation of cell potential of the cell $\text{Zn} | \text{Zn}^{2+} || \text{Cu}^{2+} | \text{Cu}$ with change in the concentration of electrolytes at room temperature.

THEORY

The potential difference between two electrodes of a galvanic cell is called 'cell potential' and is measured in volts. It is the difference between the electrode potentials of cathode and anode. When no current is drawn from the cell, it is called electromotive force (emf) of the cell.

$$E_{\text{cell}} = E_{\text{cathode}} - E_{\text{anode}} \quad (1)$$

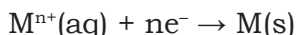
The potential of individual half-cells cannot be measured. We can measure the emf of a cell that gives the difference between two half-cell potentials. According to the International Union of Applied Chemistry (IUPAC) convention, standard Hydrogen electrode represented by $\text{Pt}, \text{H}_2(\text{g}, 1 \text{ bar}) / \text{H}^+(\text{aq}, 1 \text{ M})$ is assigned zero potential at all temperatures corresponding to the reaction.



Half-cell potentials are measured with respect to standard hydrogen electrode. A cell is constructed by taking standard hydrogen electrode as anode (reference half-cell) and the other electrode, under standard condition, of which the electrode potential is to be measured as cathode. Then, the cell potential is equal to the standard electrode potential of the other half-cell.

$$\begin{aligned} E_{\text{cell}}^{\circ} &= E_{\text{cathode}}^{\circ} - E_{\text{anode}}^{\circ} \\ &= E_{\text{cathode}}^{\circ} \\ &\text{because } E_{\text{anode}}^{\circ} = 0 \end{aligned}$$

For the electrode reaction of the type:



Nernst derived the following relation between concentration and the electrode potential.

$$E_{M^{n+}/M} = E^{\circ}_{M^{n+}/M} - \frac{RT}{nF} \ln \frac{[M]}{[M^{n+}]} \quad (2)$$

If the concentration of solid M is taken as unity, then equation (2) becomes

$$E_{M^{n+}/M} = E^{\circ}_{M^{n+}/M} - \frac{RT}{nF} \ln \frac{1}{[M^{n+}]} \quad (3)$$

Here R is the gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$), F is Faraday constant (96487 C mol^{-1}), T is the temperature in Kelvin and $[M^{n+}]$ is the molar concentration of the species, M^{n+} . $E_{M^{n+}/M}$ is the electrode potential at this concentration and $E^{\circ}_{M^{n+}/M}$ is its standard electrode potential.

For the cell



The Nernst equation is

$$E_{\text{cell}} = E^{\circ}_{\text{cell}} - \frac{2.303}{2F} \log \frac{[Zn^{2+}]}{[Cu^{2+}]}$$

$$E_{\text{cell}} = E^{\circ}_{\text{cell}} - 0.02955 \log \frac{C_1}{C_2}$$

Where, C_1 and C_2 are the molar concentrations of Zn^{2+} and Cu^{2+} ions, respectively.

In the following experiment, the variation in the cell potential of $Zn | Zn^{2+} || Cu^{2+} | Cu$ cell with concentration of electrolytes will be studied.

APPARATUS REQUIRED

Two micro test tubes, micro test tube stand and multimeter.

CHEMICALS REQUIRED

Zinc granules, Copper turnings, 1.0 M $ZnSO_4$ and 1.0 M $CuSO_4$ and saturated solution of KCl.

PROCEDURE

1. Take two micro test tubes. Label them as A and B.
2. In test tube A, put Cu turnings and 1.0 M CuSO_4 solution.
3. In test tube B, put Zn granules and 1.0 M ZnSO_4 solution.
4. Keep both the test tubes A and B in a micro test tube stand.
5. Connect both the test tubes by salt bridge made by filter paper dipped in KCl.
6. Measure the cell emf with the help of a multimeter, setting the knob of the multimeter at 20 V range. [Note that red lead should be inserted in the hole marked +ve and black in the hole marked COM.]
7. Repeat the procedure with change in the concentration of Copper sulphate or Zinc sulphate solution as given in the Table 11.1 and 11.2.

NOTE

For demonstration purpose, these solutions may be taken in two 50 mL beakers.

Table 11.1
Observation Table $[\text{Zn}^{2+}] = 1\text{M}$

| S. No. | $[\text{Cu}^{2+}]$ (M) | $\text{Log}[\text{Cu}^{2+}]$ | Obs. Ecell/V | Calculated Ecell/V |
|--------|---------------------------|------------------------------|--------------|-----------------------|
| 1. | 1.0 | 0.0 | | |
| 2. | 0.1 | -1 | | |
| 3. | 0.01 | -2 | | |

Table 11.2
 $[\text{Cu}^{2+}] = 1\text{M}$

| S. No. | $[\text{Zn}^{2+}]$ | $\text{Log}[\text{Zn}^{2+}]$ | Obs. Ecell/V | Calculated Ecell/V |
|--------|--------------------|------------------------------|--------------|-----------------------|
| 1. | 1.0 | 0 | | |
| 2. | 0.1 | -1 | | |
| 3. | 0.001 | -2 | | |

RESULT

.....

.....

.....

.....

PRECAUTIONS

Copper turnings and zinc granules should be dipped partially in the respective solutions.

APPENDIX

PREPARATION OF COMMON LABORATORY REAGENTS

I. Concentration Acids

| S. No. | Name | Approximate concentration | Specific gravity | Approximate amount per mL | Percentage by weight |
|--------|--------------------------------|---------------------------|------------------|---------------------------|----------------------|
| 1. | Acetic acid (glacial) | 17.6 M (17.6 N) | 1.06 | 1.06 g/mL | 99.5% |
| 2. | Concentrated Hydrochloric acid | 11.7 M (11.7 N) | 1.19 | 0.426 g/mL | 36.0 % |
| 3. | Concentrated Nitric acid | 15.6 M (15.6 N) | 1.42 | 0.998 g/mL | 69.5% |
| 4. | Concentrated Sulphuric acid | 18 M (36.0 N) | 1.84 | 1.76 g/mL | 98.0% |

II. Dilute Acids

| S. No. | Name | Concentration | Method of preparation |
|--------|--------------------------|---------------|--|
| 1. | Dilute Acetic acid | 5 M (5 N) | Dilute 29 mL of glacial Acetic acid with distilled water and make up the volume to 100 mL. |
| 2. | Dilute Hydrochloric acid | 5 M (5 N) | Add 43 mL of concentrated HCl in distilled water and make up the volume to 100 mL. |
| 3. | Dilute Nitric acid | 5 M (5 N) | Add 32 mL of concentrated Nitric acid to distilled water and make up the volume to 100 mL. |

| | | | |
|----|-----------------------|-------------|---|
| 4. | Dilute Sulphuric acid | 2.5 M (5 N) | Pour 14 mL of concentrated Sulphuric acid slowly and with constant stirring in 50 mL of distilled water. Cool and make up to the volume 100 mL. |
|----|-----------------------|-------------|---|

III. Bases

| S. No. | Name | Concentration | Method of preparation |
|--------|-----------------------------------|---------------|--|
| 1. | Ammonia solution (liquor ammonia) | 15 M (15 N) | As supplied |
| 2. | Dilute Ammonia solution | 2 M (2 N) | Pour 13 mL of the concentrated Ammonia solution in distilled water and make up the volume to 100 mL. |
| 3. | Sodium hydroxide | 5 M (5 N) | Dissolve 20 g Sodium hydroxide pellets in 100 mL of distilled water. |

IV. Other Reagents

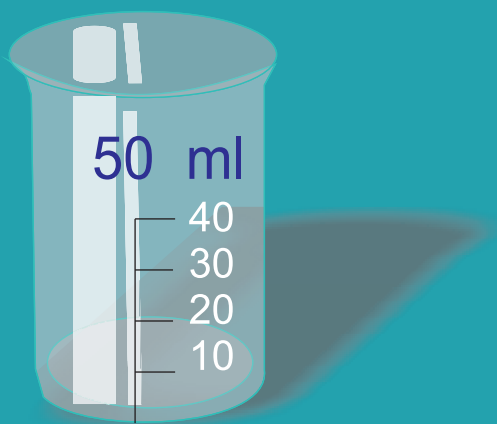
| S. No. | Name | Concentration | Molar mass (g mol^{-1}) | Method of preparation |
|--------|-------------------|---------------|------------------------------------|--|
| 1. | Ammonium acetate | 2 M (2 N) | 77 | Dissolve 15.4 g of the salt in distilled water and dilute to 100 mL. |
| 2. | Ammonium chloride | 5 M (5 N) | 53.5 | Dissolve 26.8 g of the salt in distilled water and dilute to 100 mL. |

| | | | | |
|----|--|-------------------|-------|--|
| 3. | Ammonium carbonate | 1.7 M (3.5 N) | 96 | Dissolve 16.3 g of Ammonium carbonate in 14 mL liquor Ammonia and make up the solution to 100 mL with distilled water. |
| 4. | Ammonium molybdate | — | 195.9 | Dissolve 10 g of the salt in a mixture of 10 mL of liquor Ammonia solution and add 25 g Ammonium nitrate and dilute it to 100 mL with distilled water. |
| 5. | Ammonium oxalate | 0.5 M (1 N) | 142 | Dissolve 7.1 g of the salt in distilled water and make up to 100 mL. |
| 6. | Ammonia sulphate | 1 M (2 N) | 132 | Dissolve 13.2 g of the salt in distilled water and dilute to 100 mL. |
| 7. | Barium chloride $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ | 0.5 M (0.5 N) | 244 | Dissolve 6.1 g of the salt in distilled water and dilute to 100 mL. |
| 8. | Bromine water | approx. saturated | 160 | Add 0.2 mL Bromine in 10 mL of distilled water and shake the mixture well. Keep it in a dark bottle. |

| | | | | |
|-----|---|--------------|-------|---|
| 9. | Chlorine water | — | 71 | Dissolve 1.5 g Potassium chlorate in 150 mL water and add about 10 mL of concentrated HCl slowly and with stirring. Store it in a stoppered bottle. |
| 10. | Copper sulphate | 14% | 249.5 | Dissolve 14 g of the salt in distilled water and make up the volume to 100 mL. |
| 11. | Dimethyl glyoxime | 1% | — | Dissolve 0.1 g of the solid in 10 mL Ethyl alcohol. |
| 12. | Disodium hydrogen $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ | 0.3 M (N) | 358 | Dissolve 12 g of the salt in distilled water and make phosphate up the volume 100 mL. |
| 13. | Ferric chloride $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ | 0.33 M (1 N) | 270 | Dissolve 9 g of the salt in distilled water containing 1 mL of concentrated Hydrochloric acid and make up the volume to 100 mL. |
| 14. | Iodine solution | — | 254 | Dissolve 1.0 g of iodine crystals in a solution of 2 g Potassium iodide in minimum amount of water and dilute the solution to 100 mL. |

| | | | | |
|-----|---|-----------------|-----|--|
| 15. | Lead acetate (CH ₃ COO) ₂ Pb | 0.5 M (N) | — | Dissolve 20 g of solid salt in 50 mL of distilled water containing 1.5 mL Acetic acid and make up the volume to 100 mL with distilled water. |
| 16. | Lime water Ca(OH) ₂ | 0.02 M (0.04 N) | 74 | Shake 0.2-0.3 g of Calcium hydroxide with 100 mL distilled water, filter the solution after some time and keep it in a reagent bottle. The bottle should be securely stoppered in order to protect the reagent from CO ₂ of atmosphere. |
| 17. | Litmus solution (blue) | — | — | Dissolve 1 g of litmus in distilled water and make the volume to 100 mL |
| 18. | Litmus solution (red) | — | — | To the blue litmus solution add 1 drop of dilute Hydrochloric acid. |
| 19. | Methyl orange | — | — | Dissolve 0.1 g of the solid in 100 mL of distilled water. |
| 20. | Potassium chromate K ₂ CrO ₄ | 0.25 M (0.5 N) | 194 | Dissolve 4.9 g of the salt in distilled water and make up the volume to 100 mL. |

| | | | | |
|-----|------------------------|----------------|-----|--|
| 21. | Potassium dichromate | 0.15 M (1 N) | 294 | Dissolve 4.9 g of the salt in distilled water and make up the volume to 100 mL. |
| 22. | Potassium ferrocyanide | 0.15 M (0.5 N) | 368 | Dissolve 4.6 g of the salt in distilled water and dilute to 100 mL. |
| 23. | Potassium iodide | 0.5 (0.5 N) | 166 | Dissolve 8.3 g of the salt in distilled water and make up the volume to 100 mL. |
| 24. | Potassium permanganate | 0.06 M (0.3 N) | 158 | Dissolve 1 g of the salt in 100 mL distilled water. Heat the solution and filter it through glass wool. |
| 25. | Potassium thiocyanate | 0.5 M (0.5 N) | 97 | Dissolve 4.9 g of the salt in distilled water and make up the volume to 100 mL. |
| 26. | Phenolphthalein | | 1% | Dissolve 1 g of the solid in 100 mL of Ethyl alcohol. |
| 27. | Silver nitrate | 0.1 M | 170 | Dissolve 17 g of the salt in 250 mL distilled water and store it in a brown bottle. |
| 28. | Starch | | | Prepare a paste of about 1.0 g of soluble starch in cold water and pour it gradually in 100 mL of boiling water with constant stirring. Boil it for 10 minutes and cool. |



13166

विद्यया ऽ मृतमश्नुते



एन सी ई आर टी
NCERT

राष्ट्रीय शैक्षिक अनुसंधान और प्रशिक्षण परिषद्
NATIONAL COUNCIL OF EDUCATIONAL RESEARCH AND TRAINING

ISBN 978-93-5007-861-7