



# **CHEMISTRY EXPERIMENTS USER GUIDE**

**FOR**

**SENIOR SIX**

© 2022 Rwanda Basic Education Board

*All rights reserved*

*This book is the property of the Government of Rwanda.*

*Credit must be provided to REB when the content is quoted*

## FOREWORD

Dear teacher,

Rwanda Basic Education Board (REB) is honoured to present Chemistry experiments user guide for Senior six. This book will serve as a guide to competence-based teaching and learning to ensure consistency and coherence in the learning of Chemistry.

In this book, special attention is paid to experiments that facilitate the learning process in which students can manipulate concrete apparatuses and use chemicals to carry out appropriate experiments, develop ideas, and make adequate interpretations and conclusions during activities performed individually or in pairs/ small groups.

In competence-based curriculum, experiments open students' minds and provide them with the opportunities to interact with the world, use available tools, collect data and effectively model real life problems.

For efficiency use of this user guide book, your role as a teacher is to:

- Plan your lessons and prepare appropriate teaching materials (chemicals and reagents),
- Organize group discussions for students considering the importance of social constructivism,
- Engage students through active learning methods,
- Provide supervised opportunities for students to develop different competences by giving tasks which enhance critical thinking, problem solving, research, creativity and innovation, communication, and cooperation,
- Support and facilitate the learning process by valuing students' contributions in the practical activities,
- Guide students towards the harmonization of their findings,
- Encourage individual, peer and group evaluation of the work done and use appropriate competence-based assessment approaches and methods.

To facilitate you in your teaching activities, the content of this booklet is self-explanatory so that you can easily use it. It is divided in 3 parts:

**The part I:** Explains the structure of this book and gives you the general introduction on laboratory experiments.

**The part II:** Gives the list of apparatuses and chemicals needed to perform experiments in the booklet of chemistry.

**The part III:** Details the setup of experiments, the procedures to be followed when performing experiments, interpretations of results and conclusions.

I wish to sincerely extend my appreciation to the people who contributed towards the development of this guide, AIMS – TTP in collaboration with MasterCard Foundation who provided technical and financial support and REB staff particularly those from the Mathematics and Science Subjects Unit in the Curriculum Teaching and Learning Resources Department. Special appreciation goes also to teachers and independent experts in education who supported the exercise throughout the process. Any comment or contribution would be welcome for the improvement of this booklet for next versions.

**Dr. MBARUSHIMANA Nelson**

**Director General, REB**

## ACKNOWLEDGEMENT

I wish to express my appreciation to all the people who played a major role in the development of this chemistry experiments user guide for senior six. It would not have been successful without their active participation.

Special thanks are given to, AIMS staff, IEE staff, URCE Lecturers, independent people, teachers, illustrators, designers and all other individuals whose efforts in one way or the other contributed to the success of the development of this user guide.

I owe gratitude to the Rwanda Basic Education Board staff particularly those from Mathematics and Science subjects Unit in the CTLR Department who were involved in the whole process of the development work of this user guide.

Finally, my word of gratitude goes to AIMS – TTP in partnership with MasterCard Foundation for their support in terms of human and financial resources towards the development of this guide which will strengthen STEM teaching hence improving the quality of education in Rwandan schools.

**Joan MURUNGI**

**Head of CTLR Department**

## LIST OF ACRONYMS

<b>CBC</b>	: Competence-based curriculum
<b>ICT</b>	: Information Communication Technology
<b>Lab</b>	: Laboratory
<b>STEM</b>	: Science, Technology, Engineering and Mathematics
<b>KBC</b>	: Knowledge Based Curriculum
<b>SET</b>	: Science and Elementary Technology
<b>UR-CE</b>	: University of Rwanda- College of Education
<b>CTRLR</b>	: Curriculum, Teaching and Learning Resources
<b>REB</b>	: Rwanda Basic Education Board
<b>AIMS-TTP</b>	: African Institute for Mathematical Sciences-Teacher Training Program

# Table of Content

<b>FOREWORD</b> .....	<b>iii</b>
<b>ACKNOWLEDGEMENT</b> .....	<b>v</b>
<b>LIST OF ACRONYMS</b> .....	<b>vi</b>
<b>PART 1: GENERAL INTRODUCTION</b> .....	<b>1</b>
1. Laboratory experiments in the competence based curriculum .....	1
2. Type of laboratory experiments .....	2
3. Organization, analysis, and interpretation of data .....	3
4. Organizing lab experiments .....	3
5. Safety rules and precautions during lab experiments .....	7
6. Guidance on the management of lab materials: storage management, repairing and disposal of lab equipment .....	11
7. Student experiment worksheet .....	13
8. Report template for learner .....	14
<b>PART 2: LIST OF MAIN KIT ITEMS AND LAB MATERIALS NEEDED IN SCHOOLS</b> .....	<b>15</b>
<b>PART 3: DETAILED EXPERIMENTS FOR SENIOR SIX</b> .....	<b>32</b>
<b>UNIT 1: PROPERTIES AND USES OF TRANSITION METALS</b> .....	<b>33</b>
EXPERIMENT 1.1: Ligand exchange reaction .....	33
EXPERIMENT 1.2: Preliminary and confirmatory tests for transition metal ions ( $\text{Ni}^{2+}$ , $\text{Fe}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Mn}^{2+}$ , $\text{Cr}^{3+}$ , $\text{Zn}^{2+}$ , $\text{Co}^{2+}$ , $\text{Cu}^{2+}$ ) .....	37
<b>UNIT 2: EXTRACTION OF METALS</b> .....	<b>45</b>
EXPERIMENT 2.1: Extraction of copper .....	45
<b>UNIT 5: DERIVATIVES OF BENZENE</b> .....	<b>48</b>
EXPERIMENT 5.1: Experiment to test the acidity of phenol in comparison to alcohols and carboxylic acids .....	48
EXPERIMENT 5.2: Experiment to test for the presence of phenol in a solution .....	53
EXPERIMENT 5.3: Reaction of phenol with bromine .....	57

EXPERIMENT 5.4: Test and compare alkalinity of phenyl amines, aliphatic amines and ammonia.....	60
EXPERIMENT 5.5: Reactions of phenyl amines with water, hydrochloric acid and phenol .....	64
<b>UNIT 6: POLYMERS AND POLYMERISATION .....</b>	<b>68</b>
EXPERIMENT 6.1: Preparation of casein plastic from milk .....	68
EXPERIMENT 6.2: Laboratory preparation of phenol-methanal polymer (bakelite) .....	70
<b>UNIT 7: SOLVENT EXTRACTION AND COLLIGATIVE PROPERTIES .....</b>	<b>73</b>
EXPERIMENT 7.1: Solvent extraction .....	73
EXPERIMENT 7.2: Freezing point depression .....	77
EXPERIMENT 7.3: Boiling point elevation.....	82
EXPERIMENT 7.4: Illustration of the osmotic pressure.....	85
EXPERIMENT 7.5:Laboratory fractional distillation of ethanol and water .....	88
<b>UNIT 9: pH OF ACIDIC AND ALKALINE SOLUTIONS.....</b>	<b>92</b>
EXPERIMENT 9.1: Preparation of solutions and pH measurement.....	92
EXPERIMENT 9.2: Hydrolysis of salts .....	94
EXPERIMENT 9.3: Preparation and properties of a buffer solution	98
<b>UNIT 10: INDICATORS AND TITRATION CURVES .....</b>	<b>102</b>
EXPERIMENT 10.1: Effectiveness of indicators in titrations.....	102
<b>UNIT 11: SOLUBILITY AND SOLUBILITY PRODUCT CONSTANT (K<sub>sp</sub>) FOR SPARINGLY SOLUBLE SALTS .....</b>	<b>106</b>
EXPERIMENT 11.1: Simple determination of the solubility product of a sparingly soluble compound (calcium hydroxide) .....	106
<b>UNIT 12: ELECTROCHEMICAL CELLS AND APPLICATIONS .....</b>	<b>111</b>
EXPERIMENT 12.1: Working of the galvanic cell.....	111



<b>UNIT 13: FACTORS THAT AFFECT THE RATE OF THE REACTION.....</b>	<b>114</b>
EXPERIMENT 13.1: Illustration of how different reactions have different rates .....	114
EXPERIMENT 13.2: Factors that affect the rates of reactions .....	116
<b>UNIT 14: RATE LAWS AND MEASUREMENTS .....</b>	<b>128</b>
EXPERIMENT 14.1: Measure of the rate of reaction by observing the colour change.....	128
EXPERIMENT 14.2: Measure of the rate of reaction by observing volumes changes.....	131
EXPERIMENT 14.3: Measure of the rate of reaction by observing mass changes.....	133
EXPERIMENT 14.4: Experimental determination of the reaction order using the graph .....	137
<b>REFERENCES .....</b>	<b>141</b>

## PART 1: GENERAL INTRODUCTION

### 1. LABORATORY EXPERIMENTS IN THE COMPETENCE BASED CURRICULUM

A competence-based curriculum (CBC) focuses on what learners can do and apply in different situations by developing skills, attitudes, and values in addition to knowledge and understanding. This learning process is learner-focused, where a learner is engaged in active and participatory learning activities, and learners finally build new knowledge from prior knowledge. Since 2015, the Rwanda education system has changed from KBC to CBC for preparing students that meet the national and international job market requirements and job creation. Therefore, implementing the CBC education system necessitates qualitative laboratory practical works for mathematics and science as more highlighted aspects.

In addressing this necessity, laboratory experiments play a major role. A student is motivated to learn chemistry by getting involved in handling various concrete manipulations in various experiments.

For learning chemistry concepts through the above-mentioned approach, Chemistry kits composed of chemicals and apparatuses have been distributed into schools. The kits include various items along with a manual for performing experiments. The kit broadly covers the experiments that are proposed in the syllabus. The kit has the following advantages:

- Availability of necessary and common materials at one place,
- Multipurpose use of items,
- Economy of time in doing the activities,
- Portability from one place to another,
- Provision for teacher's innovation,
- Low-cost material and use of indigenous resources.

Apart from the kit, the user guide for laboratory and practical activities to be used by teachers was developed. This laboratory experiment user guide is designed to help mathematics and science teachers to perform high-quality lab experiments for mathematics and science. This user guide structure induces learner's interest, achievement, and motivation through the qualitative mathematics and science lab experiments offered by their teachers and will finally lead to the targeted goals of the CBC education system, particularly in the field of mathematics and science.

In CBC, learners hand-on the materials and reveal the theory behind the experiment done. Here, experiments are done inductively, where experiments serve as an insight towards revealing the theory. Thus, the experiment starts, and theory is produced from the results of the experiment.

## 2. TYPE OF LABORATORY EXPERIMENTS

The goal of the practical work defines the type of practical work and how it is organized. Therefore, before doing practical work, it is important to have a clear idea of the objective. The three types of practical work that correspond with its three main goals are:

- a) **Equipment-based practical work:** the goal is for students to learn to handle scientific equipment like using a microscope, doing titrations, making an electric circuit, etc.
- b) **Concept-based practical work:** learning new concepts.
- c) **Inquiry-based practical work:** learning process skills. Examples of process skills are defining the problem and good research question(s), installing an experimental setup, observing, measuring, processing data in tables and graphs, identifying conclusions, defining limitations of the experiment etc.

### Note:

- To learn the new concept by practical work, the lesson should start with the practical work, and the theory can be explained by the teacher afterward (explore – explain).
- Starting by teaching the theory and then doing the practical work to prove what they have learned is demotivating and offers little added value for student learning.
- Try to avoid complex arrangements or procedures. Use simple equipment or handling skills to make it not too complicated and keep the focus on learning the new concept.
- If this is not possible and is necessary to use new equipment or handling skills, then first exercise these skills before starting the concept-based practical work experiments.
- The experiments should be useful for all learners and not only for aspiring scientists. Try to link the practical work as much as possible with their daily life and preconceptions.

### 3. ORGANIZATION, ANALYSIS, AND INTERPRETATION OF DATA

Once collected, data must be ordered in a form that can reveal patterns and relationships and allows results to be communicated to others. We list goals about analysing and interpreting data. By the end of secondary education, students should be able to:

- Analyses data systematically, either look for relevant patterns or test whether data are consistent with the initial hypothesis.
- Recognize when data conflict with expectations and consider what revisions in the initial model are needed.
- Use spreadsheets, databases, tables, charts, graphs, statistics, mathematics, and ICT to compare, analyze, summarize, and display data and explore relationships between variables, especially those representing input and output.
- Evaluate the strength of a conclusion that can be inferred from any data set, using appropriate grade-level mathematical and statistical techniques.
- Recognize patterns in data that suggest relationships worth investigating further. Distinguish between causal and correlational relationships.
- Collect data from physical models and analyze the performance of a design under a range of conditions.

### 4. ORGANIZING LAB EXPERIMENTS

- Methods to organize practical work. There are 3 methods of organizing practical work

#### **a. Each group does the same experiments at the same time**

All learners can follow the logical sequence of the experiments, but this implies that a lot of material is needed. The best group size is 3, as all learners will be involved. With bigger groups, you can ask to do the experiment twice, where learners change roles.

#### **b. Experiments are divided among groups with group rotation**

Each group does the assigned experiment and moves to the next experiment upon a signal by the teacher. At the end of the lesson, each group has done every experiment. This method saves materials but is not perfect when experiments are not ordered in a logical way. In some cases, the conclusion of an experiment provides the research question for the next experiment. In that case, this method is not very suitable.

The organization is also more complex. Before starting the lesson, the materials for each experiment should be placed in the different places where the groups will work. Also, the required time for each experiment should be about the same. Use a timer to show learners the time left for each experiment. Provide an extra exercise for fast groups.

### **c. All experiments are divided among groups without group rotation**

Each group does only one or two experiments. The other experiments are done by other groups. Afterward, the results are brought together and discussed with the whole class. This saves time and materials, but it means that each learner does only one experiment and 'listens' to the other experiments' description. The method is suitable for experiments that are optional or like each other. It is not a good method for experiments that all learners need to master.

#### **\* Preparation of a practical work**

- Have a look at the available material at school and make a list of what you can use, and what you need to improvise.
- Determine the required quantities by determining the method (see above).
- Collect all materials for the experiments in one place. If the learners' group is small, they can come to get the materials on that spot, but with more than 15 learners, this will create disorder. In that case, prepare for each group a set of materials and place it on their desk.
- Test all experiments and measure the required time for each experiment.
- Prepare a nice but educational extra task for learners who are ready before the end of the lesson.
- Write on the blackboard how groups of learners are formed.

#### **\* Preparation of a lesson for practical work**

In the lesson plan of a lesson with practical work, there should be the following phases:

1. The introduction of the practical work or the 'excite' phase consists of formulation of a rationale, discrepant event, or a small conversation to motivate learners and make connections with daily life and learners' prior knowledge.
2. The discussion of safety rules for the practical work:
  - Only work at the assigned place; do not walk around in the class if this is not asked.
  - Long hairs should be tied together, and safety eyeglasses should be worn for chemical experiments.

- Only the material needed for the experiment should be on the table.
- The practical work instructions: how groups are formed, where they get the materials, special treatment of materials (if relevant), what they must write down...
- When the practical work materials aren't yet at the correct location, then distribute them now. Once learners have the materials, it is more difficult to get their attention.

**\* How to conduct a practical work**

- Learners do the experiments, while the teacher coaches by asking questions (Explore phase).
- The practical work should preferably be processed immediately with an explain phase. If not, this should happen in the next lesson.

**\* How to conclude the lesson of a practical work:**

- Learners refer to instructions and conduct the experiment,
- Learners record and interpret recorded data,
- Cleaning the workspace after the practical work (by the learners as much as possible).

**\* Role and responsibilities of teacher and learners in lab experiment:**

Before conducting an experiment, the teacher will do the following:

- Decide how to incorporate experiments into class content best,
- Prepare in advance materials needed in the experiment,
- Prepare protocol for the experiment,
- Perform in advance the experiment to ensure that everything works as expected,
- Designate an appropriate amount of time for the experiment - some experiments might be adapted to take more than one class period, while others may be adapted to take only a few minutes.
- Match the experiment to the class level, course atmosphere, and your students' personalities and learning styles.
- Verify lab equipment before lab practices.
- Provide the working sheet and give instructions to learners during the lab session.

During practical work, the teacher's role is to coach instead of helping with advice or questions. It is better to answer a learner's question with another question than to immediately give the answer or advice. The additional question should help learners to find the answer themselves.

- Prepare some pre-lab questions for each practical work, no matter what the type is.
- Try and start the practical work: start with a discrepant event or questions that help define the problem or questions that link the practical work with students' daily life or their initial conceptions about the topic.
- Use coaching questions during the practical work: 'Why do you do this?', 'What is a control tube?', 'What is the purpose of the experiment?', 'How do you call this product?', 'What are your results?' etc.
- Use some questions to end the practical work: 'What was the meaning of the experiment?', 'What did we learn?', 'What do we know now that we didn't know at the start?', 'What surprised you?' etc.
- Announce the end of the practical work 10 minutes before giving learners enough time to finish their work and clean their space.

\* **The Role of a lab technician during a laboratory-based lesson**

In schools having laboratory technicians, they assist the science teachers in the following tasks:

- Maintaining, calibrating, cleaning, and testing the sterility of the equipment,
- Collecting, preparing and/or testing samples,
- Demonstrating procedures.
- The learners' responsibilities in the lab work:
  - During the lab experiment, learners have different activities to do; general learner's activities are:
    - Experiment and obtain data themselves,
    - Record data using the equipment provided by the teacher,
    - Analyse the data often this involves graphing it to produce the related graph,
    - Interpret the obtained results and deduct the theory behind the concept under the experimentation,
    - Discuss the error in the experiment and suggest improvements,
    - Cleaning and arranging material after a lab experiment.

## 5. SAFETY RULES AND PRECAUTIONS DURING LAB EXPERIMENTS

Regardless of the type of lab you are in, there are general rules enforced as safety precautions. Each lab member must learn and adhere to the rules and guidelines set, to minimize the risks of harm that may happen to them within the working environment. These encompass dress' code, use of personal protection equipment, and general behaviour in the lab. It is important to know that some laboratories contain certain inherent dangers and hazards. Therefore, when working in a laboratory, you must learn how to work safely with these hazards to prevent injury to yourself and other lab mates around you. You must make a constant effort to think about the potential hazards associated with what you are doing and think about how to work safely to prevent or minimize these hazards as much as possible. Before doing any scientific experiment, you should make sure that you know where the fire extinguishers are in your laboratory, and there should also be a bucket of sand to extinguish fires. You must ensure that you are appropriately dressed whenever you are near chemicals or performing experiments. Please make sure you are familiar with the safety precautions, hazard warnings, and procedures of the experiment you perform on a given day before you start any work. Experiments should not be performed without an instructor in attendance and must not be left unattended while in progress.

### A. Hygiene plan

A laboratory is a shared workspace, and everyone has the responsibility to ensure that it is organized, clean, well-maintained, and free of contamination that might interfere with the lab members' work or safety.

For waste disposal, all chemicals and used materials must be discarded in designated containers. Keep the container closed when not in use. When in doubt, check with your instructor.

### B. Hazard warning symbols

To maintain a safe workplace and avoid accidents, lab safety symbols and signs need to be posted throughout the workplace. Chemicals pose health and safety hazards to personnel due to innate chemical, physical, and toxicological properties. Chemicals can be grouped into several different hazard classes. The hazard class will determine how similar materials should be stored and handled and what special equipment and procedures are needed to use them safely.

Each of these hazards has a different set of safety precautions associated with them.



The following table shows hazard symbols found in laboratories and the corresponding explanations

Symbol	Meaning
 Highly flammable	Easily catches fire and burns
 Toxic	It can lead to death
 Irritant	It irritates the skin when in contact
 Radioactive	Dangerous to human health and can cause cancer



Corrosive

Can easily burn you when in contact with your skin. It can also damage wood and metals



Explosive

It can easily explode and releases small particles which can injure you



Harmful

Poisonous when inhaled and ingested and can lead to death

### C. General Laboratory Safety Rules

You are ultimately responsible for your own safety and that of your fellow students, workers and visitors. A standard list of basic laboratory safety rules are given below, and must be followed in every laboratory that uses hazardous materials or processes. These basic rules provide behavior, hygiene, and safety information to avoid accidents in the laboratory. Laboratory specific safety rules may be required for specific processes, equipment, and materials, which should be addressed by laboratory specific standard operating procedure.

1. The following Personal Protective Equipment must be worn at all times in the laboratory:
  - Lab coat.
  - Eye protection: Chemical goggles. If you do get a chemical in your eye, rinse your eye immediately using large quantities of water or an eye wash bottle if available.

- Closed shoes with socks must be worn at ALL times – open-toed shoes, backless shoes and sandals are not permitted.
  - Always wear gloves when working with unknown substances.
  - Always wear the appropriate breathing masks when working with toxic or irritating vapours.
2. DO NOT work alone in a laboratory. Know the location and proper use of fire extinguishers, fire blankets and first aid kits.
  3. Perform work with hazardous chemicals in a properly working fume hood to reduce potential exposures.
  4. Always work in a well-ventilated area.
  5. Working areas should be kept clean and tidy at all times.
  6. Eating, smoking, and drinking are not allowed in a chemistry laboratory.
  7. Labels and equipment instructions must be read carefully before use.
  8. Long hair and loose clothing must be pulled back and secured from potential capture.
  9. Avoid wearing jewellery in the lab as this can pose multiple safety hazards.
  10. All containers must have appropriate labels. Unlabelled chemicals should never be used.
  11. Do not taste or intentionally sniff chemicals.
  12. Unused chemicals should not be returned to their original container unless directed to do so by the lab instructor.
  13. DO NOT perform unauthorized experiments.
  14. Never leave containers of chemicals open.
  15. Avoid distracting or startling persons working in the laboratory.
  16. Securely replace lids, caps, and stoppers after removing reagents from containers.
  17. All flammable reagents must be removed before lighting a burner.
  18. Never pour water into concentrated acid.
  19. Mouth suction is never used to fill a pipette.
  20. Always wipe spatulas clean before and after inserting into reagent bottles.
  21. Report any accident and/or injury, however minor, to your instructor immediately.

22. Clean up any chemical spilled on the floor or any other working place immediately.
23. Before leaving the laboratory, make sure your work area is clean and dry and also ensure that all gas and water are completely turned off.
24. Wash exposed areas of the skin prior before leaving the laboratory.
25. Return materials used in the laboratory storage facility.
26. Never hesitate to ask questions especially if there is any question concerning proper operating procedure. Be sure that you understand every instruction before proceeding.
27. Never store food or beverages or apply cosmetics in areas where chemicals are used

## **6. GUIDANCE ON THE MANAGEMENT OF LAB MATERIALS: STORAGE MANAGEMENT, REPAIRING AND DISPOSAL OF LAB EQUIPMENT**

### **\* Keeping and cleaning up**

Working spaces must always be kept neat and cleaned up before leaving. Equipment must be returned to its proper place. Keep backpacks or bags off the floor as they represent a tripping hazard. Open flames of any kind are prohibited in the laboratory unless specific permission is granted to use them during an experiment.

### **\* Management of lab materials**

A science laboratory is a place where basic experimental skills are learned only by performing a set of prescribed experiments. Safety procedures usually involve chemical hygiene plans and waste disposal procedures. When providing chemicals, you must read the label carefully before starting the experiment. To avoid contamination and possibly violent reactions, do never return unwanted chemicals to their container. In the laboratory, chemicals should be stored in their original containers, and cabinets should be suitably ventilated. It is important to notify students that chemicals cannot be stored in containers on the floor. Sharp and pointed tools should be stored properly.

Students should always behave maturely and responsibly in the laboratory or wherever chemicals are stored or handled.

### \* **Hot equipment and glassware handling**

Hazard symbols should be used as a guide for the handling of chemical reagents. Chemicals should be labelled as explosives, flammable, oxidizers, toxic and infectious substances, radioactive materials, corrosives etc. All glassware should be inspected before use, and any broken, cracked, or chipped glassware should be disposed of in an appropriate container. All hot equipment should be allowed to cool before storing it.

All glassware must be handled carefully and stored in its appropriate place after use. All chemical glass containers should be transported in rubber or polyethylene bottle carriers when leaving one lab area to enter another. When working in a lab, do never leave a hot plate unattended while it is turned on. It is recommended to handle hot equipment with safety gloves and other appropriate aids but never with bare hands. You must ensure that hands, hair, and clothing are kept away from the flame or heating area and turn heating devices off when they are not in use in the laboratories.

### \* **Waste disposal considerations**

Waste disposal is a normal part of any science laboratory. As teachers or students perform demonstrations or laboratory experiments, chemical waste is generated.

These wastes should be collected in appropriate containers and disposed of according to local, state, and federal regulations. All schools should have a person with the responsibility of being familiar with this waste disposal. In order to minimize the amount of waste generated and handle it safely, there are several steps to consider. Sinks with water taps for washing purposes and liquid waste disposal are usually provided on the working table. It is essential to clean the sink regularly. Notice that you should never put broken glass or ceramics in a regular waste container. Use a dustpan, a brush, and heavy gloves to carefully pick-up broken pieces, and dispose of them in a container specifically provided for this purpose. Hazardous chemical waste, including solvents, acids, and reagents, should never be disposed of down sewer drains. All chemical waste must be identified properly before it can be disposed of. Bottles containing chemical waste must be labelled appropriately. Labelling should include the words "hazardous waste."

Chemical waste should be disposed of in glass or polyethylene bottles. Plastic coated glass bottles are best for this purpose. Aluminium cans that are easily corroded should not be used for waste disposal and storage.

### \* Equipment Maintenance

Maintenance consists of preventative care and corrective repair. Both approaches should be used to keep equipment in working order. Records of all maintenance, service, repairs, and histories of any damage, malfunction, or equipment modification must be maintained in the equipment logs. The record must describe hardware and software changes and/or updates and show the dates when these occurred. Each laboratory must maintain a chemical inventory that should be updated at least once a year.

## 7. STUDENT EXPERIMENT WORKSHEET

There should be a sheet to guide students about how they will conduct the experiment, materials to be used, procedures to be followed and the way of recording data. The following is a structure of the student experiment worksheet. It can be prepared by the teacher or be available from the other level.

1. Date
2. Name of student/group
3. The title of experiment
4. Type of experiment (concept, equipment and inquiry based)
5. Objective(s) of the experiment
6. Key question(s)
7. Materials (apparatuses and chemicals, resources, etc...)
8. Procedures & Steps of experiment
9. Data recording and presentation

Test	Results/ observation	Comments
1		
2		
3 etc		

10. Reflective questions and answers  
Question 1  
Question 2  
Question 3
11. Answer for the key question.

## 8. REPORT TEMPLATE FOR LEARNER

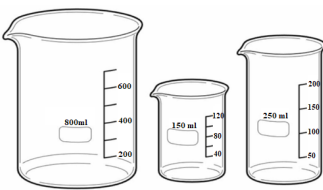



After conducting a laboratory experiment, students should write a report about their findings and the conclusion they reached. The report to be made depends on the level of students. The following is a structure of the report to be made by a group of secondary school learners (S4-S6).

1. Introduction (details related to the experiment: Students identification, date, year, topic area, unit title and lesson).
2. The title of the experiment.
3. Type of experiment (concept, equipment and inquiry based)
4. Objective(s) of the experiment.
5. Key question(s)
6. Materials (apparatuses and chemicals, resources, etc...)
7. Procedures & Steps of experiment
8. Data recording
9. Data analysis and presentation (Plots, tables, pictures, graphs)
10. Interpretation/discussion of the results, student alternative ideas for observation.
11. Theory or Main ideas concept, formulas, and application.
12. Conclusion (answer reflective questions and the key question).

As a conclusion, there are safety rules and precautions to consider before, during and at the end of a lab experiment. We hope teachers are inspired to conduct lab experiments in a conducive Competence Based Curriculum way.

## PART 2: LIST OF MAIN KIT ITEMS AND LAB MATERIALS NEEDED IN SCHOOLS

### A. List of Apparatus

#	Item and description	Picture	Description of uses
1	Beaker		Used to hold and heat liquids. Multipurpose and essential in the laboratory.
2	Brushes		Used to easily clean the inside of a test tubes and other glassware.
3	Buchner funnel		Used with vacuum flask for performing vacuum filtration.
4	Bunsen burner		<p>First, make sure your workspace is free of potential fire hazards. Once you are sure space is safe, connect the gas line and ignite the burner. Adjust the metal collar and needle gas valve at the burner's base to control the flame's size and temperature. When you're finished, close the air and gas ports, shut off the gas main line, and put the burner away once it's cool.</p> <p>Bunsen burner is used for heating and exposing items to flame.</p>



5

## Burette








## Operating the Burette

Proper burette technique is an important laboratory skill that may take some practice to develop. Although it may seem initially awkward, a right handed person should operate the burette with the left hand, and a left handed person should operate the burette with the right. This leaves your more coordinated hand to swirl the reaction flask if needed.

Before delivering any solution, record the initial burette reading in your notebook.

Open the stopcock by twisting it 90 degrees into the vertical position and allow the solution to drain. As you near the desired volume, slow the flow by turning the stopcock back toward the closed position. You should be able to control the burette to deliver one drop at a time. When the desired volume has been delivered, close the stopcock.

Wait a couple of seconds, then record the final burette reading.

6	Burette clamp		Used to hold burettes on a ring stand.
7	Clay triangle		Used to hold crucibles when they are being heated. They usually sit on a ring stand.
8	Crucible with lid		Used to heat small quantities to very high temperatures.
9	Crucible tong		Used to hold crucibles and evaporating dishes when they are hot.
10	Disposable pipette		Used for moving small amounts of liquid from place to place. They are usually made of plastic and are disposable.

11 Electronic balance



Used for weighing substances or objects, usually in grams.






Place the electronic balance on a flat, stable surface indoors.




Press the “ON” button and wait for the balance to show zeroes on the digital screen. Place the empty container you will use for the substance to be measured on the balance platform.

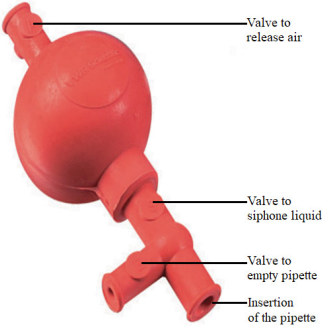


Press the “Tare” or “Zero” button to cancel automatically the weight of the container. The digital display will show zero again.

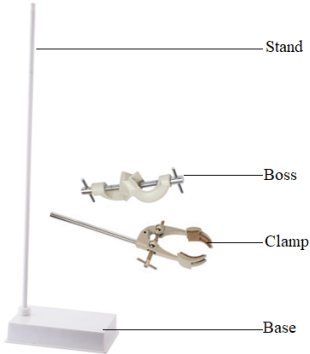



Carefully add the substance to the container. Ideally this is done with the container still on the platform, but it may be removed if necessary. Avoid placing the container on surfaces that may have substances which will add mass to the container such as powders or grease.





Place the container with the substance back on the balance platform if necessary and record the mass as indicated by the digital display.

12	Erlenmeyer flasks/Conical flask		<p>Used to heat, mix, and store liquids. The advantage to the Erlenmeyer Flask is that the bottom is wider than the top so it will heat quicker because of the greater surface area exposed to the heat.</p>
13	Evaporating dish		<p>Used to recover dissolved solids by evaporation.</p>
14	Forceps		<p>Used for picking up and moving small objects.</p>
15	Glass funnel & Polypropylene funnel		<p>Used to pour liquids into any container so they will not be lost or spilled. They are also used with folded filter paper for filtration.</p>
16	Glass stir rod		<p>Used to stir liquids. They are usually made of glass.</p>

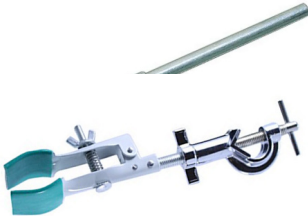



17	Graduated cylinder/ measuring cylinder		Used to measure the volumes of liquids.
18	Micropipette		<p>Used for accurately measuring and delivering very small volumes of liquid-usually 1 ml or less.</p> <p>Steps to follow when using a micropipette</p> <p>Select the volume.</p> <p>Set the tip.</p> <p>Press and hold the plunger at the first stop.</p> <p>Place the tip in the liquid.</p> <p>Slowly release the plunger.</p> <p>Pause for a second and then move the tip.</p> <p>Insert the tip into the delivery vessel.</p> <p>Press the plunger to the second stop.</p>
19	Mortar and pestle		Used to crush solids into powders for experiments, usually to better dissolve the solids.


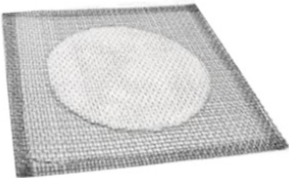


20	Pipette filler		<p>How does a pipette filler work?</p> <p>Siphon liquid into the pipette to the desired level by squeezing valve “S” on the bottom of the pipette filler. This uses the vacuum created in the bulb to draw liquid into the pipette. Be careful not to draw liquid into the pipette filler. ... This allows you to release liquid at the desired rate and to the desired level.</p>
21	Pipette with pump		<p>Used for accurately measuring and delivering small volumes of liquid- usually 0.1-10 mL.</p>
22	Ring clamp		<p>Attached to a retort stand and with wire gauze used to hold beakers or flasks while they are heated by a gas burner.</p>




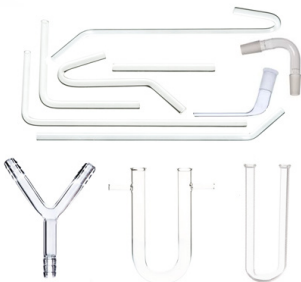
23	Retort stand and accessories		<p>Used to hold items being heated. Clamps or rings can be used so that items may be placed above the lab table for heating by Bunsen burners or other items.</p> <p>Used also to hold burette</p>
24	Rubber stopper		<p>Stoppers come in many different sizes. The sizes are from 0 to 8. Stoppers can have holes for thermometers and for other probes that may be used.</p>
25	Separator funnel		<p>For separating layers of immiscible liquids or for dropping liquids.</p>
26	Spatula		<p>Used for moving small amounts of solid from place to place.</p>

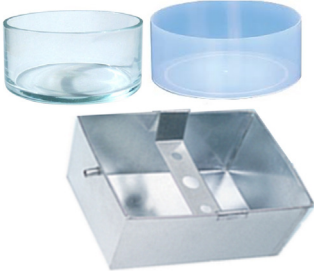





27	Test tube		Used for storing, mixing, and heating small amounts of chemicals.
28	Test tube holder		Used to hold test tubes while heating.
29	Test tube rack		Used to hold test tubes while reactions happen in them or while they are not needed.
30	Thermometer		Used to take temperature of solids, liquids, and gases.







31	Utility clamp		Used to attach test tubes and other glassware to retort stand.
32	Vacuum filter flask		Used with vacuum line and Buchner funnel for vacuum filtration.
33	Volumetric flask		Used to prepare solutions with accurate concentration.
34	Wash bottle		Used to wash; rinse containers

35	Watch glass		Used to hold solids when being weighed or transported. They should never be heated. Can also be used to cover beakers or other containers.
36	Wire gauze		Used with a ring clamp to support glassware over a Bunsen burner.
37	Borosilicate glass tube		Used to connect to other items of glassware or equipment to deliver chemicals, solvents, liquids, gases and other products.
38	Deflagrating spoon or gas jar spoon		Generally used for the burning of materials inside gas jars or similar.

39	Thistle funnel		<p>Thistle tubes are typically used to add liquid to an existing system or apparatus. Thistle funnels are used to add small volumes of liquids to an exact position.</p>
40	Cardboard cover		
41	Rubber tube		<p>Rubber tubing is often connected to a condenser, which is a laboratory tool used in the process of distillation. The rubber tubing helps cool water to flow in and out of the condenser and helps the heated water vapor in the condenser return to its liquid state.</p>
42	Borosilicate delivery tube		<p>The delivery tube is particularly useful for bubbling a gas from a gas cylinder or stoppered vessel through a liquid.</p>

43	Trough		The trough is used for collecting gases, such as hydrogen, oxygen and nitrogen. Troughs require a liquid such as water.
SENIOR TWO			
44	Beehive shelf		A beehive shelf is usually used to support a receiving jar or tube while a gas is being collected over water with a pneumatic trough.
45	Graphite rods		Graphite rods are used as electrodes
46	Metallic rod		Metallic rods are used to heat to test the heat conduction of metals
47	Aluminium foil		Can be used for temporary covering of instruments, shielding in vacuum equipment, packaging, wrapping, weighing boats, etc.
48	Sulphur rod		Sulphur rods are used to heat to test the heat conduction of non-metals

49	Charcoal rod		Charcoal rods are used as electrodes
50	Syringe		They are often used for measuring and transferring solvents and reagents where a high precision is not required.
51	Electrolyser		Electrolyser is used in the electrolysis process
52	Gas jar and cover		A container used for collecting gas from experiments.

## B. List of chemicals

SN	Chemicals
1	Alizarin yellow
2	Aluminium foil or paper towel
3	Ammonia solution
4	Ammonium chloride
5	Benzoic acid
6	Beta-naphthol, $C_{10}H_8O$
7	Blue and red litmus papers
8	Bromine water
9	Bromothymol blue
10	Butylamine, $C_4H_{11}N$
11	Calcium hydroxide
12	Charcoal powder
13	Chromium (III) nitrate, $Cr(NO_3)_3$
14	Cobalt (II) nitrate, $Co(NO_3)_2$
15	Copper (II) sulphate ( $CuSO_4$ )
16	Copper rod
17	Copper turning or powder
18	Distilled water
19	Ethanoic acid
20	Ethanol
21	Ethoxyethane
22	Formaldehyde solution
23	Fresh fat milk
24	Glacial acetic acid
25	Hydrochloric acid
26	Hydrogen peroxide, $H_2O_2$
27	Ice
28	Iodine, $I_2$

29	Iron (II) nitrate, $\text{Fe}(\text{NO}_3)_2$
30	Iron (III) chloride
31	Iron nails
32	Lead nitrate solution
33	Magnesium ribbon
34	Manganese (II) chloride, $\text{MnCl}_2$
35	Marble chips, $\text{CaCO}_3$
36	Methyl orange
37	Nickel sulphate, $\text{NiSO}_4$
38	Nitric acid, $\text{HNO}_3$
39	N-methylbutylamine, $\text{C}_5\text{H}_{13}\text{N}$
40	Phenol
41	Phenolphthalein indicator
42	Phenylamine, $\text{C}_6\text{H}_5\text{NH}_2$
43	Phthalic anhydride $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$
44	Potassium cyanide, KCN
45	Potassium hexacyanoferrate (III), $\text{K}_3[\text{Fe}(\text{CN})_6]$
46	Potassium iodide, KI
47	Potassium nitrate
48	Potassium thiocyanate, KSCN
49	Propanoic acid, $\text{CH}_3\text{CH}_2\text{COOH}$ (1M)
50	Propanol, $\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$
51	Propanone
52	Silver nitrate, $\text{AgNO}_3$
53	Sodium acetate
54	Sodium carbonate, $\text{Na}_2\text{CO}_3$ (1M)
55	Sodium chloride
56	Sodium hydrogen carbonate, $\text{NaHCO}_3$
57	Sodium hydroxide
58	Sodium metal

59	Sodium nitrite, $\text{NaNO}_2$
60	Sodium thiosulphate
61	Solid bismuthate (V), $\text{NaBiO}_3$
62	Starch indicator
63	Sulphuric acid
64	Thymol blue
65	Triethylamine, $\text{C}_6\text{H}_{15}\text{N}$
66	Universal indicator
67	Vinegar
68	Zinc granules
69	Zinc rod
70	Zinc sulphate, $\text{ZnSO}_4$
71	3-methylphenylamine



## **PART 3: DETAILED EXPERIMENTS FOR SENIOR SIX**

**EXPERIMENT 1.1:** Ligand exchange reaction**Rationale**

Ligand exchange (or ligand substitution) is when one ligand in a complex is replaced by another. Ligand exchange forms a new complex that is more stable than the original one. Ligand exchange has many applications such as metallurgy for the extraction of metals such as nickel, cobalt, and copper from their ores. It is also used in medicine to treat some diseases, chelating agents, chemical analysis, etc.

**Objective**

Learners will be able to explain the ion exchange phenomenon.

**Required materials****Apparatus**

- Beakers
- volumetric flasks
- Test tubes
- Test tubes rack
- Washing bottles
- Test tube holders
- Droppers
- Spatula
- Gloves

**Chemicals**

- Copper (II) sulphate solution (0.5M),  $\text{CuSO}_4$
- Ammonia solution (0.5M),  $\text{NH}_3$
- Distilled water

## Experimental set-up

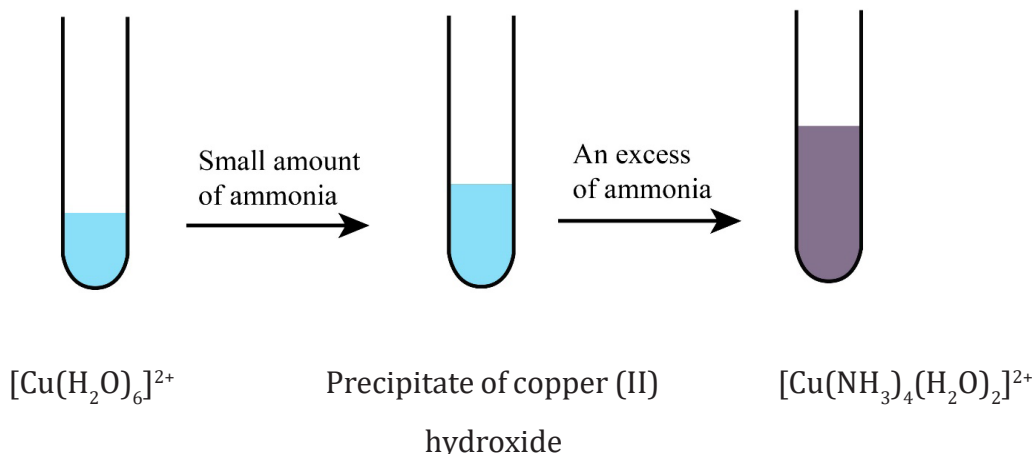


Figure 1.1: Reaction of copper sulphate with ammonia solution



## Procedure

1. Prepare a copper (II) sulphate solution (0.5M) and an ammonia solution (0.5M) using distilled water. Why is distilled water used instead of tap water?

**Answer:** Distilled water is used to avoid the interference of others ions that may be present in tap water

2. Put about 10 drops of copper (II) sulphate solution into a test tube; What is the colour of the copper (II) sulphate solution?

**Answer:** Pale blue solution

3. Add ammonia solution drop-by-drop to the copper sulphate solution (pale solution )
4. Shake the test tube gently after adding each drop. What do you observe when adding a few drops of ammonia solution?

**Answer:** Pale blue precipitate appeared

5. Add more drops of ammonia solution. What do you observe?

**Answer:** Dark blue solution appeared.

**Note:** Wear safety gloves during the experiment.

## Interpretation of results and conclusion

### Guiding questions

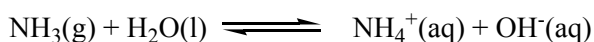
Why does precipitate disappear when an excess amount of ammonia is added?

Write a balanced chemical equation of the reaction between ammonia and copper sulphate.

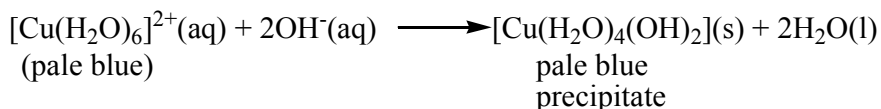
### Answer to guiding questions

When solid copper sulphate is dissolved in water, a pale blue solution is obtained. Aqueous solutions contain the hexaaqua copper (II),  $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$ .

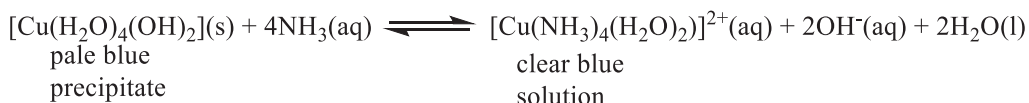
Ammonia is a weak base that forms a few ammonium and hydroxide ions in solution:



When copper sulphate reacts with ammonia, the hexaaqua-copper (II) ions react with hydroxide ions to form a precipitate according to the following equation:



When copper (II) hydroxide precipitate reacts with ammonia molecules there is formation of tetra-amine-di-aqua-copper(II) ions. This reaction involves ligand exchange where hydroxide ions are replaced with ammonia molecules:



Therefore, the overall chemical equation of the reaction is as follow:



### Evaluation

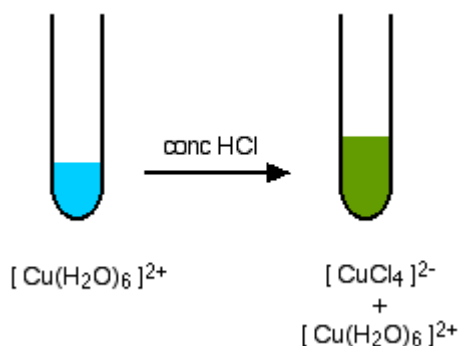
1. What does a ligand exchange reaction mean?

**Answer:** A ligand exchange reaction is a reaction in which one ligand in a complex is replaced by another ligand.

2. Perform an experiment illustrating the reaction of copper (II) ions with hydrochloric acid.

Record your observations and make a proper interpretation of your findings.

**Answer:**



When copper (II) sulphate solution reacts with concentrated hydrochloric acid, it results in the ligand substitution reaction. In the concentrated hydrochloric acid, there is a high concentration of negatively charged chloride ions that are better ligands than water. These negatively charged ions are electronically attached to the copper (II) ion ( $\text{Cu}^{2+}$ ).

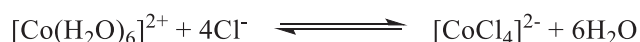
When the concentrated hydrochloric acid is added dropwise to the pale blue copper sulphate solution, the solution turns yellow green as the chloride ions ligands ( $\text{Cl}^-$ ) replaces water in the complex ion. The six water molecules in the blue complex are replaced by the 4 chloride ions to form the yellow green  $[\text{CuCl}_4]^{2-}$  complex ion.

3. Provide other examples of ligand exchange reactions and write their corresponding chemical equations.

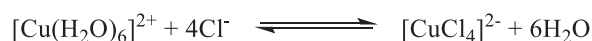
**Answer:** *Examples of ligand exchange reactions:*

#### a) Replacing water with chloride ions

- Replacing the water in the hexaaquacobalt(II) ion



- Replacing the water in the hexaaquacopper(II) ion

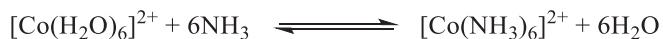


#### b) Replacing water molecules by ammonia

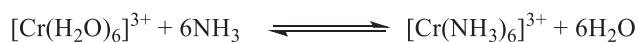
Replacing water in the hexaaquacopper(II) ion



- Replacing the water in the hexaaquacobalt(II) ion



- Replacing water in the hexaaquachromium(III) ion



## EXPERIMENT 1.2:

## Preliminary and confirmatory tests for transition metal ions ( $\text{Ni}^{2+}$ , $\text{Fe}^{2+}$ , $\text{Fe}^{3+}$ , $\text{Mn}^{2+}$ , $\text{Cr}^{3+}$ , $\text{Zn}^{2+}$ , $\text{Co}^{2+}$ , $\text{Cu}^{2+}$ )

### Rationale

The identification of metal ions helps to know the composition of a given inorganic salt or a mixture of salts. This experiment helps to choose the needed substance according to the problem to be solved for instance the choice of an inorganic fertiliser, the safety precautions to be taken when handling a given salt.

### Objective

Learners will be able to identify the presence of transition metal ions ( $\text{Ni}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cu}^{2+}$ ) in a solution.



### Required materials

#### Apparatus

- Beakers
- Test tubes
- Test tubes rack
- Test tubes holders
- Droppers
- Spatula
- Gloves
- Bunsen burner

#### Chemicals

- Nickel sulphate,  $\text{NiSO}_4$
- Iron (II) nitrate,  $\text{Fe}(\text{NO}_3)_2$
- Iron (III) nitrate,  $\text{Fe}(\text{NO}_3)_3$
- Manganese (II) chloride,  $\text{MnCl}_2$
- Chromium (III) nitrate,  $\text{Cr}(\text{NO}_3)_3$
- Cobalt (II) nitrate,  $\text{Co}(\text{NO}_3)_2$
- Copper (II) sulphate,  $\text{CuSO}_4$
- Zinc sulphate,  $\text{ZnSO}_4$

## Apparatus

## Chemicals

- Sodium hydroxide, NaOH (1M)
- Ammonia (NH<sub>3</sub>) solution (1M)
- Sodium carbonate, Na<sub>2</sub>CO<sub>3</sub> (1M)
- Distilled water
- Potassium cyanide, KCN
- Potassium hexacyanoferrate (III), K<sub>3</sub>[Fe(CN)<sub>6</sub>]
- Silver nitrate, AgNO<sub>3</sub>
- Potassium thiocyanate, KSCN
- Solid sodium bismuthate (V), NaBiO<sub>3</sub>
- Nitric acid, HNO<sub>3</sub>
- Sulphuric acid, H<sub>2</sub>SO<sub>4</sub>
- Hydrogen peroxide, H<sub>2</sub>O<sub>2</sub>

## Experimental set-up

sodium hydroxide solution in teat pipette

colourless !

NaOH(aq)

NaOH(aq)

NaOH(aq)

NaOH(aq)

NaOH(aq)

dark green Fe(OH)<sub>2</sub>(s) iron(II) hydroxide precipitate

Fe<sup>2+</sup>(aq) iron(II) ion pale green

1

dark brown Fe(OH)<sub>3</sub>(s) iron(III) hydroxide precipitate

Fe<sup>3+</sup>(aq) iron(III) ion orange

2

deep blue Cu(OH)<sub>2</sub>(s) copper(II) hydroxide precipitate

Cu<sup>2+</sup>(aq) copper(II) ion blue

3

white Zn(OH)<sub>2</sub>(s) zinc hydroxide precipitate

Zn<sup>2+</sup>(aq) zinc ion colourless

4a

adding excess NaOH(aq)

4b

Figure 1.2: Reaction of some transition cations with sodium hydroxide



## Procedure

### Preliminary tests

#### *Analysis of the colour of the solid compound or its solution*

1. Put a small amount (a half endful spatula) of each salt given in a test tube.
2. Record the appearance (colour and texture) of the solid salt.

#### *Testing using sodium hydroxide and ammonia solutions*

3. Add 15 mL of distilled water to the solids in the test tube to form a solution and note the colour of the solution.
4. Divide the solution of copper sulphate into 3 portions in different test tubes.
5. To the first test tube, add a few drops (3-4) of sodium hydroxide solution and shake. What do you observe?

*Answer: A blue precipitate is formed*

6. Add more sodium hydroxide solution to the same test tube, and shake.

What does happen when you add more sodium hydroxide solution?

*Answer: The blue precipitate is not dissolved.*

7. To the second test tube, add 3-4 drops of a 1 M ammonia solution and shake.

What do you observe?

*Answer: A blue precipitate is formed.*

8. Add more ammonia solution to the same test tube, and shake.

What does happen when you add more ammonia solutions?

*Answer: The blue precipitate is dissolved and forms deep blue solution*

9. Repeat step 4-8 by using each of the following compounds:  $\text{Fe}(\text{NO}_3)_2$ ,  $\text{Fe}(\text{NO}_3)_3$ ,  $\text{MnCl}_2$ ,  $\text{Cr}(\text{NO}_3)_3$ ,  $\text{ZnSO}_4$ ,  $\text{Co}(\text{NO}_3)_2$ ,  $\text{NiSO}_4$ , instead of copper sulphate.

Record your observations when each of the above ions is treated with a small amount of NaOH and/or  $\text{NH}_3$  solution and then with excess of each of these reagents.



## Data recording

	Appearance of the solid	Colour of the solution	Addition of NaOH solution	Addition of excess NaOH solution	Addition of ammonia solution	Addition of excess ammonia solution
$\text{Ni}^{2+}$	Green powder	Green	Green ppt of $\text{Ni}(\text{OH})_2$	The ppt does not dissolve on addition of excess NaOH	Green ppt of $\text{Ni}(\text{OH})_2$	The ppt dissolves to give a clear blue solution of complex ion $[\text{Ni}(\text{NH}_3)_6]^{2+}$
$\text{Fe}^{2+}$	Green powder	Green	Dark green ppt that turns brown on oxidation $\text{Fe}(\text{OH})_2 \rightarrow \text{Fe}(\text{OH})_3$	The ppt does not dissolve on addition of excess NaOH	Dark green ppt that turns brown on oxidation $\text{Fe}(\text{OH})_2 \rightarrow \text{Fe}(\text{OH})_3$	The precipitate does not dissolve
$\text{Fe}^{3+}$	Brown or yellow powder	Brown or yellow	Brown ppt of $\text{Fe}(\text{OH})_3$	The ppt does not dissolve on addition of excess NaOH	Brown ppt of $\text{Fe}(\text{OH})_3$	The precipitate does not dissolve
$\text{Mn}^{2+}$	Pink powder	Pink	White ppt but darkens with oxidation $\text{Mn}(\text{OH})_2 \rightarrow \text{Mn}_2\text{O}_3 \rightarrow \text{MnO}_2$	The ppt does not dissolve on addition of excess NaOH	White ppt but darkens with oxidation $\text{Mn}(\text{OH})_2 \rightarrow \text{Mn}_2\text{O}_3 \rightarrow \text{MnO}_2$	The precipitate does not dissolve
$\text{Cr}^{3+}$	Green powder	Green	Green ppt of $\text{Cr}(\text{OH})_3$	The ppt dissolves to give clear green solution of complex ion $[\text{Cr}(\text{OH})_6]^{3+}$	Green ppt of $\text{Cr}(\text{OH})_3$	dissolves - clear green solution of complex ion $[\text{Cr}(\text{NH}_3)_6]^{3+}$

Co <sup>2+</sup>	Pink powder	Pink	Blue ppt that turns pink on standing Co(OH) <sub>2</sub>	The ppt does not dissolve on addition of excess NaOH	Blue ppt that turns pink on standing Co(OH) <sub>2</sub>	The ppt dissolves to give a clear brown solution of complex ion [Co(NH <sub>3</sub> ) <sub>6</sub> ] <sup>2+</sup>
Cu <sup>2+</sup>	Blue crystal, white when anhydrous	Blue	Blue ppt. of Cu(OH) <sub>2</sub>	The ppt does not dissolve on addition of excess NaOH	Blue ppt. of Cu(OH) <sub>2</sub>	The ppt dissolves to give a clear blue solution of complex ion [Cu(NH <sub>3</sub> ) <sub>4</sub> (H <sub>2</sub> O)] <sup>2+</sup>
Zn <sup>2+</sup>	White powder	Colourless	White ppt. of Zn(OH) <sub>2</sub>	The ppt dissolves to give a clear colourless solution of the complex ion [Zn(OH) <sub>4</sub> ] <sup>2-</sup>	White ppt. of Zn(OH) <sub>2</sub>	The ppt dissolves- clear solution, colourless of complex ion [Zn(OH) <sub>4</sub> ] <sup>2-</sup>

### Confirmatory tests

The third test tube of each solution is treated as follows.:

- To the solution of copper sulphate, add potassium hexacyanoferrate (II) solution and record your observations.
- To the solution of iron (II) nitrate, add potassium hexacyanoferrate (III) solution.  
Write down your observations.
- To the solution of iron (II) nitrate, add potassium thiocyanate.  
What happens?
- To the solution of manganese (II) chloride, add little concentrated nitric acid followed by little solid lead (IV) oxide or solid sodium bismuthate (V) and boil the mixture.

What do you observe?

5. To the solution of chromium (III) nitrate add excess aqueous sodium hydroxide followed by little hydrogen peroxide and boil the resultant mixture.

What do you observe?

6. Then add silver nitrate solution and write down your observations.

7. To the solution of zinc sulphate add potassium ferrocyanide.

What happens?

8. To the solution of cobalt (II) nitrate, add potassium thiocyanate or ammonium thiocyanate solution and record your observations.

9. Add potassium cyanide to the solution of nickel sulphate. What do you observe?

Record all your observations in a summarised table

### Data recording

	Reagent	Observation
<b>Ni<sup>2+</sup></b>	Addition of potassium cyanide solution	<b>Yellow-green</b> precipitate of Nickel (II) cyanide  The ppt dissolves in excess reagent to give a dark yellow solution tetracyanonickel (II) ion.
<b>Fe<sup>2+</sup></b>	Addition of potassium hexacyanoferrate (III) solution	<b>Dark blue</b> precipitate
<b>Fe<sup>3+</sup></b>	Addition of potassium thiocyanate	<b>Blood red</b> coloration solution
<b>Mn<sup>2+</sup></b>	Conc. HNO <sub>3</sub> nitric acid followed by little solid sodium bismuthate (V) and boil the mixture.	A <b>purple</b> solution is formed due to MnO <sub>4</sub> <sup>-</sup> ion.
<b>Cr<sup>3+</sup></b>	NaOH + H <sub>2</sub> O <sub>2</sub> , then AgNO <sub>3</sub>	<b>Yellow</b> solution, then <b>brick red</b> ppt of silver chromate
<b>Co<sup>2+</sup></b>	Addition of potassium thiocyanate solution	a <b>blue</b> coloured product of potassium cobalt(II) tetrathiocyanate
<b>Cu<sup>2+</sup></b>	Potassium hexacyanoferrate (II) solution	<b>Brown</b> precipitate of Cu <sub>2</sub> Fe(CN) <sub>6</sub> (s) is formed.
<b>Zn<sup>2+</sup></b>	Potassium ferrocyanide	A <b>white</b> precipitate of Zn <sub>2</sub> [Fe(CN) <sub>6</sub> ](s)

## Interpretation of results and conclusion

### Guiding questions

1. Why are aqueous hydroxide ions used to test for cations?
2. What does happen when only a small amount of dilute ammonia solution is added to the hexaqua ions,  $[M(H_2O)_6]^{2+}$ ?
3. Which of the following ions produced identical observations when NaOH or ammonia solutions are added until in excess
  - a)  $[Fe(H_2O)_6]^{2+}$
  - b)  $[Cr(H_2O)_6]^{3+}$
  - c)  $[Co(H_2O)_6]^{2+}$
  - d)  $[Cu(H_2O)_6]^{2+}$
4. How does ammonia react with a solution of  $Cu^{2+}$ ?

### Answer to guiding questions

On reaction with a small amount of hydroxide ions, all ions under study, i.e,  $Ni^{2+}$ ,  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Mn^{2+}$ ,  $Cr^{3+}$ ,  $Zn^{2+}$ ,  $Co^{2+}$ ,  $Cu^{2+}$  form the corresponding hydroxides with **characteristic colours**. Only  $Zn^{2+}$  with complete **d orbital** forms a **colourless** hydroxide.

In addition to large amounts of hydroxide ions, only  $Cr^{3+}$  and  $Zn^{2+}$  form the corresponding complex ions,  $[Cr(OH)_6]^{3+}$  and  $[Zn(OH)_4]^{2-}$  respectively.

On standing in the air,  $Fe(OH)_2$  is oxidised to  $Fe(OH)_3$  and  $Mn(OH)_2$  is oxidised to  $MnO_2$ .

On addition of excess ammonia solution,  $Ni(OH)_2$ ,  $Cr(OH)_3$ ,  $Co(OH)_2$ ,  $Cu(OH)_2$ , and  $Zn(OH)_2$  undergo ligand exchange reaction to give the corresponding complex ions i.e  $[Ni(NH_3)_6]^{2+}$ ,  $[Cr(NH_3)_6]^{3+}$ ,  $[Co(NH_3)_6]^{2+}$ ,  $[Cu(NH_3)_4(H_2O)]^{2+}$  and  $[Zn(OH)_4]^{2-}$  respectively.

Note that the use of tap water may affect the expected results as it contains ions that may interfere with one's under study.

## Evaluation

1. Explain why on standing in the air,  $\text{Fe}(\text{OH})_2$  is oxidised to  $\text{Fe}(\text{OH})_3$  and  $\text{Mn}(\text{OH})_2$  is oxidised to  $\text{MnO}_2$ .
2. Write the balanced chemical equations of formation of the hexammine nickel (II),  $[\text{Ni}(\text{NH}_3)_6]^{2+}$ , the hexammine chromium (III),  $[\text{Cr}(\text{NH}_3)_6]^{2+}$  and the tetrahydroxozincate,  $[\text{Zn}(\text{OH})_4]^{2-}$  from the corresponding hydroxides.
3. You are provided a substance **X** which contains a cation of a transition metal. You are required to identify the cation in **X**.

Perform the following tests, record your observations and deduce the conclusion in the table below.

Tests	Observations	Deductions
Take a spatula endful of compound <b>X</b> , record its appearance, and put it in clean test tube		
To the content of the test tube, add 15 mL of distilled water to make a solution of compound <b>X</b> and record its colour.		
Divide the prepared solution into three portions.		
To the first portion add 5 drops of sodium hydroxide.		
Add more sodium hydroxide until there is no observable change.		
To the second portion, add 5 drops of ammonia solution		
Add more ammonia solution until there is no observable change		
To the third portion, add potassium cyanide solution		

## EXPERIMENT 2.1: Extraction of copper

### Rationale

Metals are extracted from their ores through chemical reactions. They are reduced by carbon to produce pure metals. For example, the copper is extracted from its oxide ore. Copper is used in electrical equipment such as wiring and motors. It also has uses in construction and industrial machinery. This experiment is very important to understand the concept of oxidation and reduction reaction.

### Objective

Learners will be able to extract copper using copper oxide and charcoal (carbon) based on its position in reactivity series.



### Required materials

#### Apparatus

- Beakers
- Ignition tube/  
borosilicate test tube
- Test tubes rack
- Test tube holders
- Spatula
- Eye protective  
goggles
- Gloves
- Bunsen burner
- Pair of tongs

#### Chemicals

- Copper oxide
- Charcoal powder

## Experimental set-up

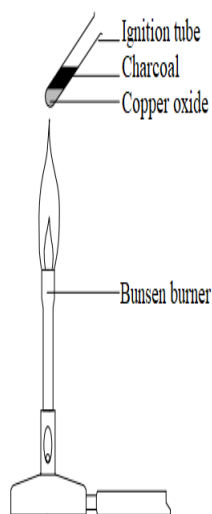


Figure 2.1: The laboratory extraction of copper from copper oxide



### Procedure

1. Hold the ignition tube with tongs
2. Put a full spatula of copper oxide (with precaution) into the ignition tube.
3. Cautionary, add spatula full of charcoal powder on top after putting copper oxide, do not mix. Sturdily heat the two layers in the ignition tube for five minutes over a Bunsen burner flame.
4. Allow to cool and then look closely at the ignition tube where the powders meet. What do you observe?

**Answer:** A red powder is observed in the ignition test tube

5. Add 5 mL of water after the mixture has cooled. What do you observe?

**Answer:** Charcoal floats on the water while copper (denser) sinks to the bottom.

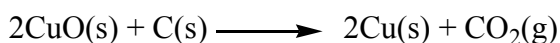
## Interpretation of results and conclusion

### Guiding questions

1. What does the red powder observed in the test tube after heating the mixture of copper oxide with charcoal powder mean?
2. Name the chemical reaction that occurs.
3. Write a balanced chemical equation of the extraction of copper from copper oxide.

### Answer to guiding questions

Metals in their compounds are in oxidised form (have lost electron(s) and are positively charged). The reduction of copper oxide with a reducing agent such as carbon (charcoal) yields the formation of copper through cuprous oxide as the intermediate product. Carbon in the reactivity series reduces the metal oxides to the metals and gets oxidised to carbon dioxide:



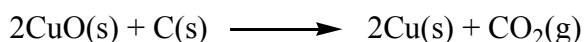
Smelting is a process by which a metal is obtained from its oxide ore by heating it beyond the melting point in presence of reducing agents like coke.

### Evaluation

1. What happens to the charcoal (carbon) after taking oxygen from the copper oxide?

**Answer:** When charcoal (carbon) reacts with copper oxide, carbon dioxide and copper are produced.

*The chemical equation reaction:*



What is the importance of adding water after the reaction?

**Answer:** After the reaction, water is added to separate copper from any excess charcoal that was used. The charcoal can be skimmed off because it will float on the water while copper (more dense) will sink to the bottom.



## EXPERIMENT 5.1:

## Experiment to test the acidity of phenol in comparison to alcohols and carboxylic acids

## Rationale

Phenol or phenolic compound is a class of organic compounds that have a hydroxyl group (-OH) attached directly to an aromatic ring. They are widely used in the food and medicinal industries as antioxidant, anticarcinogenic, or antimutagenic and anti-inflammatory agents. This experiment helps to differentiate the phenol from alcohols and carboxylic acids.

## Objective

Learners will be able to distinguish the relative acidities of alcohols, phenols and carboxylic acids



## Required materials

## Apparatus

- Test tubes
- Test tubes rack
- Beakers
- Tongs
- Retort stand
- Dropper
- Knife
- Delivery tube

## Chemicals

- Ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$
- Propanol,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$
- Phenol (2M),  $\text{C}_6\text{H}_5\text{OH}$
- Ethanoic acid,  $\text{CH}_3\text{COOH}$  (1M)
- Propanoic acid,  $\text{CH}_3\text{CH}_2\text{COOH}$  (1M)
- Sodium hydrogen carbonate,  $\text{NaHCO}_3$  (1M)

### Apparatus

- Whistle

### Chemicals

- Sodium carbonate,  $\text{Na}_2\text{CO}_3$  (1M)
- Sodium hydroxide,  $\text{NaOH}$  (1M)
- Sodium metal
- Blue litmus paper

### Experimental set-up

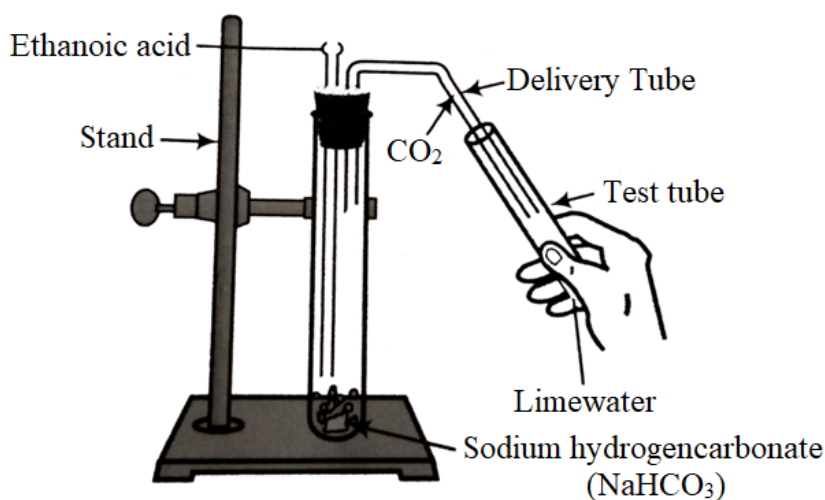


Figure 5.1: Reaction of carboxylic acids with sodium hydrogen carbonate



### Procedure

You are provided ethanol, ethanoic acid, propanol, propanoic acid and phenol as substances to be tested.

1. Put 1 drop of each of them on a piece of wet blue litmus paper. What do you observe?

**Answer:** Blue litmus paper turns red in contact with ethanoic acid and propanoic acid. It turns pale red (pink) in contact with phenol. Its colour does not change in contact with ethanol or propanol.

2. To 1 mL of each of the solutions above, add 5 drops of a 1M sodium hydrogen carbonate solution. What do you observe?

**Answer:** Effervescence with only ethanoic and propanoic acids

3. Repeat step 2 using a 1M sodium carbonate solution. What does it happen?

**Answer:** Effervescence with only ethanoic and propanoic acids

4. Repeat step 2 using a 1M sodium hydroxide solution and record your observations.

**Answer:** There is formation of soluble salts, with only phenol and carboxylic acids

5. To 10 mL of the substance tested, carefully add a small piece of sodium (a quarter of the size of pea). What do you observe?

**Answer:** A colourless gas (that gives a pop sound when it is burnt) is released.

6. Record all your observations in the table below.

### Data recording

Reagent	ANALYTES				
	Ethanol	Ethanoic acid	Propanol	Propanoic acid	Phenol
Blue litmus paper	no change	turns red	no change	turns red	turns pale red
NaHCO <sub>3</sub>	no reaction	effervescence	no reaction	effervescence	no reaction
Na <sub>2</sub> CO <sub>3</sub>	no reaction	effervescence (react vigorously)	no reaction	effervescence (react vigorously)	no reaction
NaOH	no reaction	No observable change	no reaction	No observable change	No observable change
Na metal	colourless gas released	colourless gas released	colourless gas released	colourless gas released	colourless gas released

**Caution:**

Phenol is highly toxic. Therefore, handle it with great care.

Sodium reacts vigorously with water present in the air. Therefore, quickly close the container when you have taken the required amount for the experiment.

**Interpretation of results and conclusion****Guiding questions**

According to this experiment, how can you explain the acidity strength of ethanol, phenol and propanoic acid?

**Answer to guiding questions**

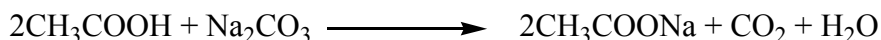
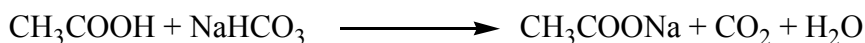
In aqueous ethanol solution, the colour of blue litmus paper is not affected. That is, ethanol does not release  $H^+$  ions in aqueous solution. Propanol behaves in a similar way.

Ethanoic acid turns blue litmus paper red that is, ethanoic acid releases  $H^+$  ions in aqueous solution. Likewise, propanoic acid turns blue litmus paper red.

Phenol turns blue litmus paper pale red. This means that it is not acidic enough to release  $H^+$  ions in aqueous solutions.

Ethanol, propanol and phenol do not react with sodium hydrogen carbonate. They are not acidic enough to decompose a metal hydrogen carbonate or carbonate.

When ethanoic acid and propanoic acid are reacted with sodium hydrogen carbonate, bubbles of a carbon dioxide colourless gas are released. The chemical equations of the reaction are given below:



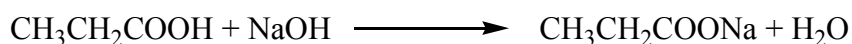
This means that carboxylic acids are more acidic than phenols and alcohols.

Ethanol and propanol do not react with sodium hydroxide but phenol does according to the following chemical equation:

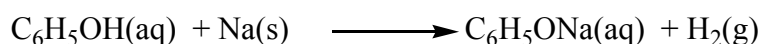
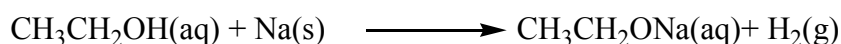


This means that phenol is more acidic than alcohol.

Ethanoic and propanoic acids also react with sodium hydroxide to give sodium ethanoate and propanoate respectively.



All analytes react with sodium metal by releasing a hydrogen gas that gives a pop sound when it is burnt. The chemical equations of the reaction are given below:



Considering the results above, the order of increasing acidity is:

***Alcohols < phenol < carboxylic acids***

This symbol "<" means "is less acidic than"

### Evaluation

How would you differentiate experimentally between alcohols, phenol and carboxylic acids?

**Answer:** See interpretation

## EXPERIMENT 5.2:

## Experiment to test for the presence of phenol in a solution

### Rationale

Phenols are a class of organic compounds with hydroxyl group (-OH) attached directly to aromatic rings. These aromatic hydrocarbons have a wide range of applications. In industry, phenols are used as raw materials for making drugs, preservatives, explosives, disinfectants. Since one or more phenolic groups may be attached to one aromatic ring, it is very important to identify phenol in an aromatic compound. This experiment shows how the phenolic functional group can be distinguished among other aromatic compounds by various tests to identify their properties and applications.

### Objective

Learners will be able to identify phenol using some simple tests



### Required materials

#### Apparatus

- Test tubes
- Test tubes racks
- Droppers
- Beakers
- Spatula
- Test tube holders
- Bunsen burner
- Filter paper

#### Chemicals

- Iron (III) chloride ( $\text{FeCl}_3$ ) solution
- Phenol,  $\text{C}_6\text{H}_5\text{OH}$  (to be tested)
- Phthalic anhydride  $\text{C}_6\text{H}_4(\text{CO})_2\text{O}$
- Sodium nitrite,  $\text{NaNO}_2$
- Blue litmus paper
- Conc. Sulphuric acid,  $\text{H}_2\text{SO}_4$
- Distilled water

### Caution:

- Phenol is highly toxic and corrosive, so handle it with great care.
- The ferric chloride solution used should be freshly prepared, neutral and very dilute.
- Bromine should not be inhaled because it causes irritation.
- Concentrated acids should be handled with care.



## Procedure

### a) Ferric Chloride Test:

1. Wear safety gloves.
2. Put a spatula endful of iron (III) chloride in a test tube and add 10 mL of distilled water;
3. Add diluted solution of sodium hydroxide to ferric chloride [iron (III) chloride] solution drop by drop until a small but permanent brown precipitate appears,
4. Filter the solution and use the clear filtrate for the test;
5. Dissolve 1 spatula of phenol in 10 mL of water
6. Add a neutral solution of ferric chloride (filtrate) slowly dropwise. What do you observe?

**Answer:** Formation of a purple, blue, green or violet solution.

### b) Libermann's Test:

1. Place 1 g of the crystals of sodium nitrite in a clean dry test tube;
2. Add 5 mL of distilled water to the sodium nitrite;
3. Add 1 g of phenol to sodium nitrite solution and shake the test tube well;
4. Heat the mixture gently and allow it to cool;

Add 1 mL of concentrated sulphuric acid to it and shake the content.

What do you observe?

**Answer:** formation of deep blue or green colored product

5. Dilute the solution with water. What do you observe?

**Answer:** blue or green colored solution changes to red or brown colour

6. Add sodium hydroxide solution.

What do you observe?

**Answer:** Red or brown colour solution turns again to blue or green product

### c) Phthalein Dye Test:

1. Use a spatula to take 100 mg of phenol and put it in a clean and dry test tube.
2. Add 100 mg of phthalic anhydride to it.
3. Add 2-3 drops of concentrated sulphuric acid to the mixture.
4. Heat the contents of the test tube into a water bath for 2-3 minutes.
5. Allow the mixture to cool for some time and pour it into a beaker containing dilute sodium hydroxide solution.
6. Dilute the whole mixture with equal volume of water. What do you observe?

*Answer: colourless solution is formed and turns to pink*

### d) Litmus Test:

1. Place the drop of phenol solution or a small crystal on moist blue litmus paper.
2. Observe the change in colour.

*Answer: if it changes to red then phenolic group may be present*

### e) Bromine water test

1. Take 5 mL of bromine add 100 mL of distilled water and shake well to make a bromine reagent
2. Decant off the clear orange liquid,
3. Dissolve the given organic compound (phenol) in glacial acetic acid.
4. Add bromine water reagents to this dropwise. What do you observe?

*Answer: Bromine colour disappears, and white precipitate is formed.*



## Interpretation of results and conclusion

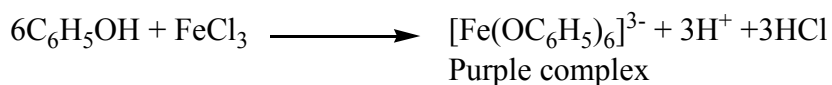
### Guiding questions

1. What is the product formed during the preparation of the ferric chloride solution?
2. How can phenol be test? Use table for showing all the observations.

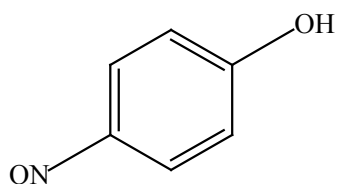
### Answer to guiding questions

During the preparation of the ferric chloride solution, the brown precipitate formed is ferric hydroxide

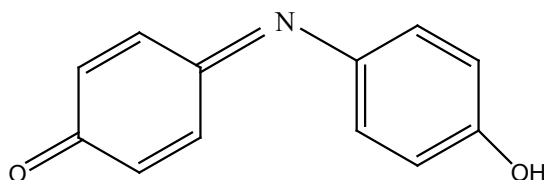
- When a phenol reacts with iron (III) solution, the solution becomes **purple**, blue or green. The reaction that takes place is:



Phenol, in reaction with sodium nitrate in the presence of sulphuric acid gives a deep blue or green coloured solution that is *4-nitrosophenol*. When distilled water is added, the colour of the solution changes to red or brown colour. With excess of phenol and sulphuric acid a deep blue indophenol complex is formed. On dilution, a **red colour indophenol** is formed which turns to **deep blue colour sodium** salt solution of indophenol on treatment with sodium hydroxide.



Yellow 4-nitrosophenol



Red indophenol

- When phenol is heated with phthalic anhydride in the presence of concentrated sulfuric acid, yields a colourless solution, phenolphthalein, which turns pink in the presence of sodium hydroxide.
- When bromine water is added to a solution containing phenol, the bromine water is decolorized. In addition, a white precipitate form.

Reagents	Observation
Blue litmus test	Phenol turns blue litmus paper red.
Ferric Chloride test	Violet or blue colouration shows presence of phenol.
Libermann's test	Deep blue colour solution shows the presence of phenol.
Phthalic dye test	Pink colour solution shows the presence of phenol.
Bromine water test	Formation of white precipitate shows presence of phenol.

### Evaluation

Describe how to test phenol using different reagents.

*Answer: see the interpretation*

## EXPERIMENT 5.3: Reaction of phenol with bromine

### Rationale

Phenols are a class of organic compounds with hydroxyl group (-OH) directly attached to aromatic rings. These aromatic hydrocarbons have a wide range of applications. In industry, phenols are used as raw materials for making drugs, preservatives, explosives, disinfectants, etc. Since one or more phenolic groups may be attached to one aromatic ring, it is very important to identify phenol among other aromatic compounds. This experiment reveals how the phenolic functional group can be differentiated from other aromatic compounds by various tests to identify their properties and applications.

### Objective

Learners will be able to determine the products of the reaction of phenol with bromine



## Required materials

### Apparatus

- Test tubes
- Test tubes rack
- Beakers
- Test tubes holders
- Droppers
- Protective gloves
- Eyes goggles
- Conical flask

### Chemicals

- Chemicals
- Phenol,  $C_6H_5OH$  (2M)
- Bromine water
- Distilled water
- Glacial acetic acid

## Experimental set-up

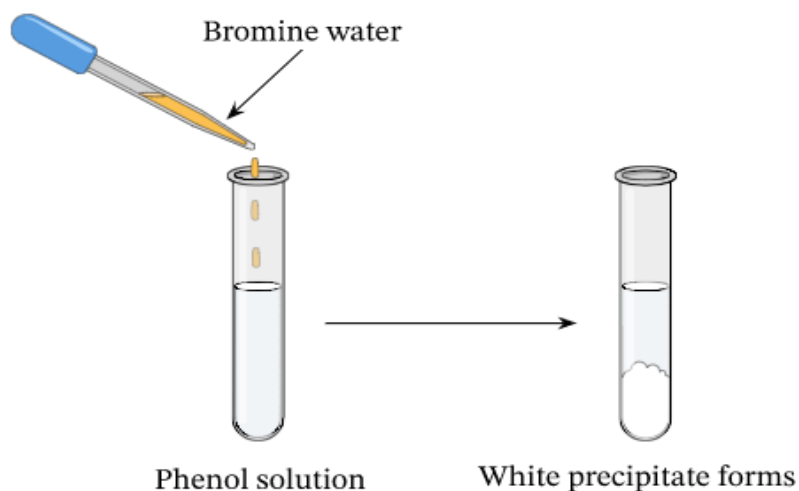


Figure 5.2: Reaction of bromine with a phenol

## Safety precautions

- Glacial acetic acid should be handled very carefully since it is a corrosive poison that can cause injury or death of human tissue exposed to it.
- Bromine water and phenol should be handled very carefully because. They may cause irritation to eyes, skin, throat, nose, etc.



## Procedure

1. Put 3 mL of bromine in a clean conical flask, add 20 mL of distilled water and shake well
2. Decant off the clear liquid
3. Dissolve 5 mL of phenol 2M in 10 mL of glacial acetic acid
4. Add bromine water solution dropwise. What do you observe?

**Answer:** White precipitate is formed

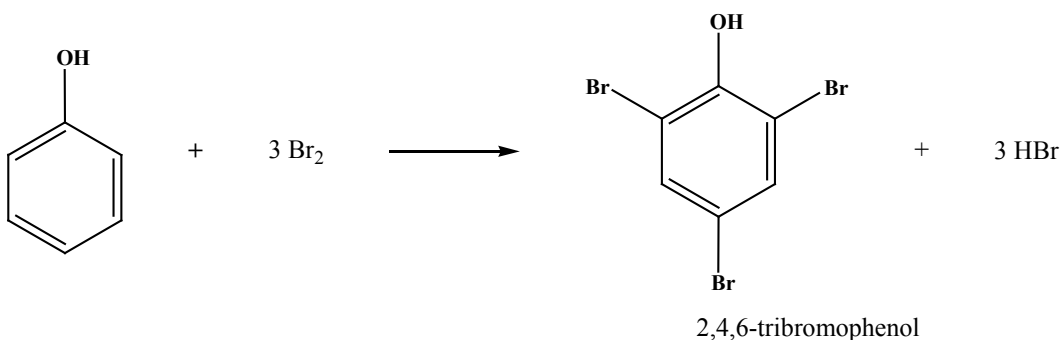
## Interpretation of results and conclusion

### Guiding questions

1. Explain how phenol reacts with bromine.
2. Write a balanced equation of reaction of phenol and bromine

### Answer to guiding questions

When bromine water is added to a solution of phenol in water, it is decolourised, and a white precipitate is formed. The precipitate is 2,4,6-tribromophenol. The hydroxyl group attached to the aromatic/benzene ring being an activator, will direct bromine to ortho and para position toward it.



## Evaluation

Why can phenol react with bromine without a catalyst whereas benzene reacts with it in the presence of halogen carrier catalyst?

**Answer:** In phenol, OH group is an activator of benzene ring.

## EXPERIMENT 5.4 :

## Test and compare alkalinity of phenyl amines, aliphatic amines and ammonia

### Rationale

Aliphatic amines, aromatic amines and ammonia present many applications in the manufacture of rubber industry, synthesis of azo dye used in the manufacture of clothes, drugs, pesticides, polymers like nylon. This experiment is therefore important to compare the basicity of ammonia, aliphatic amines and aromatic amines to identify their properties and uses.

### Objective

Learners will be able to test and compare the alkalinity of ammonia, aliphatic and aromatic amines.



### Required materials

#### Apparatus

- Test tubes with corks
- Test tubes racks
- Beakers
- Spatula
- Test tube holders
- Droppers
- pH meter/
- Eye goggles
- Protective gloves
- Wash bottle
- Watch-glass

#### Chemicals

- Ammonia ( $\text{NH}_3$ ) solution, 2M
- sodium nitrite ( $\text{NaNO}_2$ ) 20%
- Phenylamine,  $\text{C}_6\text{H}_5\text{NH}_2$
- Distilled water
- Copper (II) sulphate ( $\text{CuSO}_4$ ) solution, 1M
- Hydrochloric acid, HCl
- Ice
- Butylamine,  $\text{C}_4\text{H}_{11}\text{N}$
- N-methylbutylamine,  $\text{C}_5\text{H}_{13}\text{N}$
- Triethylamine,  $\text{C}_6\text{H}_{15}\text{N}$
- Beta-naphthol,  $\text{C}_{10}\text{H}_8\text{O}$
- Sodium hydroxide, NaOH, 2M
- indicator paper



## Procedure

### a) Test of amines

1. Dissolve 2-3 drops of liquid butylamine in 2.0 mL of 2.0 M HCl in a small test tube.
2. Cool to 5-10 °C in an ice bath.
3. Add 5 drops of cold 20% aqueous sodium nitrite solution.

What do you observe?

**Answer:** *A colourless gas is produced.*

4. Repeat steps 1-3 using successively N-methyl butylamine, triethylamine and phenylamine (aniline). What do you observe?

**Answer:** *With N-methyl butylamine, an insoluble yellow liquid is observed. With triethylamine there is no gas evolved and no yellow liquid separated. With phenylamine, there is no gas evolved and no yellow liquid separated.*

5. If there is no observation, then add a few drops of the cold reaction mixture to a cold solution of 50 mg beta-naphthol in 2 mL of 2.0 M sodium hydroxide.

What do you observe?

**Answer:** *The solution turns orange red when phenylamine is tested.*

### b) Alkalinity strength of amines

1. Prepare 50 mL of a 0.001M aqueous solution of ammonia, butylamine, N-methyl butylamine, triethylamine, phenylamine, and 4-methylphenylamine;
2. Test the nature of each solution using blue and red litmus papers.

What do you observe?

**Answer:** *All solutions turn red litmus paper blue*

3. Measure the pH of each solution using indicator paper and a pH metre and record your findings in the table below.

## Data recording

### a) Tests of amines

	Observations
Butylamine	Colourless
N-methylbutylamine	Insoluble yellow liquid
Triethylamine	No observable change
Phenylamine (aniline)	No observable change

### b) Alkalinity strength of amines and ammonia

	pH of the solution
Butylamine	
N-methylbutylamine	
Triethylamine	
Phenylamine (aniline)	
4-methylphenylamine	

## Interpretation of results and conclusion

### Guiding questions

1. How can you distinguish aliphatic amines, aromatic amines and ammonia? Support your answer by using the chemical test.
2. What is the feature that makes the above compounds basic?
3. Explain why phenylamine is such a weak base?

### Answer to guiding questions

#### a) Test of amines

When amine reacts with sodium nitrite, there is immediate evolution of a colourless gas (nitrogen) which indicates the presence of a primary aliphatic amine.

If no gas was evolved but an insoluble yellow or orange liquid separated from the solution, then this indicates the presence of a secondary aliphatic or

aromatic amine.

If no gas is evolved and no yellow liquid separated, then this means that no reaction occurred, and the amine tested is a tertiary amine.

Another possibility is that the compound is a primary aromatic amine. These form diazonium salts that are stable at low temperatures.

The formation of a yellow/orange precipitate when treated with a phenol indicates a primary aromatic amine.

### **b) Alkalinity strength and ammonia**

All amines like ammonia are weak bases. They are partially dissociated in aqueous solution.

The measure of the pH should show the following order in alkalinity strength in aqueous solution.

**Secondary amines > tertiary amines > primary amines > ammonia > phenylamines**

Where ">" means "more alkaline than"

Ammonia and amines are basic compounds because of the free electron pair on nitrogen.

### **Evaluation**

Arrange the following compounds according to their decreasing order of alkalinity.  $C_6H_5NH_2$ ,  $(CH_3)_3N$ ,  $NH_3$ ,  $CH_3CH_2CH_2NH_2$  and  $CH_3CH_2NHCH_3$

**Answer:**  $CH_3CH_2NHCH_3 > (CH_3)_3N > CH_3CH_2CH_2NH_2 > NH_3 > C_6H_5NH_2$

Where ">" means "more alkaline than"



## EXPERIMENT 5.5:

## Reactions of phenyl amines with water, hydrochloric acid and phenol

### Rationale

Anilines are a class of organic compounds with an amino group ( $-\text{NH}_2$ ) directly attached to aromatic rings. These aromatic amines have a wide range of applications. In industry, anilines are used as raw materials for making drugs, polymers, dyes, fungicides, insecticides, explosives. This experiment demonstrates that it is important to know the reactivity of phenyl amines and relate them to their uses.

### Objective

Learners will be able to explain the reaction of phenyl amines with water, hydrochloric acid and phenols.



### Required materials

#### Apparatus

- Test tubes with corks
- Test tubes racks
- Beakers
- Spatula
- Test tube holders
- pH metre
- Droppers
- Eye goggles
- Protective gloves
- Wash bottle
- Stirring rod
- Thermometer

#### Chemicals

- 3-methylphenylamine
- Phenol
- Phenylamine
- Hydrochloric acid
- Sodium hydroxide
- Distilled water
- Indicator paper
- Blue and red litmus papers



## Procedure

### a) Reaction with water

1. To 10 mL of distilled water in a beaker, add 1 mL of phenylamine.
2. Using a glass rod, stir to mix.
3. Put 2 drops of the solution on red and blue litmus papers. What do you observe?

*Answer: Aqueous solution of phenylamine turns red litmus paper blue and has no effect on blue litmus paper.*

4. Repeat the procedure using 3-methylphenylamine and record your observations

*Answer: Aqueous solution of 3-methylphenylamine turns red litmus paper blue and has no effect on blue litmus paper.*

### b) Reaction with hydrochloric acid

1. To 10 mL of distilled water in a beaker, add 1 mL of phenylamine.
2. Using a glass rod, stir to mix.
3. Record the temperature of the solution.
4. To 3 mL of the solution, add 5 mL of hydrochloric acid and record the temperature. What do you observe?

*Answer: The pungent smell of phenylamine disappears and the temperature of the mixture rises.*

5. Then add excess sodium hydroxide to the solution. What do you observe?

*Answer: The smell of phenylamine returns*

### c) Reaction with phenol

1. Take 3 large test tubes and label them 1, 2, and 3;
2. In test tube 1, put 1 g of aniline, 5 mL of water and 2.5 mL of concentrated hydrochloric acid, shake to dissolve and keep the mixture in an ice bath.
3. In test tube 2, put 1 g of sodium nitrite ( $\text{NaNO}_2$ ) and 5 mL of distilled water. Shake to dissolve and place in ice bath

4. In test tube 3, put 1 g of phenol and add enough 10 mL of 10% NaOH solution. Shake to dissolve and keep the mixture in ice bath.
5. Slowly mix the content of test tube 1 and 2.
6. Then carefully add mixture to test tube 3. What do you observe?

*Answer: A yellow orange precipitate forms.*

## Interpretation of results and conclusion

### Guiding questions

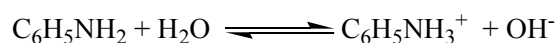
1. Explain why does phenylamine form an alkaline solution?
2. What is the name given to the reaction between phenylamine and a mixture of sodium nitrite and hydrochloric acid?
3. During the reaction between aniline and phenol, nitrous acid is produced in situ. Explain why.
4. Give the use of the product formed when phenylamine react with phenol

### Answer to guiding questions

#### 1) Reaction with water

Phenylamine reacts reversibly with water to give phenyl ammonium ions and hydroxide ions. The position of equilibrium lies well to the left of the corresponding ammonia or aliphatic amine equilibria - which means that not many hydroxide ions are formed in the solution.

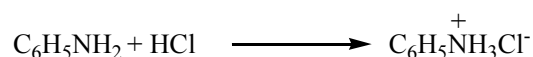
The chemical equation for the reaction:



#### 2) Reaction with hydrochloric acid

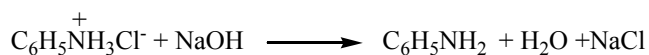
When hydrochloric acid is added to phenylamine, a soluble salt is formed. Like other salts it is nonvolatile and hence has no smell.

The chemical equation of the reaction:



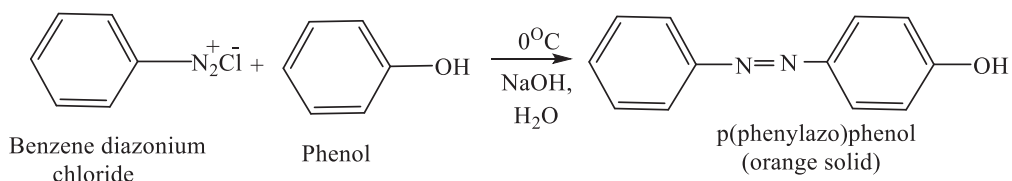
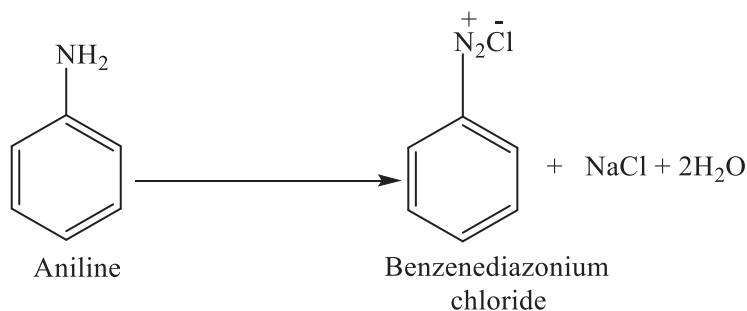
When sodium hydroxide, a strong base is added to the salt, protons are removed from it and the free amine is regenerated.

The chemical equation of the reaction:



### 3) Reaction with phenol

When benzene diazonium chloride reacts with phenol in basic medium a **yellow-orange** azo dye (benzene azo phenol) is formed. This is a coupling reaction.



### Evaluation

1. Write chemical equations of the reactions of 4-bromophenyl amine with:
  - a) water,
  - b) hydrochloric acid
  - c) Phenol.

**Answer:** see the interpretation

2. Give an example and highlight the importance of the reactions of phenylamines with phenols.

**Answer:** These reactions produce azo dyes

**EXPERIMENT 6.1: Preparation of casein plastic from milk****Rationale**

Plastic has gradually become an indispensable assistant for our everyday lives. It is lightweight, inexpensive, pleasant to touch and possible to use for almost any purpose. Plastics are mainly polymers. The plastic made by the present experiment can be used to make many different plastic ornaments. These include buttons, decorative buckles, beads, fancy comb and brush sets.

**Objective**

Learners will be able to prepare casein plastic from fresh milk.

**Required materials****Apparatus**

- One beaker of 100 mL
- Two beakers of 1 L each
- 1 spatula
- 1 strainer
- Stand
- Thermometer
- Hot plate or Bunsen burner
- Matches if using a Bunsen burner
- Stirring rod

**Chemicals**

- 500 mL fresh fat milk
- 60 mL vinegar
- Aluminium foil or paper towel



## Procedure

1. Turn on the Bunsen burner or hotplate.
2. Place the beaker of milk (500 mL) over heat. Stir constantly.
3. Place the thermometer into the milk. Heat milk until it reaches 37°C.
4. Remove the milk from heat and immediately add 60 mL vinegar and stir constantly.

What do you observe?

**Answer:** *The solution is rapidly separated into a soft, white substance suspended in a clear yellow liquid.*

5. Strain the solution through the strainer into the empty 1L beaker. Hold up the beaker to see the clear liquid.
6. Scoop the curd onto a large piece of aluminium foil and press out into a thin layer. Pass the aluminium foil around the curd. Then allow the curd to dry overnight.

## Interpretation of results and conclusion

### Guiding question

What happens to milk when an acid like vinegar or lemon is added to it?

### Answer to guiding question:

When acidic substances like vinegar or lemon juice are added to milk, the milk protein coagulates into solid masses called curd (soft, white substance). The water separated out and is drained off to retain only the curd. This process of milk coagulating to form curd is known as curdling.

Milk is a colloid, which is defined as a suspension of large molecules; casein proteins in a solution such as water. On the addition of acid to the milk, these proteins change their nature and are prone to unfolding and rearranging into the long chains of polymer. The process then causes the casein to precipitate out of the milk, leaving a clear watery substance behind. The casein can then be formed into various shapes before drying. Once it has dried enough it is called casein plastic.

## Evaluation

Why is hot milk mostly preferred in this experiment?

**Answer:** thermal energy in the form of heat was applied to speed the process and cause a more complete separation.

## EXPERIMENT 6.2:

### Laboratory preparation of phenol-methanal polymer (bakelite)

## Rationale

Phenol formaldehyde resins also called phenolic resins are synthetic polymers made by the reaction of formaldehyde with phenol or substituted phenol. They have a wide range of applications including laboratory countertops, adhesives, billiard balls, laminating resins, surface coatings, binders. They are human made and have no effects on human health.

## Objective

Learners will be able to prepare phenol-formaldehyde resin on a small scale.



## Required materials

### Apparatus

- Glass rod
- Beakers
- Funnel
- Measuring cylinder
- Dropper
- Filter paper
- Bunsen burner

### Chemicals

- Phenol
- 40% formaldehyde solution
- Conc.  $\text{H}_2\text{SO}_4$
- Glacial acetic acid

## Safety precautions

- Concentrated sulphuric acid is corrosive. Wear safety gloves when handling it.
- Formalin (40% formaldehyde solution) is irritant to the skin, eyes, and mucous membranes. Take great care when using it.
- Phenol is toxic via skin contact. It is listed as a carcinogen.
- Glacial acetic acid is an irritant and can cause burns on contact.
- Work under a fume hood.



## Procedure

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40% formaldehyde solution in a 500 mL beaker inside the fume hood and add 2 grams of phenol.
2. Add 2 mL of concentrated sulphuric acid into the mixture and carefully heat for at least 5 minutes. What do you observe?

*Answer: A large mass of plastic is formed.*

3. Wash the residue obtained several times with distilled water, filter and dry the product.
4. Weigh the mass of the product.

## Data recording

Mass of the product obtained (Bakelite) formed= .....g

## Interpretation of results and conclusion

### Guiding questions

1. What are the reagents needed during the laboratory preparation of Bakelite?
2. Suggest the role for each of the following reagents
  - a) Glacial acetic acid
  - b) Sulphuric acid
3. Why is it suggested to perform the experiment in the fume board?



## Answer to guiding questions

A mixture of phenol and formaldehyde are allowed to react in the presence of an alkali or acid catalyst. The process is the condensation between phenol and formaldehyde, and it involves formation of methylene bridges in *ortho*, *para* or both *ortho and para* positions.

This results first in the formation of a linear *polymer* (called NOVOLAC) and then into *cross-linked polymer* called phenol-formaldehyde resin or bakelite.

## Evaluation

1. Differentiate between “novolac” and “Bakelite” and give at least one use of each.

**Answer:** See interpretation

*NOVOLAC is used in microelectronics*

*Bakelite is a good insulator that is used as electrical devices such as sockets, wire insulators, switches.*

2. What type of reaction is the preparation of bakelite and under what conditions it is performed?

**Answer:** Polymerisation in acidic medium and heating needed.

3. Is bakelite:

- a) A thermosoftening or thermosetting polymer? Explain.

**Answer:** Bakelite contains cross links, or heavily branched polymer chains. Bakelite polymer, upon heating, is hardened and cannot be softened again. Hence, it is a thermosetting polymer.

- b) Biodegradable or non-biodegradable polymer? Give a reason.

**Answer:** It is non-biodegradable, it is chemically very stable

**EXPERIMENT 7.1: Solvent extraction****Rationale**

Solvent extraction also known as liquid-liquid extraction or partitioning is a technique for separating chemicals based on their respective solubilities in immiscible liquids. It can be used to recover a valuable substance from the original solution, or to purify the original solvent by removing an unwanted component. This technique is used in various applications such as ore processing, pharmaceuticals, agriculture, industrial chemicals, petrochemicals, food industry, purification and refining of precious metals.

**Objective**

Learners will be able to correctly extract ammonia based on its partitioning between water and ethoxyethane

**Required materials****Apparatus**

- Separating funnel
- Erlenmeyer flask
- Graduated cylinder
- Retort stand and accessories
- Cork
- Pipette
- Burette

**Chemicals**

- Ammonia solution (0.1M)
- Ethoxyethane
- Hydrochloric acid (0.1M)
- Phenolphthalein indicator

## Apparatus

- Dropper
- Filter funnel
- Washing bottle

## Chemicals

### Experimental set-up

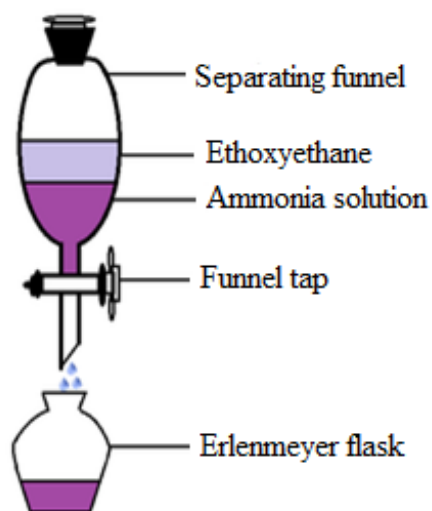


Figure 7.1: A one-extraction when the organic layer is on the top

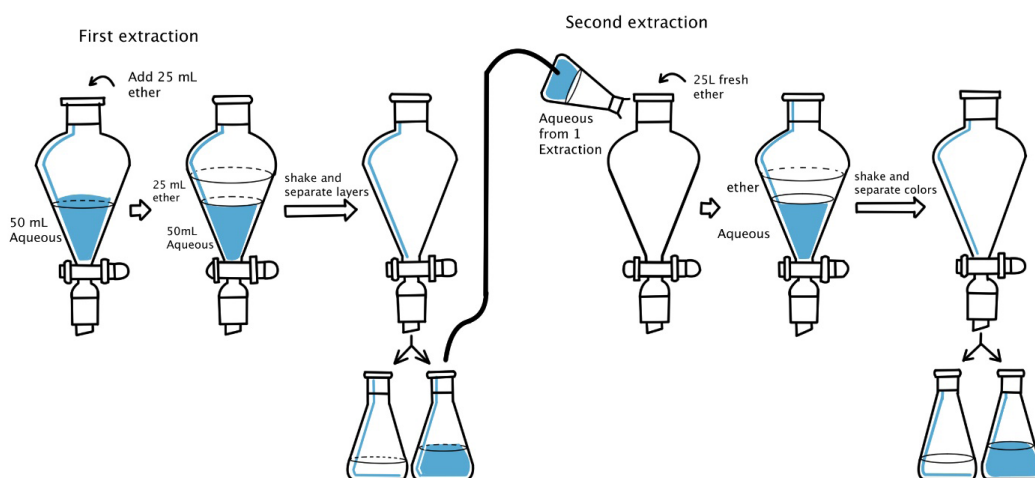


Figure 7.2: Two successive extractions when the organic layer is on the top

## Safety precautions

Never point the stopcock toward someone as you vent a separating funnel, as it is possible some liquid may splatter onto him or her.



## Procedure

1. Measure 50 mL of a 0.1M ammonia solution using a graduated cylinder;
2. Transfer, using a funnel, the ammonia solution to a separating funnel;
3. Measure 25 mL of ethoxyethane using a graduated cylinder;
4. Add ethoxyethane to the content of the separating funnel without mixing;
5. Stopper the separating funnel and shake it gently, venting frequently for 2 minutes and record your observations;
6. Take the stopper off to prevent pressure build-up;
7. Let the mixture sit in the ring clamp until the two layers have fully separated then remove the bottom layer;
8. Drain most of the bottom layer into a clean conical flask, positioning the ring clamp so that the tip of the separating funnel is well settled in the conical flask to prevent splashing;
9. Stop draining when the interface is within 1 cm of the bottom of the stopcock;
10. Gently shake the funnel to dislodge any droplets clinging to the glass. A glass stirring rod can be used to knock down stubborn clinging droplets;
11. Further drain the bottom layer, stopping when the interface just enters the stopcock chamber. Label the conical flask (e.g. "bottom layer");
12. Perform a second extraction;
13. Return the **aqueous layer** to the separating funnel. There is no need to wash the funnel in between extractions;
14. Add a **fresh** 25 mL portion of diethylether to the separating funnel. Stopper the funnel, invert and shake with venting, then allow the layers to separate;

15. Repeat the process of extraction and separation one more time;
16. Pull together all the organic layers collected;
17. Use a sample of 15 mL each of the organic layer and the aqueous layer and titrate ammonia using a 0.1 molar hydrochloric acid.

## Interpretation of results and conclusion

### Guiding questions

1. What are immiscible solvents used in solvent extraction experiment?
2. Between water and ethoxyethane solvent, which one do you think will dissolve more ammonia? Why?
3. (a) What is the difference between simple extraction and multiple extraction in solvent extraction experiment?  
(b) Explain the reason why multiple extractions are recommended in solvent extraction

### Answer to guiding questions

Solvent extraction or liquid-liquid extraction is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent.

The process of extraction when carried out with the total amount of a given solvent in a single operation is referred to as simple extraction. But when the extraction is made in two or more successive operations using small portions of the solvent provided, this is called multiple extractions.

It is more efficient to use a solvent in several smaller portions in order to recover the maximum amount of the substance from a solution.

When a solution is placed in a separating funnel and shaken with an immiscible solvent, solutes often dissolve in part in both layers.

The components are said to *partition* between the two layers or *distribute themselves* between the two layers.

When equilibrium has been established, the ratio of concentration of solute in each layer is constant for each system and this can be expressed by a value  $K_D$ .  $K_D$  is called the *partition coefficient* or *distribution coefficient*.

The distribution coefficient  $K_D$  is expressed by the following formula.

$$K_D = \frac{\text{Concentration in organic phase}}{\text{Concentration in aqueous phase}}$$

The higher the  $K_D$  value, the more effective the extraction.

For the extraction carried out,

- Calculate the concentration of ammonia in ethoxyethane.
- Calculate the concentration of ammonia in aqueous solution
- Then calculate the distribution coefficient for the extraction of ammonia from water using ethoxyethane.

$$K_D = \frac{\text{Concentration in organic phase}}{\text{Concentration in aqueous phase}}$$

### Evaluation

- Describe the procedure steps that may be used to separate iodine from a mixture of an aqueous iodine and potassium iodide using ethoxyethane.
- What are the characteristics of a good solvent used in solvent extraction? Give one example that fulfils those requirements.
- Describe any three applications of solvent extraction.

## EXPERIMENT 7.2: Freezing point depression

### Rationale

Freezing point depression refers to the lowering of the freezing point of solvents when solutes are added. The freezing point depression has many applications. For example, it prevents radiators from freezing in winter. It also allows the street ice to melt at lower temperatures, preventing the accumulation of dangerous, slippery ice. The ability to form ice cream without a freezer is another application because it is due to the freezing temperature of water lowered to below freezing point.

### Objective

Learners will be able to explain the freezing point depression of a solution.



## Required materials

### Apparatus

- Plastic coffee cup
- 500 mL beaker
- Thermometer
- Tea spoon
- Stopwatch

### Chemicals

- Tap water
- Ice
- Sodium chloride



## Procedure

1. Place about 1 cup of ice (coffee cup) in a 500 mL plastic beaker.
2. Put about 200 mL of tap water in the cup with the ice.
3. Start your stopwatch.
4. Record the temperature for every 30 seconds in the table below.
5. Stir the solution continuously to keep the temperature even throughout the cup.
6. When measuring the temperature, keep the thermometer **near the ice** in the cup, remembering that ice floats in water.
7. Record the temperature at each time point (every 30 seconds) in the table below.
8. After recording is completed (when the readings remain consistent for at least 5 consecutive readings), empty the water and ice into a sink.
9. Dry the cup completely. Repeat steps 1 and 2.
10. Pour 1.0 teaspoon (tsp) of table salt into the cup over the ice and stir.
11. Repeat steps 3 to 9.
12. Rinse the cup with tap water three times, pouring out the rinse water each time.

13. Dry the cup completely. Repeat steps 1 and 2.
14. Pour 2.0 teaspoon (tsp) of table salt into the cup over the ice and stir.
15. Repeat steps 3 to 8.

### Data recording

Time (s)	Temperature of tap water (°C)	Temperature (°C) of solution (1.0 tsp NaCl)	Temperature (°C) of solution (2.0 tsp NaCl)
30			
60			
90			
120			
150			
180			
210			
240			
270			
300			
330			
360			
390			
420			
450			
480			

### Interpretation of results and conclusion

#### Guiding questions

1. Explain how do solutes lower the freezing point of solvent?
2. By referring to the data obtained in above table, plot the graph by putting temperature on the y-axis and time on the x-axis and interpret the results.

(The 5 consecutive readings indicate the freezing point for the solution)



### Answer to guiding questions

The freezing point of a solution is less than the freezing point of the pure solvent. This means that a solution must be cooled to a *lower* temperature than the pure solvent for freezing to occur.

Freezing point depression is a colligative property of matter. Colligative properties depend on the number of particles present, not on the type of particles or their mass. So, for example, if both calcium chloride ( $\text{CaCl}_2$ ) and sodium chloride ( $\text{NaCl}$ ) completely dissolve in water, the calcium chloride would lower the freezing point more than the sodium chloride because it would produce three particles (one calcium ion and two chloride ions), while the sodium chloride would only produce two particles (one sodium and one chloride ion).

Consequently, freezing point depression is proportional to the concentration (molality) of the solution.

$$\Delta T_f \propto m,$$

$$\Delta T_f = K_f m$$

Where  $m$  is the concentration of the solute in molality units,

$K_f$  is the *molal freezing point depression constant*.

If the solute dissociates in solution, then the vapour pressure lowering is increased by the van't Hoff factor. The formula becomes:  $\Delta T = iK_f m$

The van't Hoff factor ( $i$ ) is the number of particles that the solute will dissociate upon mixing with the solvent.

For example:

We can calculate the freezing point of an aqueous solution of  $\text{NaCl}$  with a concentration of  $0.25 \text{ m}$  if the  $K_f$  of water is  $1.86 \text{ }^\circ\text{C}/\text{m}$ . In this case, it is 2 because salt dissociates into 2 ions in water.

$$\Delta T = iK_f m = (2)(1.86 \text{ }^\circ\text{C}/\text{m})(0.25 \text{ m}) = 0.93 \text{ }^\circ\text{C}.$$

So, this means the freezing point of the solution is  $0.93 \text{ }^\circ\text{C}$  lower than the normal freezing point of water ( $0 \text{ }^\circ\text{C}$ ). The new freezing point is  $0 - 0.93 = -0.93 \text{ }^\circ\text{C}$ .

## Evaluation

Ethylene glycol is common automobile antifreeze. It is a water soluble and fairly non-volatile (BP 197°C). Calculate the freezing point depression of a solution containing 651g of this substance in 2505g of water. (The molar mass of ethylene glycol is 62.07g and  $K_f$  is 1.86°C/m).

### Answer

$\Delta T = iK_f m$  Where  $\Delta T_f$  is the freezing point lowering,  $K_f$  is the freezing point constant, and  $m$  is the molality,  $i$  (van't Hoff factor) = 1 since ethylene glycol is a non-electrolyte.

$$T_f \text{ of pure water}(T^0_f) = 0.00^\circ\text{C}$$

$$T_f \text{ of solution} = ?^\circ\text{C}$$

$$K_f = 1.86^\circ\text{C}/m$$

$$\Delta T = iK_f m = (1)(1.86^\circ\text{C}/m)(m)$$

Molality  $m$  = moles of solute/kg of solvent = moles of ethylene glycol / kg of water

$$\text{Mol ethylene glycol} = 685 \text{ g} \times (1 \text{ mol} / 62.07 \text{ g}) = 11.0 \text{ mol ethylene glycol}$$

$$\text{Kg of water} = 2075 \text{ g} \times (1 \text{ kg} / 1000 \text{ g}) = 2.075 \text{ kg of water}$$

$$\text{Molality} = 11.0 \text{ mol} / 2.075 \text{ kg} = 5.30m$$

$$\text{Therefore, } \Delta T = iK_f m = (1)(1.86^\circ\text{C}/m)(5.30m) = 9.86^\circ\text{C}$$

$$T_f^0 - T_f = 9.86^\circ\text{C}$$

$$0.00 - T_f = 9.86^\circ\text{C}$$

$$T_f = 0.00 - 9.86^\circ\text{C} = -9.86^\circ\text{C}$$

## EXPERIMENT 7.3: Boiling point elevation

### Rationale

Boiling point elevation is the difference in temperature between the boiling point of the pure solvent and that of the solution. It refers to the increase of the boiling point of solvents when non-volatile solutes are added. Boiling point elevation has many applications in our daily life like cooking with salt, measuring the molar masses of solutes, sugar refining.

### Objective

Learners will be able to explain the boiling point elevation of a solution.



### Required materials

#### Apparatus

- Three pyrex beakers (1L)
- Thermometer
- Bunsen burner
- Tripod and wire gauze
- Stand and clamp
- Balance
- Glass rods (3)
- Match box

#### Chemicals

- Sodium chloride
- Distilled water

### Safety precautions

Do not let the thermometer touch the wall or the bottom of the beaker



## Procedure

1. Put in each of the three 1 L pyrex beakers 400 ml of distilled water and label them A, B, C;
2. Weigh 20g, 50g and 80g of sodium chloride and put it in beaker **A**, **B** and **C** respectively.
3. Using a glass rod, stir to dissolve the salt in each beaker. Do not use the same glass rod for all the beakers.
4. Place beaker **A** on a tripod stand fitted with wire gauze and heat the solution until boiling (until temperature is stabilised for 2-3 minutes).
5. Record temperature value (boiling point temperature).
6. Extinguish the Bunsen burner and take the solution off the tripod carefully using folded paper towels.
7. Repeat steps 4-6 for each of the 2 other solutions and record boiling temperature.
8. Repeat the experiment one time for each solution, recording boiling point temperatures
9. Extinguish the Bunsen burner.
10. Discard the salt-water in the sink.

## Data recording

	Pure water	Solution in beaker A	Solution in beaker B	Solution in beaker C
Boiling temperature (°C)				
$\Delta T$ (°C)				

## Interpretation of results and conclusion

### Guiding questions

1. Explain why when a non-volatile, non-electrolyte is added to pure solvent, the boiling point (BP) of the solvent increases.
2. Describe how the boiling point can be used to determine the molar mass of a solute.

The boiling point elevation is described by the equation:

$$\Delta T_b = i \times K_b \times m$$

Where  $\Delta T_b$  is the boiling point elevation,

$i$ , is the *van't Hoff factor*,

$K_b$  is the boiling point constant of the solvent,

$m$  is the molality of the solution.

The boiling point elevation,  $\Delta T_b$  is the difference between the boiling point of the pure solvent and the boiling point of the solution.

### Answer to guiding questions

In theory, a van't Hoff factor of a given compound is approximately equal to the number of all ions present after its dissociation in water. For example, a solution of NaCl has a van't Hoff factor of 2, a solution of  $MgCl_2$  has a van't Hoff factor of 3, and a solution of a non-dissociating substance like sugar would have a van't Hoff factor of 1.

For the performed experiment:

- a) Calculate the average boiling point found for each different solution.
- b) Plot a graph having the X-axis as *molality* and the Y-axis as the *boiling point temperature elevation from pure water (100 °C)*.
- c) Add all three data points to your graph including a data point for pure water at (0, 0).
- d) Plot a linear trend line connecting the four points.
- e) Relate the equation to the above given equation ( $\Delta T_b = i \times K_b \times m$ ) and see how the slope of the line relates to the boiling point constant and van't Hoff factor.
- f) From this, determine the boiling point constant of water.

## Evaluation

The boiling point elevation constant of carbon tetrachloride is  $4.95\text{ }^{\circ}\text{C}/\text{m}$ . The density of carbon tetrachloride is  $1.584\text{ g/mL}$ . The normal boiling point of carbon tetrachloride is  $76.8^{\circ}\text{C}$ .

- Calculate the boiling point of a solution made by mixing 17 grams of solid iodine with 165 mL carbon tetrachloride.
- Calculate the boiling point of a solution made by mixing 17 grams of solid naphthalene ( $\text{C}_{10}\text{H}_8$ ) with 165 mL carbon tetrachloride.

## EXPERIMENT 7.4:

## Illustration of the osmotic pressure

### Rationale

Osmotic pressure is the minimum pressure required to prevent the inward flow of a solution's pure solvent through a semipermeable membrane. It is also a colligative property because it depends on the number of particles in solution but not on their chemical identity.

Plants maintain their upright shape with the help of osmotic pressure. The measurement of osmotic pressure can also be used to determine molecular weights of compounds. Another important application of osmotic pressure is in the desalination and purification of sea water which involves the process of reverse osmosis.

### Objective

Learners will be able to explain the concept of osmotic pressure and its applications.



### Required materials

#### Apparatus

- Two cups
- Plastic films to cover the cups
- Ruler
- Plate

#### Chemicals

- Two eggs
- Vinegar



## Procedure

1. Place two eggs of similar size into two cups, one egg in one cup.
2. Pour enough white vinegar with about 5.0% of acetic acid into the cup until the eggs are submerged in the vinegar.
3. Cover the cup with plastic films and let it stand for about 48 hours.
4. After about 48 hours, wash off the remaining eggshell with hand in the running tap water.
5. Place both de-shelled eggs side-by-side on a plate and measure the length and width of them. Record the data in the table below.
6. Place one egg in a cup with salty water; make sure there is enough water, so the egg is fully submerged.
7. Place the other egg in pure water, make sure there is enough water, so the egg is fully submerged.
8. Cover the cups with plastic films and let them stand for about 72 hours.
9. Take the eggs out of the cup and measure the length and width of the eggs. Record the data in the table below.

### Data recording

	Before the experiment	72 hours in water	72 hours in salty water
Length of the egg (cm)			
Width of the egg (cm)			

## Interpretation of results and conclusion

### Guiding questions

1. What does mean the following terms?
  - a) Osmosis
  - b) Osmotic pressure
  - c) Semi-permeable membrane
2. What is the name given to the solutions that have the same osmotic pressures?
3. Suggest and write the formula used to calculate the osmotic pressure and use it to determine the osmotic pressure of the solution prepared by dissolving one mole of table salt into 1L of water at a temperature of 27°C.
4. A solution is prepared by dissolving 35.0 g of haemoglobin in enough water to make up 1.00 L in volume. The osmotic pressure of the solution is found to be 10.0 mmHg at 25.0 °C. Calculate the molar mass of haemoglobin.

### Answer to guiding questions

The net movement of solvent molecules through a semi-permeable membrane from a pure solvent or from a dilute solution to a more concentrated solution is called **osmosis**. The **osmotic pressure ( $\Pi$ )** of a solution is *the pressure required to stop osmosis*.

A semipermeable membrane may have small pores enough to let only the solvent molecules pass through. In other cases, a different mechanism may be responsible for the membrane's selectivity—for example, the solvent's greater "solubility" in the membrane.

The osmotic pressure of a solution is given by  $\Pi = MRT$

Where M is the molarity of solution,

R is the gas constant (0.0821 L.atm/K.mol),

T is the absolute temperature.

Usually, the solvent moves towards the region having a higher concentration.

For the experiment above, in a cup of water, the inside of the egg has a saltier its surroundings. Water is going to cross the membrane into the egg and make it swell. Thus, the egg become bigger than it was before the experiment.



But in the salt water, the surroundings of the egg are saltier than its inside. So, in this case, water is going to leave the egg and make it shrink up. Thus, it gets smaller than it was before the experiment.

### Evaluation

1. Why is it not advisable to use seawater to irrigate our crops?
2. What happens when a thirsty person drinks salt water to try to quench his/her thirst?
3. Why does salted popcorn dry your lips?
4. What happens to a cell's water when the exterior liquid is saltier than its interior?
5. What happens to water outside the cell when the interior is saltier than its surroundings?
6. Saltwater fish are hypotonic (less salty) to their surroundings while freshwater fish are hypertonic (more salty) to their surroundings. Assuming the salt cannot move, what must each fish do with its fluids to compensate for the difference in salinity between the body and the surrounding environment?

### EXPERIMENT 7.5:

### Laboratory fractional distillation of ethanol and water

#### Rationale

Fractional distillation is a separation technique that separates liquid components of a solution/mixture according to their boiling points. Fractional distillation is mostly used when the boiling points differences of components to be separated are not large. Fractional distillation is used in refining of petroleum oil, purification of ethanol and creation of alcoholic beverages, separation of many organic compounds, preparation of nitrogen from air. This experiment will help in understanding the manufacture of many useful components compounds from their mixtures.

#### Objective

Learners will be able to obtain ethanol from a mixture of ethanol and water



## Required materials

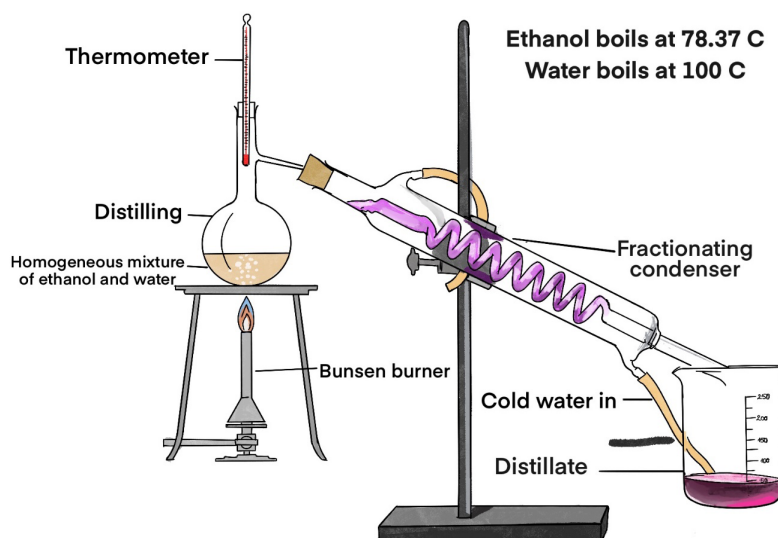
### Apparatus

- Round-bottom flask
- Conical flask
- Fractionating column
- Thermometer
- Condenser
- Retort stands and clamps
- Tripod and wire gauze
- Rubber stopper
- Bunsen burner
- Source of cold water (Tap)

### Chemicals

- Mixture of ethanol and water
- (ethanol and distilled water)

## Experimental set-up



*Figure 7.5: Fractional distillation of ethanol and water*



## Procedure

1. Fill the round-bottom flask with the ethanol-water mixture, then assemble the fractional distillation apparatus by connecting the fractionating column to the round-bottom flask.
2. Set up the rest of the apparatus as shown in Figure. 7.5
3. Heat the mixture to above the boiling point of ethanol (80-degree Celsius) using the Bunsen burner under the round-bottom flask.
4. Collect the liquid that is distilled out from 78-degree Celsius
5. Maintain a consistent temperature in the mixture until the boiling has stopped.

## Interpretation of results and conclusion

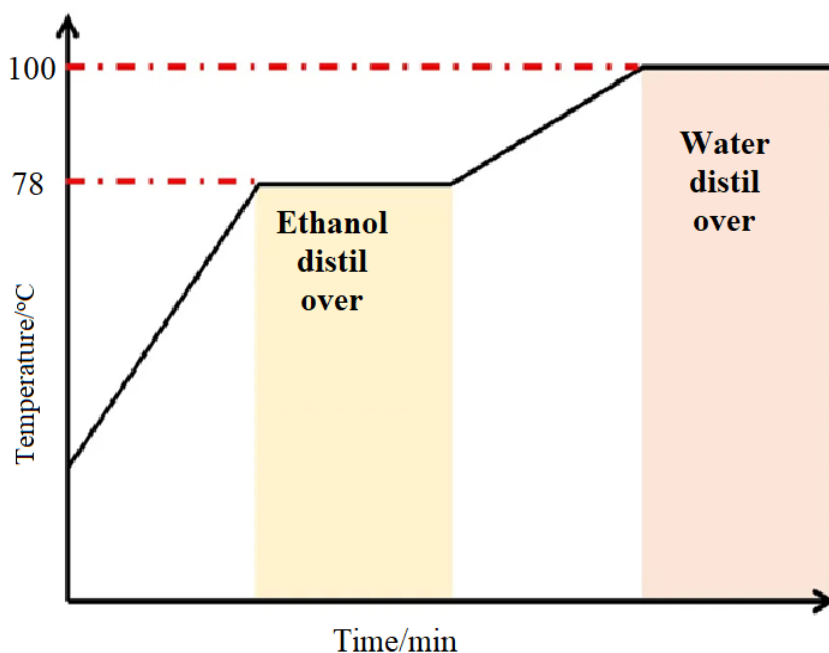
### Guiding questions

1. What is the principle that underlies fractional distillation?
2. During fractional distillation, which component is formed first?
3. Draw the labelled diagram of fractional distillation explaining how the temperature change during fractional distillation of ethanol and water

### Answer to guiding questions

During fractional distillation components in a chemical mixture are separated into different substances according to the difference in their boiling points. When the mixture is heated, the substance with the lower boiling point begins to boil and evaporate first.

The boiling points of ethanol and water are 78°C and 100°C respectively. In this experiment when the mixture is heated up to 78°C, vapor of ethanol moves up the fractionating column and repeatedly condenses inside the column. By the time the vapour reaches the top of the fractionating column, it has become almost pure ethanol. This vapour passes into the condenser where it is cooled and condensed into liquid ethanol. The thermometer shows a constant temperature of 78°C when the ethanol is being distilled. The water, which has a higher boiling point, remains in the flask until almost all the ethanol has distilled.



## Evaluation

1. Distinguish between simple from fractional distillation

**Answer:** *simple distillation is used to separate substances in mixtures with widely disparate boiling points (greater than a 100 °C difference in boiling points). whereas fractional distillation is used for mixtures containing chemicals with similar boiling points.*

## UNIT: 9

# pH OF ACIDIC AND ALKALINE SOLUTIONS

### EXPERIMENT 9.1:

### Preparation of solutions and pH measurement

#### Rationale

A solution is prepared by dissolving or diluting a known amount of solute into a specific amount of solvent. The measure of pH of solution involves the determination of how acidic or alkaline the solution is. pH is an important quantity that reflects the relative amount of free hydrogen and hydroxide ions in a solution, it can control the availability of nutrients, biological functions, microbial activity, and the behaviour of chemicals.

#### Objective

Learners will be able to prepare different solutions and measure their pH using indicators and/or a pH-metre.



#### Required materials

##### Apparatus

- 1L volumetric flask
- beakers
- Test tubes
- Droppers
- pH-metre
- Electronic balance
- Wash bottle
- Filter funnel
- Glass rod

##### Chemicals

- Distilled water
- Red and blue litmus papers
- Universal indicator
- Sodium hydroxide
- Ammonium chloride
- Sodium acetate
- Sodium chloride
- Benzoic acid



## Procedure

1. Using a balance, weigh the required mass of sodium chloride to prepare a 0.1 M solution;  
Calculate the mass (g) of NaCl that corresponds to 0.1M

*Answer: Mass of NaCl to be used is 5.85 g*

2. Put 5.85 g of sodium chloride in a 100 mL beaker containing 50 mL of distilled water;
3. Stir using a glass rod until the salt is completely dissolved;
4. Using a filter funnel transfer the solution into a 1000 mL volumetric flask;
5. Stopper the flask and gently shake the mixture;
6. Add more water until the level is about 1 mL below the graduation mark of the flask;
7. Using a dropper, fill the volumetric flask with distilled water, drop by drop, until the graduation mark is reached;
8. Stopper the volumetric flask and gently shake again.

*The solution prepared is 1000 mL of 0.1M NaCl*

9. Using indicators, determine the pH of 0.1M sodium hydroxide solution;
10. Using pH-metre, measure the pH of solution obtained in step 8;
11. Repeat the procedure to prepare a 0.1M ammonium chloride, sodium acetate solution, and Benzoic acid.
12. Record your findings in the table below.

### Data recording

Salt	NaCl	NH <sub>4</sub> Cl	CH <sub>3</sub> COONa	C <sub>6</sub> H <sub>5</sub> COOH
Concentration	0.1M	0.1M	0.1M	0.1M
pH				

## Interpretation of results and conclusion

### Guiding questions

How are the solutions prepared classified as acidic, neutral or alkaline?

### Answer to guiding questions

The pH of a solution is a **measure of hydrogen ion concentration**, which in turn is a measure of its acidity. It is a negative logarithm of the hydrogen ions concentrations.

$$\text{pH} = -\log[\text{H}^+]$$

The pH of NaCl solution equals 7. Therefore, the solution is neutral. The pH of ammonium chloride and benzoic acid are less than 7. The 2 solutions are acidic. The pH of sodium acetate is greater than 7. The solution is basic.

Pure water dissociates slightly into equal concentrations of hydrogen and hydroxide ( $\text{OH}^-$ ) ions. For a neutral solution,  $[\text{H}^+]$  is  $10^{-7}\text{M}$ , or  $\text{pH} = 7$

The pH of a solution may be estimated using pH indicators. There are more accurate methods to measure the pH of a solution such as the use of a pH metre for the substances above.

The pH ranges from 0 to 14.

A pH value less than 7 means that the solution is acidic whereas a pH value greater than 7 indicates that the solution is alkaline. The pH of 7 indicates a neutral solution.

### Evaluation

Prepare 250 mL of iron (III) chloride and measure its pH. State whether the solution is acidic or alkaline.

## EXPERIMENT 9.2: Hydrolysis of salts

### Rationale

Salt is a chemical substance formed when either part or all the ionizable hydrogen of an acid have been replaced by a metallic ion or ammonium ion. Salt hydrolysis is defined as a reaction in which the cation and the anion or both of a salt react with water to form an acidic or alkaline solution. Salt hydrolysis theory is used to understand that salt solutions can be acidic or basic or neutral.

## Objective

Learners will be able to perform and explain the hydrolysis of sodium chloride, ammonium chloride, sodium ethanoate, iron (III) chloride



## Required materials

### Apparatus

- 50 mL beakers
- Glass rod
- Indicators
- pH-meter

### Chemicals

- Sodium chloride
- Ammonium chloride
- Sodium ethanoate
- Iron (III) chloride



## Procedure

1. To a spatula endful of sodium chloride in a beaker, add 20 mL of distilled water;
2. Stir using a glass rod;
3. Add more water and stir until the salt is completely dissolved and measure its pH.
4. Repeat the procedure using ammonium chloride, sodium ethanoate, iron (III) chloride.
5. Record your observations in the table below.

## Data recording

Salt	NaCl	NH <sub>4</sub> Cl	CH <sub>3</sub> COONa	FeCl <sub>3</sub>
pH of the solution				

## Interpretation of results and conclusion

### Guiding questions

Compare the pH of different salts (NaCl, NH<sub>4</sub>Cl, CH<sub>3</sub>COONa and FeCl<sub>3</sub>) and explain.



### Answer to guiding questions

Salt hydrolysis is a reaction in which one of the ions from a salt reacts with water, forming either an acidic or basic solution.

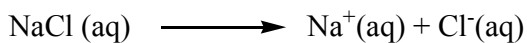
#### ❖ *Salts formed from strong acid and strong base*

These salts dissolve in water to give neutral solutions.

Examples: NaCl, Na<sub>2</sub>SO<sub>4</sub>, KNO<sub>3</sub>

Aqueous solutions of the salts consist of ions with very little affinity for hydrogen ions or hydroxide ions in water. The ions of such salts are only hydrated (i.e. surrounded by water molecules).

For example: Sodium chloride dissociates in water to give the anion Cl<sup>-</sup>



H<sub>3</sub>O<sup>+</sup> and Cl<sup>-</sup> constitute an acid-base conjugate pair.



Since hydrochloric acid is a strong acid, Cl<sup>-</sup> is very weak base. Cl<sup>-</sup> is unable to accept a proton (H<sup>+</sup>) from an acid, particularly water. That is why Cl<sup>-</sup> does not hydrolyze.

Na<sup>+</sup> and NaOH are also acid-base conjugate pairs. Since sodium hydroxide is a strong base, its conjugate acid is weak. Na<sup>+</sup> can't be hydrolysed.

The pH of sodium chloride solution remains 7 because no ion from the salt is hydrolysed to release H<sup>+</sup> or HO<sup>-</sup> ions.

#### ❖ *Salts formed from weak acid and strong base*

These are salts which undergo cation hydrolysis to form alkaline solutions.

**Examples:** CH<sub>3</sub>COONa, Na<sub>2</sub>CO<sub>3</sub>, C<sub>6</sub>H<sub>5</sub>COONa, K<sub>3</sub>PO<sub>4</sub>

Consider sodium ethanoate, CH<sub>3</sub>COONa compound which ionises completely in water as follows: CH<sub>3</sub>COONa (aq) → CH<sub>3</sub>COO<sup>-</sup>(aq) + Na<sup>+</sup>(aq)

Ethanoate ions disturb the ionic equilibrium of water where they accept hydrogen ions from water. CH<sub>3</sub>COO<sup>-</sup>(aq) + H<sub>2</sub>O(l) → CH<sub>3</sub>COOH(aq) + OH<sup>-</sup>(aq)

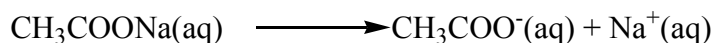
This makes the resultant solution alkaline due to excess hydroxide ions present. Such a solution has a pH value greater than 7.

#### ❖ *Salts formed from strong acid and weak base*

These are salts which undergo cation hydrolysis to form acidic solutions.

**Examples:**  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{NO}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{C}_6\text{H}_5\text{NH}_3\text{Cl}$

Consider ammonium chloride,  $\text{NH}_4\text{Cl}$  compound which ionises completely in water as follows:



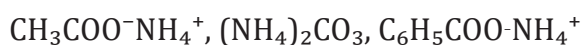
Ammonium ions disturb the ionic equilibrium of water by accepting hydroxyl ions from water. This makes the resultant solution acidic due to excess hydrogen ions from water thus a pH value less than 7.



### ❖ *Salts formed from weak acid and weak base*

These are salts which undergo both cation and anion hydrolysis when dissolved in water.

Examples of such salts include:



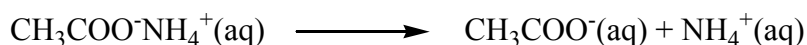
The nature of the resultant solution depends on the relative strength of the weak base and the weak acid. It may finally be acidic, alkaline or neutral.

If  $K_b$  of the anion is greater than  $K_a$  for the cation, the solution formed is alkaline because the anion is greatly hydrolysed to produce more hydroxide ions in solution.

When  $K_b$  is less than  $K_a$  of the cation, the resultant solution is acidic because the cation will be hydrolysed to produce hydrogen ions in solution.

And if  $K_b$  is approximately equal to  $K_a$ , the resultant solution is neutral.

Ammonium acetate as an example ionises in water as follows;



Since the acid and the base are both weak, their conjugate base,  $\text{CH}_3\text{COO}^-$  and conjugate acid,  $\text{NH}_4^+$  ions are relatively strong. They accept  $\text{H}^+$  and  $\text{OH}^-$  ions respectively from water and undergo considerable hydrolysis. Therefore, the resultant solution of the above salt hydrolysis is neutral.

### Evaluation

1. Write the chemical equation of the hydrolysis of:
  - a) Aluminium chloride
  - b) Potassium benzoate

- c) Potassium sulphate
  - d) Barium carbonate
  - e) Sodium nitrite
2. State in each salt above if the solution is acidic, neutral or alkaline.

### EXPERIMENT 9.3: Preparation and properties of a buffer solution

#### Rationale

Buffer solution is a solution that resists a pH change when a small amount of a base or acid is added. A buffer solution can be acidic or basic. Buffer solutions are used to control the pH of various solutions in relation to their properties, mode of preparation and their uses. The application of buffer solutions ranges from natural systems, biological and agricultural to industrial processes. This experiment shows on how to prepare buffer solutions and their applications.

#### Objective

Learners will be able to prepare and demonstrate the properties of / buffer solution



#### Required materials

##### Apparatus

- 1L volumetric flask
- 100mL beakers
- Test tubes
- Test tubes rack
- pH-metre
- Dropper
- Electronic balance

##### Chemicals

- Sodium ethanoate
- Ethanoic acid
- Distilled water
- Red and blue litmus papers



## Procedure

1. To 100 mL of 0.01M ethanoic acid, add 100 mL of 0.01M sodium ethanoate solution.
2. Using red litmus paper and blue litmus paper, determine the nature of the solution obtained in step 1. What do you observe?

**Answer:** Blue litmus paper turns red while no colour change for red litmus paper, so the solution is acidic.

3. Using a pH-metre, measure the pH of the solution obtained in step 1.

**Answer :** pH is around 4.74

4. Compare the results obtained in steps 2 and 3 to know the nature of the solution.

**Answer:** The pH is under 7. The solution is acidic

5. To the solution obtained in step 1, add one drop of 0.01 M sodium hydroxide solution.

6. Using the pH-metre, what is the pH of this solution after adding sodium hydroxide?

**Answer:** pH slightly lower than 4.74

7. To solution obtained in step 1, add one drop of 0.01 M hydrochloric acid.

8. Using the pH-metre, what is the pH of this solution after adding hydrochloric acid?

**Answer:** pH slightly greater than 4.74

## Interpretation of results and conclusion

### Guiding questions

1. What is buffer solution meant ?
2. Discuss the two types of buffer solutions. Give two examples for each.
3. Using chemical equation, explain how a mixture of acetic acid and ethanoate ion resist change in pH when:
  - i. Hydrochloric acid is added
  - ii. Sodium hydroxide is added

iii. It is ten times diluted.

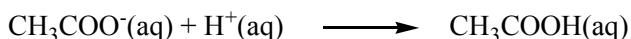
### Answer to guiding questions:

Buffer solution is a solution that resists a pH change when a small amount of a base or acid is added. We may have two types of such solutions: acidic and basic.

➤ **An acidic buffer:** Buffer solution consisting of a solution of a weak acid and its salt with a strong base. Example:  $\text{CH}_3\text{COOH}/\text{CH}_3\text{COONa}$

Such a solution contains few hydrogen ions from the acid and a large proportion of ethanoate ions from the salt.

When a small amount of an acid is added, the added hydrogen ions react with ethanoate ions to form ethanoic acid. This prevents an increase in the concentration of hydrogen ions in the solution hence pH does not change or changes very slightly.



When a small amount of a base is added, the added hydroxyl ions react with ethanoic acid to form water. This prevents an increase in the concentration of hydroxyl ions hence pH does not change or changes very slightly.



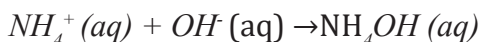
In fact, for an acidic buffer solution,

If,  $\text{pH} = \text{pK}_a$

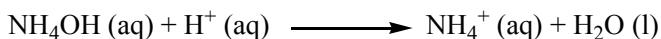
➤ **A basic or alkaline buffer:** Buffer solution consisting of a solution of a weak base and its salt of strong acid. Example:  $\text{NH}_4\text{OH}/\text{NH}_4\text{Cl}$ .

Such a solution contains a few hydroxyl ions and a large proportion of ammonium ions from the salt.

When a small amount of a base is added, the added hydroxide ions react with ammonium ions to form unionised aqueous ammonia. This prevents any increase in the concentration of hydroxide ions in the solution hence no change in pH.



Similarly, when a small amount of an acid is added, the added hydrogen ions react with un ionised aqueous ammonia to form water. This prevents an increase in the concentration of hydrogen ions thus keeping pH constant.



In fact, for a basic buffer solution,

$$\text{pH} = \text{pK}_w - \text{pK}_b + \log \left( \frac{[\text{Salt}]}{[\text{Base}]}\right)$$

In general, for a buffer solution,

- Its pH value does not change on dilution.
- Its pH does not change or changes very little when a small amount of strong acid base is added to it.

### Evaluation

1. Explain how a buffer works to resist large pH changes. Provide enough information to indicate your understanding on buffers solution.
2. Which of the following solutions can be classified as buffer systems?
  - a)  $\text{KH}_2\text{PO}_4/\text{H}_3\text{PO}_4$
  - b)  $\text{NaClO}_4/\text{HClO}_4$
  - c)  $\text{KF}/\text{HF}$

Explain your answer.

## EXPERIMENT 10.1: Effectiveness of indicators in titrations

## Rationale

Food processing, chemical manufacturing, and pharmaceutical manufacturing are the three businesses in the manufacturing sector that heavily rely on titration methods.

Typically, acid-base titrations are performed to analyse the purity of medicines because many medicines use either acids or bases as ingredients or reagents. Since a noticeable pH change occurs near the equivalence point of acid-base titrations, an indicator is used to signal the end of a titration. When selecting an indicator for acid-base titrations, check its effectiveness by choosing an indicator whose pH range falls within the pH change of the reaction.

## Objective

Learners will be able to explain how the effectiveness of indicators is related to the pH changes during titrations



## Required materials

## Apparatus

- 50 mL Burette
- 25 mL pipette
- Retort stand
- Conical flasks
- Filter funnel
- Droppers
- Suck bulb (or a pipette filler)

## Chemicals

- 0.1M sodium hydroxide
- 0.1M hydrochloric acid
- 0.1M ethanoic acid
- 0.1M ammonia
- Distilled water
- Phenolphthalein indicator
- Bromothymol blue
- Alizarin yellow
- Methyl orange
- Thymol blue



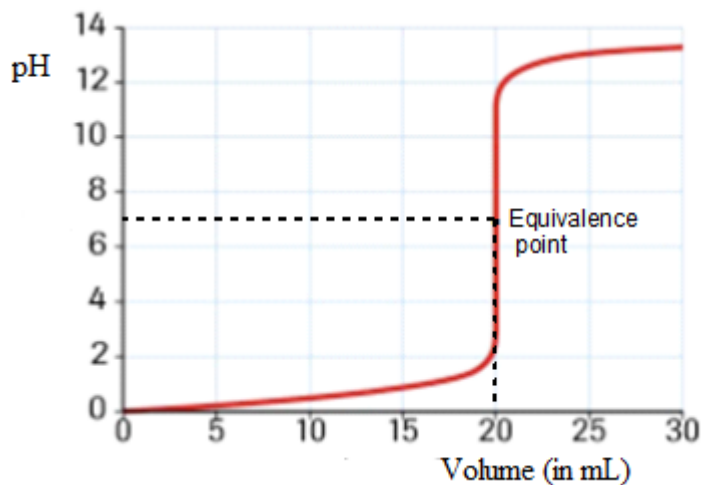
## Procedure

1. Wash a 250 mL conical flask with tap water and then rinse with distilled water;
2. Using a pipette equipped with a suck bulb (or a pipette filler), pipette 20 mL of 0.1M hydrochloric acid;
3. Transfer the acid in the clean conical flask;
4. Add 2-3 drops of phenolphthalein to acid;
5. Wash a burette with tap water, and then with distilled water and rinse three times with small amounts of sodium hydroxide (the titrant);
6. Fix the burette to the retort stand;
7. Using a funnel, fill the burette with a 0.1M sodium hydroxide solution;
8. Run enough solution of NaOH through the tip of the burette to bring the level down the zero line
9. If the level falls below the zero line, record the initial volume ( $V_i$ ) to 0.1 mL. Otherwise record 0.0 mL for the initial volume (bottom of the meniscus);
10. Put the conical flask containing the hydrochloric acid (HCl) under the burette;
11. Add slowly the base from the burette to the acid in the conical flask. The flask is continuously agitated to mix the reactants;
12. Continue this process until the indicator just changes colour;
13. Continue the titration until a large excess of the base has been added;
14. Calculate the pH of the mixture after addition of each mL of the base. Or monitor the reaction using an electronic pH-meter;
15. Repeat the process for the titration of acetic acid with sodium hydroxide
16. Repeat the process for the titration of ammonia with hydrochloric acid





For example, for the titration of hydrochloric acid with sodium hydroxide, the titration curve should look as follows.



**Figure.10.1:** Titration curve when a strong base is added to a strong acid

Therefore, bromothymol blue, phenol red, phenolphthalein whose pH range is 6.0-7.6, 6.8-8.4, 8.2-10.0 respectively can fit for a strong acid-strong base titration whose pH at the equivalence point is 7.0.

Analyse the titration curve you have plotted and suggest appropriate indicators.

### Evaluation

Perform the titration of 20 ml of 0.05M sodium carbonate with 20 ml of 0.1M hydrochloric acid.

- Plot the titration curve for this reaction.
- What indicator is the most suitable for this titration? Give your reason

# UNIT: 11

## SOLUBILITY AND SOLUBILITY PRODUCT CONSTANT ( $K_{sp}$ ) FOR SPARINGLY SOLUBLE SALTS

### EXPERIMENT 11.1:

Simple determination of the solubility product of a sparingly soluble compound (calcium hydroxide)

#### Rationale

Solubility is a term that refers to the maximum amount of solid (either in moles or grams) that dissolves in a solvent at equilibrium producing ions. It can also be an equilibrium between solid and ions in a solution. A compound is referred to as sparingly soluble depending on how much of it dissolves in a solvent. When a concentration of 0.1 g or more of the solute dissolves in a 100 ml of the solvent, then the compound is said to be sparingly soluble.

The solubility product is the equilibrium constant expressed in terms of concentrations of the ions produced from a sparingly soluble solid in contact with a saturated solution. The solubility product is used in volumetric analysis, production of soap (saponification), in inorganic qualitative analysis, buffering solutions, purification and precipitation of salts, manufacturing of baking soda, water treatment.

#### Objective

Learners will be able to determine the solubility product of calcium hydroxide.



#### Required materials

##### Apparatus

- Electronic balance
- Stand and clamp
- Conical flask
- Beaker

##### Chemicals

- Calcium hydroxide(s)
- Hydrochloric acid (1M)
- Phenolphthalein indicator

- Volumetric pipette
- Burette
- Funnels
- Filter paper
- Distilled water

## Experimental set-up

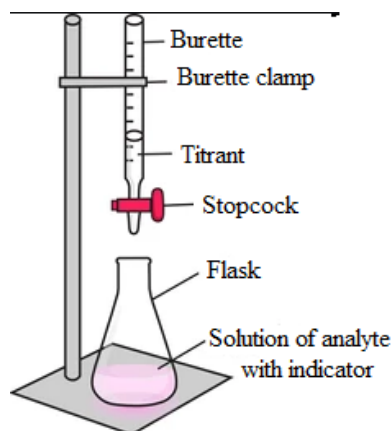


Figure 11.1: Titration of a saturated  $\text{Ca}(\text{OH})_2$  solution with hydrochloric acid



## Procedure

### Part 1: Preparation of dilute hydrochloric acid

1. Measure exactly 2.00 mL of a 1.00M HCl solution and transfer it in a 50 mL volumetric flask using your 10 mL graduated cylinder.
2. Fill to the line with distilled water and mix well. This diluted solution is to be used in all titrations.
3. Take a small portion (approximately 5 mL) of the diluted HCl solution and rinse the insides of the burette with it. Fill the burette with the diluted HCl solution.

### Part 2: Determination of $K_{sp}$ for $\text{Ca}(\text{OH})_2$ at room temperature

1. Prepare a saturated solution of calcium hydroxide by adding 0.10-0.30 g of  $\text{Ca}(\text{OH})_2$  to 30 mL of distilled water in a 50 mL beaker. You should see some undissolved calcium hydroxide at the bottom of the beaker. If not, add a little more calcium hydroxide.

- Record the temperature of the solution.
- Filter the saturated  $\text{Ca}(\text{OH})_2$  solution through a piece of filter paper in a funnel into a 100 mL beaker.
- Using a 10 mL graduated cylinder, measure out 5.00 mL of the filtered solution with a plastic pipette. Pour the 5.0 mL of solution into a clean 125 mL conical flask. Add 10 mL of distilled water to the solution and 3 drops of bromothymol blue.
- Record the initial volume of HCl in the burette to the nearest 0.01 mL.
- Shake the solution gently while titrating with the diluted HCl until the colour of the solution changes from blue to greenish yellow.
- Record the final volume of HCl solution added to the nearest 0.01 mL.
- Replicate the titration 3 times.

### Data recording

Concentration of stock HCl solution \_\_\_\_\_

Temperature of saturated  $\text{Ca}(\text{OH})_2$  Solution \_\_\_\_\_

In the space below record your burette readings.

	Trial 1	Trial 2	Trial 3
Final burette reading			
Initial burette reading			
Volume of HCl solution used			

### Interpretation of results and conclusion

#### Guiding questions

- Find average volume of HCl solution used in the titration
- Calculate the number of moles of HCl reacted with  $\text{Ca}(\text{OH})_2$
- Calculate the concentration of calcium and hydroxide ions.
- Write the expression relating the solubility product constant and the concentration of its ions.

5. Calculate the value of the solubility product constant (K<sub>sp</sub>).

### Answer to guiding questions

#### Concentration of diluted HCl

The following formula can be used to determine the concentration of the dilute HCl used in the titrations. Set up the equation to solve for M<sub>2</sub>, the concentration of the diluted HCl.

$$M_1 V_1 = M_2 V_2$$

M<sub>1</sub> = concentration of stock solution, M<sub>2</sub> = concentration of diluted solution

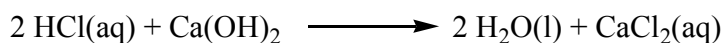
V<sub>1</sub> = volume of stock solution used, V<sub>2</sub> = total volume of diluted solution

#### Moles HCl used in the titration

Multiply the volume of diluted HCl solution added (in litres) during the titration by the concentration of diluted HCl solution.

#### Moles Ca(OH)<sub>2</sub> in the saturated solution

For every mole of Ca(OH)<sub>2</sub> used, you need 2 moles of HCl, so divide the moles of HCl used in the titration by 2 to find the moles of Ca(OH)<sub>2</sub> in the titrated solutions.



#### Concentration of Ca(OH)<sub>2</sub> in the saturated solution

Calculate the concentration of Ca(OH)<sub>2</sub> in the saturated solution by dividing the number of moles of Ca(OH)<sub>2</sub> by the volume of saturated solution used in litres.

#### Concentration of Ca<sup>2+</sup> and OH<sup>-</sup> in the saturated solution

Calculate the concentrations of Ca<sup>2+</sup> and OH<sup>-</sup> in the saturated solution by using the following relationships:

$$[\text{Ca}^{2+}] = [\text{Ca(OH)}_2]$$

$$[\text{OH}^-] = 2 \times [\text{Ca(OH)}_2]$$

Values of K<sub>sp</sub> for Ca(OH)<sub>2</sub>

Enter the concentrations for Ca<sup>2+</sup> and OH<sup>-</sup> into the K<sub>sp</sub> expression for Ca(OH)<sub>2</sub> and calculate a value for K<sub>sp</sub> at room temperature.

$$K_{sp} = [\text{Ca}^{2+}] [\text{OH}^-]^2$$

## Evaluation

1. Dissolve two spatulas full of copper (II) sulphate in 100 mL of distilled water. State what is observed.
2. Without changing the volume of water add extra spatula full of copper (II) sulphate crystals stepwise, state what is observed.
3. Keep on stirring until there is no further change and write down your observations.
4. List some areas where solubility products can be used in modern society.

**EXPERIMENT 12.1:** Working of the galvanic cell**Rationale**

Galvanic cells also known as voltaic cells are types of electrochemical cells. These are very important to our lives because they provide the foundation of generating electric current spontaneously from a chemical reaction. Galvanic cells harness the electrical energy available from the electron transfer in a redox reaction to perform useful electrical work. The principles of these cells are used to make electrical batteries. Batteries have many uses such as in torches, electrical appliances like cell phones, digital cameras, hearing aids, digital watches or military applications. This experiment aims on generation of electricity through electrochemical cells and their applications in different fields.

**Objective**

Learners will be able to explain the working principle of an electrochemical cell

**Required materials****Apparatus**

- One (1) voltmeter
- Connecting wires
- Crocodile clips
- Plastic tube (or a U-tube)
- Cotton wool

**Chemicals**

- Zinc sulphate solution (1M)
- 2 M potassium nitrate
- Zinc rod
- Copper rod
- Copper sulphate solution (1M)



## Experimental set-up

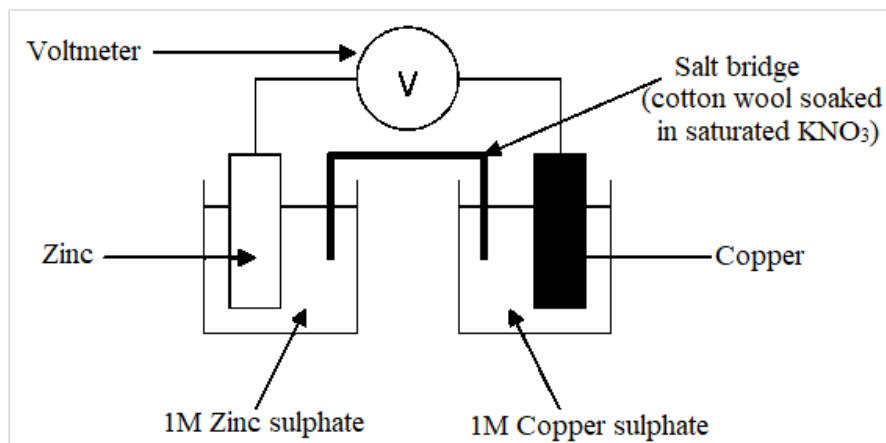


Figure 12.1: Working principle of Cu-Zn electrochemical cell



## Procedure

1. Set up the apparatus as displayed in figure 12.1.
2. Connect the voltmeter to the two electrodes.
3. Read the voltmeter and write the value of volts
4. With the voltmeter connected correctly, remove the salt bridge. What do you observe.

**Answer:** The voltmeter marks 0 volt

5. What is the role of the salt bridge in the electrochemical cell?

**Answer:** A salt bridge helps in maintaining the charge balance in the two half cells. A salt bridge prevents mechanical flow of solution, but it provides a free path for the migration of ions, to maintain an electric current through the electrolyte solution. It prevents the accumulation of charges. A salt bridge minimises or eliminates the liquid junction potential.

## Data recording

	With the salt bridge	Without the salt bridge
Voltmeter		0 v

## Interpretation of results and conclusion

### Guiding questions

1. What are the main parts that constitute an electrochemical cell?
2. The volts measured by the voltmeter shows that the electric current is being produced. Explain the source of this current.
3. Why does the voltmeter display 0.00V when the salt bridge is removed?
4. By using ionic equations explain what happens at electrodes.
5. What is the direction of the electrons?
6. What is the direction of electricity?

### Answer to guiding questions

The following reactions happen in the half cells.

In Zn/ZnSO<sub>4</sub> half-cell, oxidation reaction occurs:  $\text{Zn(s)} \rightarrow \text{Zn}^{2+}(\text{aq}) + 2\text{e}^{-}$

In Cu/CuSO<sub>4</sub> half-cell, reduction reaction occurs:  $\text{Cu}^{2+}(\text{aq}) + 2\text{e}^{-} \rightarrow \text{Cu(s)}$   
The overall cell reaction is  $\text{Zn(s)} + \text{Cu}^{2+}(\text{aq}) \rightarrow \text{Zn}^{2+}(\text{aq}) + \text{Cu(s)}$

The Daniell cell can be conventionally noted as:  $\text{Zn/Zn}^{2+} \parallel \text{Cu}^{2+}/\text{Cu}$

In a Daniell cell electrons flow from zinc electrode to copper electrode through an external circuit, while metal ions move from one half cell to the other through the salt bridge.

Here current flows from copper electrode to zinc electrode that is cathode to anode via an external circuit.

### Evaluation

1. Explain how electrolytic cell/ Daniel cell works

**EXPERIMENT 13.1: Illustration of how different reactions have different rates****Rationale**

The speed at which the products are formed from the reactants in a chemical reaction is known as rate of reaction. By finding out how fast products are made and what causes reactions to slow down, we can develop methods to improve production. This information is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning items.

**Objective**

Learners will be able to explain how fast or slow a chemical reaction is.

**Required materials****Apparatus**

- Bunsen burner
- Crucible
- Test tubes
- Test tube rack
- Test tube holder
- Dropper
- Beaker

**Chemicals**

- Ethanol
- Iron nails
- Lead nitrate solution
- Potassium iodide solution

**Safety precautions**

Lead and its compounds are toxic. Wear protective gloves when you handle them.



## Procedure

1. Burn 5 mL of ethanol in a crucible and record the time taken for this amount of ethanol to be completely burned.
2. Put 5 mL of lead (II) nitrate aqueous solution in a test tube and add three drops of aqueous potassium iodide solution, then record the time taken for the yellow precipitate to be formed.
3. Put 2 or 3 iron nails in an open beaker containing water and predict what will happen to these nails after three weeks or one month

## Interpretation of results and conclusion

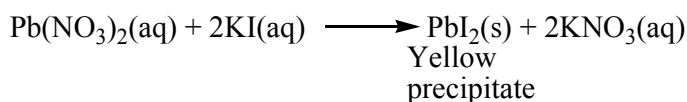
### Guiding questions

1. Suggest the name of a reaction that takes place in each of the above activities.
2. In the above three activities, indicate the slowest and fastest reaction.
3. Using books or the internet, describe your understanding of the rate of chemical reaction.

### Answer to guiding questions

During a chemical reaction, reactants are converted into products by breaking bonds in reactants and forming new bonds in products.

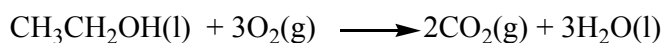
Some chemical reactions can be slow or fast. For example, the chemical reaction between a solution of lead (II) nitrate and potassium iodide is a fast reaction:



The precipitation of silver chloride is also fast, it occurs instantaneously by mixing aqueous solutions of silver nitrate and sodium chloride.

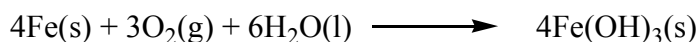
Likewise, the combustion of ethanol is fast; it takes less than 1 second.

The products of the chemical reaction are carbon dioxide and water:



However, the rusting of iron is a slow process that takes place over many days, even over the years. Similarly, the formation of diamond and other minerals in the earth's crust takes millions of years.

The chemical equation of the rusting process is



If we consider how quickly or slowly each of the chemical reactions above proceeds, we can say that the rate of the rusting of iron is much smaller than the rate of combustion of ethanol or that of the precipitation of lead iodide.

### Evaluation

Classify the following reactions as fast or slow.

- Reaction of potassium with water
- Digestion
- Decomposition of nitroglycerine
- Formation of stalactites and stalagmites
- Formation of coal
- Alcoholic fermentation
- Reaction of potassium iodide with silver nitrate

## EXPERIMENT 13.2:

### Factors that affect the rates of reactions

#### 13.2.1. Effect of concentration on the reaction rate

##### Rationale

Reaction rate in chemical kinetics is the measure of how fast or slow a reaction is. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning items. The understanding of the effect of concentration on the rate of reaction is crucial to predict the speed of that reaction.

##### Objective

Learners will be able to explain the effect of the concentration on the speed of the reaction.



## Required materials

### Apparatus

- Conical flask
- Measuring cylinder
- Stopwatch
- Piece of paper marked X
- Graph paper

### Chemicals

- 2M hydrochloric acid
- Sodium thiosulphate solution of concentrations (0.06M, 0.12M, 0.18M, 0.24M and 0.30M)

## Experimental set-up

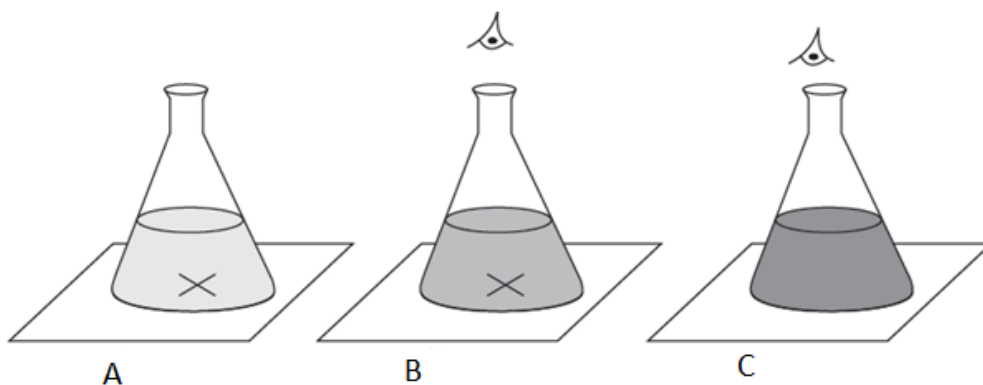


Figure 13.1: Effect of the concentration on the rate of the reaction

## Safety precautions

Wear your eye and mask protection.

Sulphur dioxide is toxic and corrosive. Do not inhale fumes of sulphur dioxide.



## Procedure

1. Put 50 mL of 0.06M sodium thiosulphate solution into a conical flask.
2. Measure 5 mL of 2M hydrochloric acid into the measuring cylinder.

3. Add the acid to the conical flask and immediately start the stopwatch. Swirl the flask to mix the solutions and place it on a piece of paper marked with a cross.
4. Look down at the cross from above as it is shown in the figure 13.1. When the cross disappears, stop the watch and note the time taken. Record your results in the table shown below.
5. Repeat the procedure using a 0.12M, 0.18M, 0.24M and 0.30M sodium thiosulphate solution.

### Data recording

Experiment	1	2	3	4	5
Concentration of sodium thiosulphate in mol/dm <sup>3</sup>	0.06M	0.12M	0.18M	0.24M	0.30M
Time taken for the cross to disappear in seconds					
$\frac{1}{t}$ (s <sup>-1</sup> )					

### Interpretation of results and conclusion

#### Guiding questions

1. Explain why the cross is disappearing when sodium thiosulphate reacts with hydrochloric acid ?
2. Write a well-balanced chemical equation for the reaction taking place in question 3. For each set of results, calculate the value of 1/t and fill in the table. (This value can be taken as a measure of the rate of reaction).
3. Plot a graph of 1/t on the vertical (y) axis and concentration of the sodium thiosulphate on the horizontal (x) axis
4. Comment on the shape of the graph.
5. What is the effect of raising the concentration on the reaction rate?

#### Answer to guiding questions:

When sodium thiosulphate and dilute hydrochloric acid are mixed, a yellow precipitate of sulphur is formed. The higher the concentration of sodium thiosulphate solution, the less the time taken for the cross to disappear.

The chemical reaction that happens can be represented as follows.



From the above experiment the rate of reaction is directly proportional to the concentration of reacting species. By increasing the concentration, the number of reacting particles is increased and hence the effective collision is increased which results in the increase of reaction rate.

### Evaluation

1. What is the relationship between concentration and rate of reaction?
2. Explain how you can speed up the reaction represented by the chemical equation below:  $\text{NaOH}(\text{aq}) + \text{HCl}(\text{aq}) \longrightarrow \text{NaCl}(\text{aq}) + \text{H}_2\text{O}(\text{l})$

### 13.2.2. Effect of particles size on the reaction rate

#### Rationale

Reaction rate in chemical reaction is the measure of how fast or slow a reaction is. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning items. The understanding of the effect of changing the size/surface area of reacting particles on the rate of reaction is crucial to predict the speed of that reaction.

#### Objective

Learners will be able to explain the effect of particle size on the rate of the reaction rate.



#### Required materials

##### Apparatus

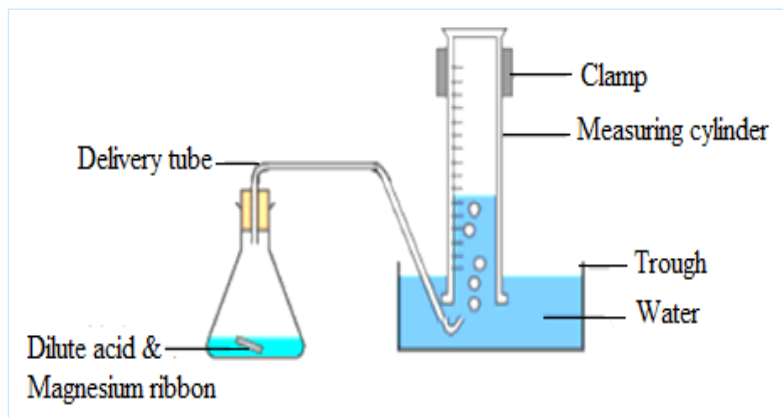
- Measuring cylinder (100 mL)
- Retort stand
- Weighing balance
- Glass trough
- Stopwatch
- Sand paper
- Delivery tube

##### Chemicals

- Magnesium ribbon
- Hydrochloric acid (1M)
- Distilled water



## Experimental set-up



**Figure 13.2:** *Effect of the particles size on the rate of the reaction*



## Procedure

1. Clean the magnesium ribbon using a sandpaper to remove any oxides that may be coating its surface. This will reduce reaction errors related to impurities.
2. Cut three equal sizes (10 cm each) of magnesium from the freshly cleaned magnesium ribbon, weigh each of them using a digital weighing balance and record their weights. Are they the same?
3. Wrap the magnesium pieces immediately in an aluminium foil to prevent them from being re-oxidised.
4. Fold the first magnesium ribbon (10 cm) and keep it for the next procedure.
5. Take the second magnesium ribbon and cut it into smaller pieces of 1 cm.
6. Take the third magnesium ribbon and cut it into smaller pieces of 0.1 cm.
7. Measure 20 mL of 1M hydrochloric acid using a clean dry measuring cylinder and pour into each of three clean 100 mL conical flasks and label them A, B and C.
8. Successively put the folded magnesium ribbon 10 cm in A, the fine pieces of magnesium 1 cm into conical flasks B and 0.1 cm magnesium ribbon in C. Immediately start the stopwatch.

What do you observe?

**Answer:** A colourless gas evolved and the speed of the reaction in A is slow compared to the reaction speed in B and in C. The reaction in C is more faster than others.

9. Monitor the reaction progress closely and stop your stopwatch when the magnesium ribbon completely dissolves in the acid or reaction comes to stop. In each case, record the reaction duration in seconds in the table below.

### Data recording

	A	B	C
Time taken for the complete reaction (s)			

### Interpretation of results and conclusion

#### Guiding questions

1. What is the effect of particles size on the reaction rate?
2. Write down a word and symbolic equation for the reaction taking place.

#### Answer to guiding questions:

The reaction involving magnesium in very fine pieces (0.1 cm) takes less time to come to an end than the reactions involving magnesium in larger pieces (1 and 10 cm). Also, effervescence was more rapid with very fine pieces.

The chemical reaction that occurs is represented by the following chemical equation:  $\text{Mg(s)} + 2\text{HCl(aq)} \longrightarrow \text{MgCl}_2\text{(aq)} + \text{H}_2\text{(g)}$

Decreasing the size of the particles, which make up a given weight, will increase the number of particles represented by the same weight. Smaller particles size results in an increase in the speed of reaction because the surface area of the interacting reactants has been increased.

### Evaluation

Suggest at least two examples in daily life illustrating the effect of particles size on the reaction speed.

### 13.2.3. Effect of temperature on the reaction rate

#### Rationale

Reaction rate is the measure of how fast or slow a reaction is. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning items etc. The understanding of the effect of changing the temperature on the rate of reaction is important in order to increase or decrease the speed of that reaction.

#### Objective

Learners will be able to explain the effect of temperature on the speed of the reaction.



#### Required materials

##### Apparatus

- 250 mL conical flask
- Thermometer
- Stop clock
- Stand and clamp
- 50 mL measuring cylinder
- Wire gauze
- Graph paper
- Piece of white paper
- Test tubes
- 10 mL measuring cylinder
- Beaker (1L)
- 50 mL conical flask
- Tripod stand and clamp

##### Chemicals

- Sodium thiosulphate (0.25M)
- Hydrochloric acid (2M)
- Distilled water

## Experimental set-up

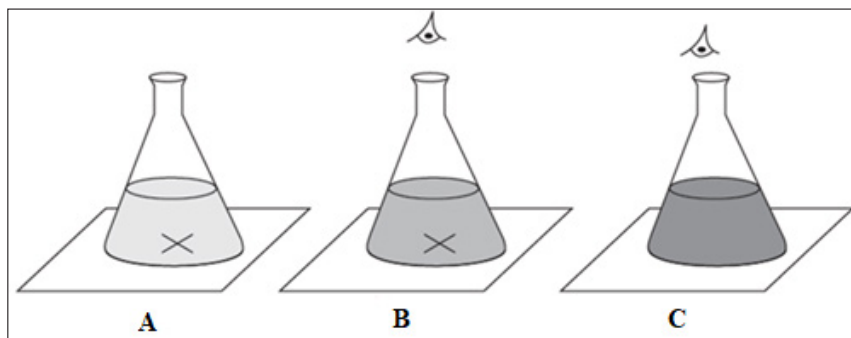


Figure 13.3: Effect of the temperature on the reaction rate

### Safety precautions

Wear your eye and mask protection.

Sulphur dioxide is toxic and corrosive. Do not inhale fumes of sulphur dioxide.



### Procedure

1. Using a 50 mL measuring cylinder, put into a conical flask 10 mL of the 0.25M sodium thiosulphate solution and add 40 mL of distilled water.
2. Using a 10 mL measuring cylinder, measure 5 mL of the 2M hydrochloric acid and put it into a test tube.
3. Prepare a water bath by half-filling a 1 L beaker with water and place the beaker over a tripod stand and wire gauze.
4. Clamp the conical flask in the water bath and place the test tube in the water bath.
5. Place the thermometer in the conical flask and very gently heat the water bath until the contents of the flask reach the temperature of 30°C.
6. Remove the conical flask and the test tube from the water bath.
7. Place the conical flask on the paper marked with a cross and immediately, add the acid from the test tube as you start the stopwatch. Gently stir the mixture with the thermometer.

*What do you observe?*

**Answer:** A yellow solid forms in the solution.

8. Observe the cross from above through the solution and stop the stopwatch as soon as the cross disappears from your view.
9. Record the time taken for the cross to be hidden and record the final temperature of the mixture.
10. Repeat the experiment for each of the temperatures of 25°C, 35°C, 40°C, 45°C, 50°C and record all your results.

### Data recording

Experiment	1	2	3	4	5	6
Temperature (°C)	25	30	35	40	45	50
Time taken for the cross to disappear (s)						
$\frac{1}{t}$ (s <sup>-1</sup> )						

### Interpretation of results and conclusion

#### Guiding questions

1. For each set of results from the reaction between sodium thiosulphate and hydrochloric acid, calculate the value of  $\frac{1}{t}$ . (This value can be taken as a measure of the rate of reaction for this experiment).
2. Plot a graph of  $\frac{1}{t}$  on the vertical (y) axis and average temperature on the horizontal (x) axis.
3. Comment on the shape of the graph.
4. What is the effect of increasing temperature on the reaction time?

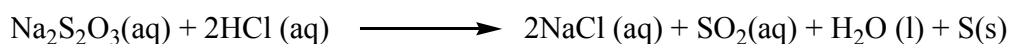
#### Answer to guiding questions

When sodium thiosulphate reacts with an acid, a yellow precipitate of sulphur is formed.

To follow this reaction, we can measure how long it takes for a certain amount of sulphur to form.

This is done by observing how long it takes the black cross on white paper to be hidden.

The chemical reaction that happens can be represented as follows:



The speed or rate of this reaction can be expressed as  $x$  **amount of sulphur** per unit time, so the rate is proportional to  $\frac{1}{\text{time}}$  for a given set of temperatures.

When the temperature of the reactants is increased, the heat is supplied to the particles involved in the reaction and then they move more quickly and they collide with each other more frequently and with more energy necessary to overcome the activation energy barrier to form the product.

Sulphur is produced more quickly as it takes less time for the cross to disappear. This means that in this reaction the speed increases as the temperature is increased.

### Evaluation

What are everyday examples of reactions whose rate is affected by temperature?

#### 13.2.4. Effect of catalyst on the reaction rate

Reaction rate in chemical kinetics is the measure of how fast or slow a reaction is. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning items. The understanding of the effect of using a catalyst or not on the rate of reaction is crucial to predict the speed of that reaction.

### Objective

Learners will be able to explain the effect of the catalyst on the rate of the reaction



### Required materials

#### Apparatus

- 3 conical flasks
- Measuring cylinder (10 mL)
- Electronic balance
- Pipette
- Syringe
- Delivery tube
- A cork with one hole

#### Chemicals

- Zinc granules
- Dilute sulphuric acid (0.5M)
- Copper turning or powder
- Copper (II) sulphate solution

## Experimental set-up

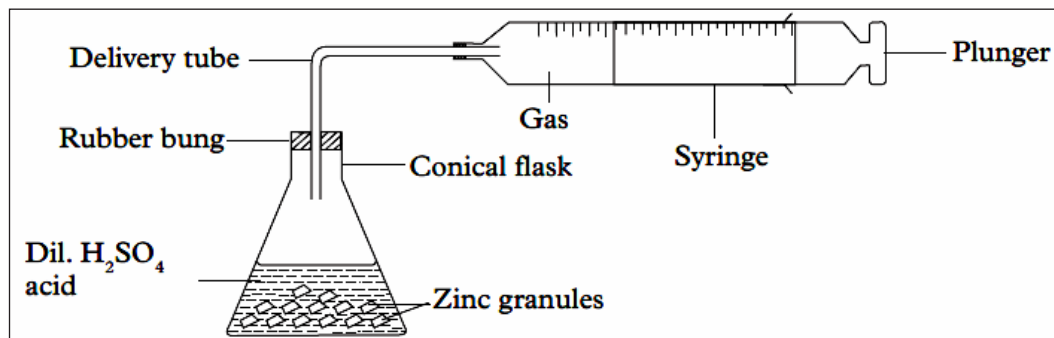


Figure 13.4: Reaction of zinc and hydrochloric acid



## Procedure

1. Put a few pieces of zinc granules into each of three conical flasks and label them **A**, **B** and **C**. (Weigh approximately 5 g of zinc granules into each conical flask).
2. Add 5 mL of dilute sulphuric acid to conical flask **A**. Measure the amount of produced gas as shown in figure 13.4.
3. In conical flask **B**, add a few copper turnings. Make sure they are in contact with the zinc granules. Add 5 mL of dilute sulphuric acid and using a cork with one-hole fitted with a delivery tube stopper quickly the conical flask.
4. Measure the amount of gas produced. Why are copper turnings added to zinc?

**Answer:** *Copper turnings act as catalyst*

5. Add 5 mL of dilute sulphuric acid to conical flask **C**. Add about 1 mL of copper (II) sulphate solution using a dropping pipette. Quickly insert the cork with one-hole fitted with a delivery tube. Measure the amount of produced gas as in procedure 2 above.
6. What happens when copper sulphate is added to zinc?  
**Answer:** *More gas is produced.*
7. Record all your results.

## Data recording

Experiment	A	B	C
Amount of gas produced (in mL)			

## Interpretation of results and conclusion

### Guiding questions

1. What is the term catalyst meant?
2. With the help of examples, differentiate homogeneous from heterogeneous catalyst.
3. Describe the effect of copper on the speed of the reaction between zinc granules and dilute sulphuric acid?

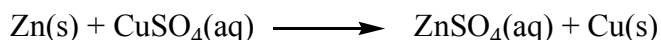
### Answer to guiding questions:

When pure zinc reacts with dilute sulphuric acid, the reaction is slow. Bubbles of hydrogen form on the surface of the zinc. The chemical reaction that occurs is:  $\text{Zn(s)} + \text{H}_2\text{SO}_4(\text{aq}) \longrightarrow \text{ZnSO}_4(\text{aq}) + \text{H}_2$

In conical flask B, copper turnings are the catalyst for the reaction, and the reaction is faster than in conical flask A, but is not as fast as that in conical flask C.

In conical flask C, zinc displaces copper from the copper (II) sulphate solution and the surface of the zinc goes black. The displaced copper metal then acts as a catalyst for the reaction.

The chemical reaction that happens is represented below:



## Evaluation

1. Does the concentration of a catalyst affect reaction rate?
2. How does a catalyst affect a chemical reaction?
3. Explain the relationship between a catalyst and activation energy of a chemical reaction.
4. Give any other five examples of catalysts and the reactions they catalyse.
5. Distinguish, by providing examples, homogeneous and heterogeneous catalysis.



**EXPERIMENT 14.1:** Measure of the rate of reaction by observing the colour change**Rationale**

Reaction rate in chemical kinetics is the determination of how quickly or slowly reactants turn into products. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacturing of many chemicals including fertilisers, drugs and household cleaning substances. For this experiment, we can measure the rate at which the reactant is consumed, or the product is formed at a given time by observing the colour change of reactants or products.

**Objective**

Learners will be able to measure the rate of reaction by observing the colour change in the reaction of sodium thiosulphate and hydrochloric acid.

**Required materials****Apparatus**

- 250 mL conical flask
- Thermometer
- Stop clock
- Stand and clamp
- 50 mL measuring cylinder
- Gauze
- Graph paper
- Piece of white paper
- Test tubes
- 10 mL measuring cylinder

**Chemicals**

- Sodium thiosulphate (0.1M)
- Hydrochloric acid (0.4M)
- Distilled water

### Apparatus

- Beaker (1L)
- 50 mL conical flask
- Tripod

### Chemicals

### Experimental set-up

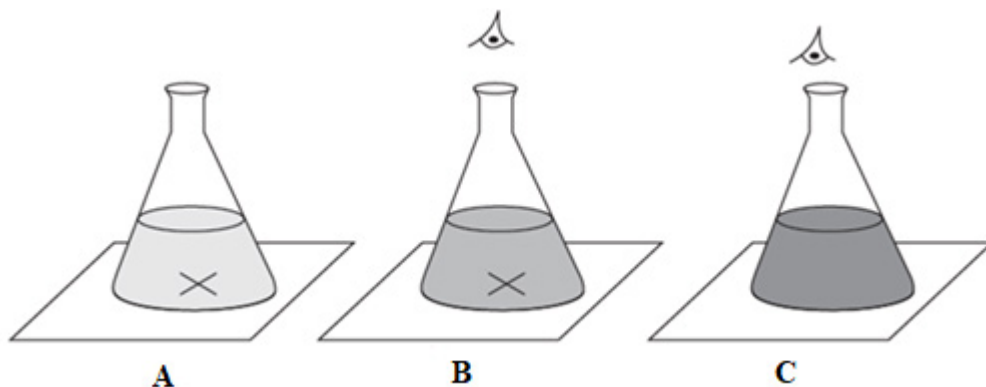


Figure 14.1: Effect of the temperature on the reaction rate



### Procedure

1. Put 100 mL of 0.1M sodium thiosulphate solution into a conical flask.
2. Measure 25 mL of 0.4M hydrochloric acid into the measuring cylinder.
3. Add the acid to the conical flask and immediately start the stopwatch. Swirl the flask to mix the solutions and place it on a piece of paper marked with a cross.
4. Look down at the cross from above. When the cross disappears, stop the watch and note the time taken. Record your results in the table shown below.
5. Repeat the procedure four times reducing the concentration of sodium thiosulphate solution by adding water, the volume of 25 mL of 0.4M hydrochloric acid is kept constant. The total volume is kept equal to 125 mL as indicated in the table below.

**Caution:**

Great care should be taken because this reaction generates sulphur dioxide gas, which is a skin and eye irritant. Perform this demonstration in a well-ventilated lab only. Avoid contact of all chemicals with eyes and skin.

**Data recording**

Volume of $\text{Na}_2\text{S}_2\text{O}_3$ (mL)	Volume of $\text{H}_2\text{O}$ (mL)	Volume of HCl (mL)	Time(s) for the cross to disappear	$\Delta V$	$\Delta t$	
100		25				
50		25				
25		25				
12.5		25				
6.25		25				

**Interpretation of results and conclusion****Guiding question**

1. (a) What causes the cross on paper under the conical flask to disappear?  
(b) what is the name of the main substance formed?
2. Write equation for the reaction that occurs
3. What other observations are made during the reaction?

Plot a graph of time for the cross to disappear against volume of  $\text{Na}_2\text{S}_2\text{O}_3(\text{aq})$

**Answer to guiding questions:**

When sodium thiosulphate reacts with an acid, a yellow insoluble solid of sulphur is formed.

To follow this reaction, we can measure how long it takes for a certain amount of sulphur to form. This is done by observing how long it takes the black cross on white paper to be hidden.

The chemical reaction that happens can be represented as follow:



**As the concentration of the reactant decreases, the number of reacting molecules decreases.** Because of the decrease in the number of molecules, the number of collisions also decreases as a result the rate of the reaction decreases.

## Evaluation

1. Using the collision theory explains how the change in concentration affects the rate of reaction.
2. Describe the chemical test of the gas produced in the reaction between hydrochloric acid and sodium thiosulphate.

## EXPERIMENT 14.2:

## Measure of the rate of reaction by observing volumes changes

### Rationale

Reaction rate in chemical kinetics is the measure of how fast or slow a reaction is. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of many chemicals including fertilisers, drugs and household cleaning agents. We can hence measure the rate at which the reactant is consumed, or product is formed at a given time by observing the volume change if one of the products is a gas.

### Objective

Learners will be able to measure the rate of reaction by observing the volume changes in the reaction of hydrochloric acid with marble chips



### Required materials

#### Apparatus

- Graduated measuring cylinder
- Graduated syringe
- Balance
- Funnel
- Conical flask
- Stop clock
- Stopper

#### Chemicals

- 3.0M HCl
- Calcium carbonate in form of marble chips

## Experimental set-up

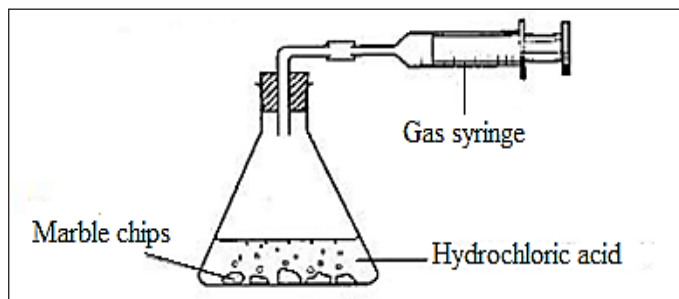


Figure 14.2: Measuring the rate of reaction by observing the volume change



## Procedure

1. Weigh 40g of marble chips (calcium carbonate) and put them in the conical flask.
2. Measure 100 mL of 3.0M HCl and transfer all the acid into the conical flask using a funnel.
3. Close the mouth of the conical flask with a stopper.
4. Connect the conical flask to a flexible graduated syringe. Clamp the flexible graduated syringe.
5. Start the stopwatch and record the total volume of carbon dioxide collected in the flexible graduated syringe.
6. Record your results in the table below:

## Data recording

Time taken(s)	0	10	20	30	40	50	60	70	80	90	100	110	120
Volume of CO <sub>2</sub> (mL)													

## Interpretation of results and conclusion

### Guiding questions

1. What causes the plunger of the syringe to be pushed out?
2. Write a well-balanced chemical equation for the occurring reaction.

3. How could you identify whether the gas has evolved?
4. Describe a chemical test for the evolved gas.

### Answer to guiding questions

Plot a graph of the gas volume of carbon dioxide evolved on the y-axis against time on the x-axis.

Determine the rate of evolution of gas at 10 seconds, 20 seconds, and 100 seconds. What can you conclude about the reaction rate as time progresses?

Marble chips are alkaline compounds which when reacted with hydrochloric acid, **gives carbon dioxide, calcium chloride and water**. Marble chips are made up of calcium carbonate.

The chemical equation of the reaction is



The rate of the reaction increases as time progresses and tends to become stable when the reaction tends to an end.

### Evaluation

1. Why the volume of the gas rapidly increases and decreases before it becomes constant as time progresses.
2. Give any other two examples of reactions that produce gas and write their balanced chemical equations.
3. How could you test the gases evolved in the question 2 above.

### EXPERIMENT 14.3:

### Measure of the rate of reaction by observing mass changes

### Rationale

Reaction rate in chemical kinetics is the determination of how quickly or slowly reactants turn into products. The rate of a reaction is a powerful diagnostic tool as it is essential for the large-scale manufacture of several chemicals including fertilisers, drugs and household cleaning agents. Since the rate of reaction is the change in the amount of a reactant or product per unit time; it can be measured following the change in mass of reactants against time.

## Objective

Learners will be able to measure the rate of reaction by observing the mass changes in the reaction of hydrochloric acid with marble chips.



## Required materials

### Apparatus

- Graduated measuring cylinder
- Graduated syringe
- Electronic balance
- Funnel
- Stop clock
- Watch glass
- Cotton wool

### Chemicals

- 3.0M HCl
- Calcium carbonate in form of marble chips

## Experimental set-up

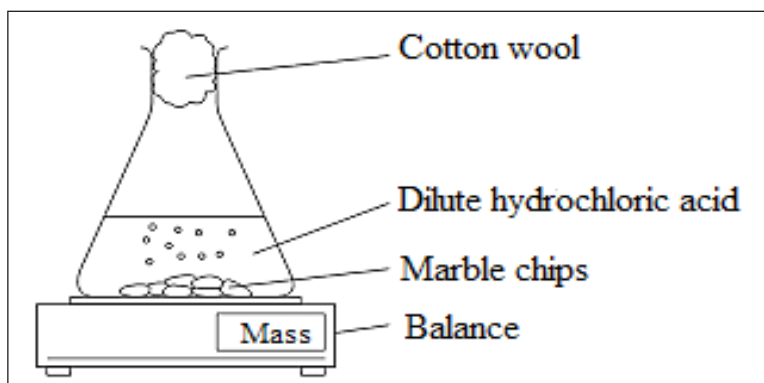


Figure 14.3: Measurement of the reaction rate following change in mass of reactants against time



## Procedure

1. Wear safety glasses.
2. Set up the apparatus as presented in figure 14.3.
3. Put the following items on a top-pan balance:
4. Small conical flask containing about 10 g of marble in six or seven lumps.
5. Measuring cylinder containing 20 mL of 1 M hydrochloric acid.
6. Plug of cotton wool for the top of the conical flask
7. Pour the acid into the conical flask, plug the top with the cotton wool, and put the measuring cylinder back on the balance pan.
8. Allow a few seconds to pass so that the solution is saturated with carbon dioxide. Then start timing and taking mass readings.
9. Record the total mass of the whole reaction mixture and apparatus ( $m$ ) at intervals of 10 seconds at the start, increasing to 30 seconds, until the reaction is over, and the mass no longer changes. Record the final mass ( $m$  final).
10. Record your results in a table like the one below.
11. When the reaction is over, the total mass of carbon dioxide evolved and lost to the atmosphere can be found by subtracting  $m$  final from  $m$  at time = 0 s. This is proportional to the concentration of the hydrochloric acid now when timing started.
12. So  $(m - m$  final) is proportional to the concentration of hydrochloric acid at each time  $t$ .



## Data recording

Time taken(s)	Total mass $m_t$ (g)	$M_t - m_{\text{final}}$ (g)
10		
20		
30		
40		
50		
60		
70		
80		
90		
100		
Etc		

## Interpretation of results and conclusion

### Guiding questions

1. For each set of results, calculate the value for  $(m_t - m_{\text{final}})$ .
2. Plot a graph of  $(m_t - m_{\text{final}})$  against time  $t$ .
3. Interpret the shape of the graph obtained in question 2.

Plot a graph of  $(m_t - m_{\text{final}})$  against  $t$ , putting  $t$  on the horizontal axis.

### Answer to guiding questions

Marble chips are alkaline compounds which when reacted with hydrochloric acid gives **carbon dioxide, calcium chloride and water**. Marble chips are made up of calcium carbonate.

The chemical equation of the reaction is:



### Evaluation

1. Is the reaction of marble chips with hydrochloric acid zero, first or second order with respect to hydrochloric acid?
2. (a) What is/are the environmental effect(s) of the produced gas in the reaction between marble chips and HCl?  
(b) How could you reduce the emission of that gas in the environment?

## EXPERIMENT 14.4: Experimental determination of the reaction order using the graph

### Rationale

Reaction order is defined as the sum of the concentration term exponents in the rate law equation. Rate equation is a mathematical equation that relates the reaction rate with the concentration or pressure of reacting species and a constant rate. The order of reaction provides an indication of how changing the concentration of the reactant will affect the reaction's speed. Its determination carries important applications in chemistry. It explains the relationship between the rate of a chemical reaction's rate and the species concentration in it.

### Objective

Learners will be able to experimentally determine the rate of the reaction between propanone and iodine



### Required materials

#### Apparatus

- Conical flasks
- Measuring cylinder
- Stopwatch
- Piece of paper marked X
- Graph paper
- Pipette

#### Chemicals

- FA1 (0.02M Iodine) prepared by 5g  $I_2$  plus 33 g of KI per litre.
- FA2 (1M Propanone) prepared by 73 mL per litre
- FA3 is 0.01M sodium thiosulphate
- 1M Sulphuric acid
- 0.5M Sodium hydrogencarbonate
- Starch indicator

## Experimental set-up

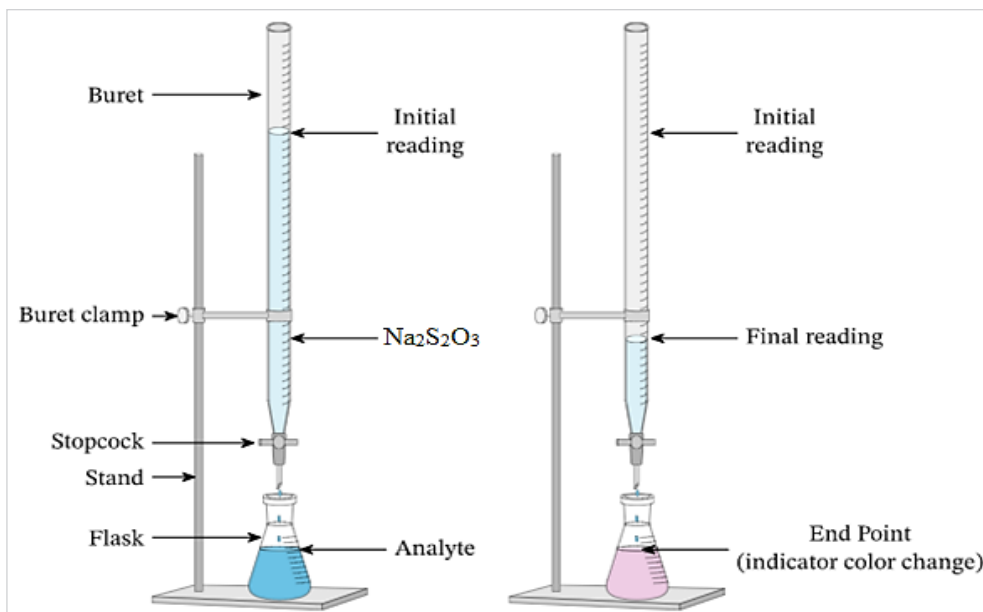


Figure 14.4.: Titration of sodium thiosulphate with iodine as indicator



## Procedure

1. Using a measuring cylinder, measure 25 mL of FA2 and 25 mL of 1M sulphuric acid, and then mix them into a conical flask.
2. Then 0.06M sodium thiosulphate solution into a conical flask. Then 50 mL of FA1 is added with shaking and at the same time the stopwatch is started. The mixture is shaken well.
3. 10 mL of 0.5M sodium hydrogencarbonate is measured into another conical flask.
4. Now 10 mL of acidified mixture of FA1 and FA2 is pipetted into a flask containing  $\text{NaHCO}_3$  and the time noted when this is done.
5. This is then titrated with FA3 using starch as the indicator.
6. The procedure (2) is repeated and the amount of iodine remaining is determined at intervals of about 10 minutes.
7. Plot a graph of  $\text{S}_2\text{O}_3^{2-}$  (vertical axis) against time in minutes.

## Data recording

Time (min)	3	13	23	33	43
Final reading in mL					
Initial reading in mL					
Volume of FA3 used					

## Interpretation of results and conclusion

### Guiding questions

- Describe the reaction order below
  - Zero
  - First
  - Second
- For each set of results, calculate the value of the volume of  $S_2O_3^{2-}$  (vertical axis) against time in minutes.
- Interpret the shape of the graph.
- State the role of the substance below in the experiment to determine the rate of the reaction between propanone and iodine
  - sodium bicarbonate
  - sulphuric acid

### Answer to guiding questions

The plot is a straight line which shows that the rate of disappearance of iodine (rate of reaction) does not depend on concentration of iodine. Therefore, the order of reaction is zero with respect to iodine.

The volume of the thiosulphate is proportional to the concentration of iodine left at each time during the reaction.

Considering reaction:  $A \rightarrow B$

If **a** is the initial concentration of **A** and **x** is the concentration of the product **B** after a time **T**.

Therefore, rate of reaction is given by  $\frac{[B]}{dt} = K(a - x)^0$

i.e.

$\frac{d[B]}{dt} = \frac{dx}{dt} = K(a - x)^0$  where **K** = rate constant

Therefore,  $\frac{dx}{dt} = K$   $dx = Kdt$

Integrating both sides of the equations

$$\int dx = K \int dt$$

$$x = Kt$$

$$x = [A] \text{ at time } t.$$

The integrated rate equation for zero order reaction is  $x = Kt$

$$K = \frac{x}{t}$$

Sodium hydrogen carbonate is used to stop the reaction between the acidified propanone and iodine. Sulphuric acid is used as a catalyst.

### Evaluation

1. From the shape of the graph plotted, deduce the order of reaction with respect to iodine.
2. Determine the rate constant of the reaction.
3. What is the role of sodium hydrogencarbonate ions in the experiment?

## References

1. J.A.S. Howell, P.M. Buckinshaw-Ligand Substitution Reactions at Low-Valent Four-, Five-, and Six-Coordinate Transition-Metal Complexes, *Chem. Rev.* 83, 557, 1983.
2. D.J. Darensbourg-Mechanistic Pathway for Ligand Substitution Processes in Metal Carbonyls, *Adv. Organomet. Chem.* 21, 113, 1982.
3. Chemistry S6, Student book, REB, Kigali, August 2018
4. Chang Raymond, General Chemistry, The Essential concepts, 6<sup>th</sup> Edition; McGraw-Hill, 2011
5. Graham Hill & John Holman, Chemistry in context, 5<sup>th</sup> Ed. Nelson Thornes Ltd, 2000
6. Atkins, Peter (2006). *Atkins' Physical Chemistry*. Oxford University Press. ISBN 0198700725
7. <https://www.scientificamerican.com/article/bring-science-home-playing-with-polymers/>