



CHEMISTRY EXPERIMENTS
USER GUIDE
FOR
SENIOR FIVE

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FOREWORD

Dear teacher,

Rwanda Basic Education Board (REB) is honoured to present Chemistry experiments user guide for Senior five. 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

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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 five. It would not have been successful without their active participation.

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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

CBC	: Competence-Based Curriculum
ICT	: Information Communication Technology
Lab	: Laboratory
STEM	: Science, Technology, Engineering and Mathematics
CPD ITMS	: Continuous Professional Development Certificate in Innovative Teaching Mathematics and Science
IBL	: Inquiry Based Learning
KBC	: Knowledge Based Curriculum
SET	: Science and Elementary Technology
UR-CE	: University of Rwanda- College of Education
CTRLR	: Curriculum, Teaching and Learning Resources
AIMS	: African Institute for Mathematical Sciences

TABLE OF CONTENTS

FOREWORD	iii
ACKNOWLEDGEMENT	v
LIST OF ACRONYMS	vi
TABLE OF CONTENTS	vii
PART 1: GENERAL INTRODUCTION	1
1. Laboratory experiments in the Competence Based Curriculum	1
2. Type of laboratory experiments	2
3. Organization, analysis, and interpretation of data	2
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	13
PART 2: LIST OF MAIN KIT ITEMS AND LAB MATERIALS NEEDED IN SCHOOLS	15
PART 3: DETAILED EXPERIMENTS PER GRADE OF O'LEVEL	28
UNIT 2: ALKANES	29
Experiment 2.1: Laboratory preparation of methane gas	29
UNIT 3: ALKENES AND ALKYNES	32
Experiment 3.1: Laboratory preparation of ethene gas and testing it with bromine water	32
Experiment 3.2: Laboratory preparation of ethyne and test using bromine water	34

UNIT 5: ALCOHOLS AND ETHERS 38

Experiment 5.1: Comparison of oxidation reactions of primary, secondary and tertiary alcohols.....38

Experiment 5.2: Distinction of primary, secondary and tertiary alcohols by Lucas test40

Experiment 5.3: Experiment to distinguish between methyl and non-methyl alcohols42

UNIT 6: CARBONYL COMPOUNDS..... 45

Experiment 6.1: Differentiation between carbonyl compounds and other organic compounds by Brady's test.....45

Experiment 6.2: Preparation of ethanal47

Experiment 6.3: Preparation of propanone50

Experiment 6.4: Distinction between ketones and aldehydes using Fehling's reagent or Benedict's solution52

Experiment 6.5: Distinction between ketones and aldehydes using Tollens's reagent.....55

Experiment 6.6: Distinction of methyl ketones from aldehydes and other ketones by Iodoform test.....58

UNIT 7: CARBOXYLIC ACIDS AND ACYL CHLORIDES 62

Experiment 7.1: Experiment to distinguish the carboxylic acids from other organic compounds using sodium carbonate.....62

Experiment 7.2: Preparation of carboxylic acid by oxidation of primary alcohol by using acidified potassium permanganate (VII).....64

UNIT 8: ESTERS, ACID ANHYDRIDES, AMIDES AND NITRILES..... 67

Experiment 8.1: Esterification reaction.....67

Experiment 8.2: Saponification reaction.....69

Experiment 8.3: Experiment to prepare aspirin using acetic anhydride and salicylic acid72

UNIT 9: AMINES AND AMINO ACIDS	75
Experiment 9.1: Distinction of primary, secondary, and tertiary amines in a solution	75
Experiment 9.2: Experiment to test for the presence of amino acid in a solution	78
UNIT 11: SOLUTIONS AND TITRATION	80
Experiment 11.1: Preparation of solutions of different concentrations..	80
Experiment 11.2: Titration of sodium hydroxide solution by standard solution of hydrochloric acid.....	83
Experiment 11.3: Determination of moles of water of crystallization in oxalic acid by titration.....	86
Experiment 11.4: Determination of the percentage purity of an impure sample	90
Experiment 11.5: Determination of the relative atomic mass of the metal M in a metal hydrogen sulphate ($MHSO_4$).....	94
Experiment 11.6: Back titration of magnesium oxide.....	97
Experiment 11.7: Redox titration using potassium permanganate..	100
Experiment 11.8: Redox titration of iodine using sodium thiosulfate	103
UNIT 12: CONDUCTIVITY OF SOLUTIONS	107
Experiment 12.1: Verification of the conductivity of solutions.....	107
Experiment 12.2:	110
Comparison of electrolytic conductivity and metallic conductivity.....	110
UNIT 13: ELECTROLYSIS	113
Experiment 13.1: Electrolysis of water	113
Experiment 13.2: Electrolysis of concentrated sodium chloride solution	116
Experiment 13.3: Electrolysis of dilute sodium chloride solution.....	119

Experiment 13.4: Electrolysis of concentrated copper (II) sulphate solution using carbon electrode..... 122

Experiment 13.5: Electroplating of graphite using copper (II) sulphate solution 125

UNIT 14: ENTHALPY CHANGE OF REACTIONS128

Experiment 14.1: Determination of enthalpy change of combustion of ethanol 128

Experiment 14.2: Determination of enthalpy change of neutralization of hydrochloric acid with sodium hydroxide solution..... 131

Experiment 14.3: Determination of enthalpy change of dissolution of sodium hydroxide in water 133

REFERENCES:..... 136

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 for ordinary level 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 key question, 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

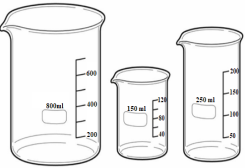



After conducting a laboratory experiment, students should write a report about their findings and the conclusion they reached. The report to be made depends on the level of students. The following is a structure of the report to be made by a group of secondary school learners (S4-S6).




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.




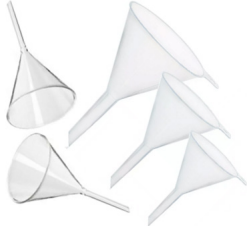

PART 2: LIST OF MAIN KIT ITEMS AND LAB MATERIALS NEEDED IN SCHOOLS




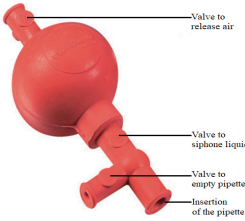
A. List of Apparatus



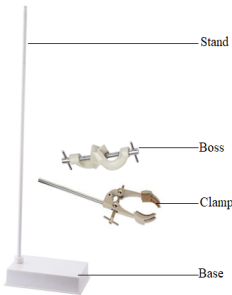


#	Item and description	Picture	Description of uses
1	Beaker		Used to hold and heat liquids. Multipurpose and essential in the laboratory.
2	Brushes		Used to easily clean the inside of a test tubes and other glassware.
3	Buchner funnel		Used with vacuum flask for performing vacuum filtration.
4	Bunsen burner		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. Bunsen burner is used for heating and exposing items to flame.







5	Burette		<p>Operating the Burette</p> <p>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.</p> <p>Before delivering any solution, record the initial burette reading in your notebook.</p> <p>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.</p> <p>Wait a couple of seconds, then record the final burette reading.</p>
6	Burette clamp		<p>Used to hold burettes on a ring stand.</p>
7	Clay triangle		<p>Used to hold crucibles when they are being heated. They usually sit on a ring stand.</p>





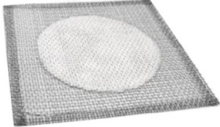

8	Crucible with lid		Used to heat small quantities to very high temperatures.
9	Crucible tong		Used to hold crucibles and evaporating dishes when they are hot.
10	Disposable pipette		Used for moving small amounts of liquid from place to place. They are usually made of plastic and are disposable.
11	Electronic balance		<p>Used for weighing substances or objects, usually in grams.</p> <p>Place the electronic balance on a flat, stable surface indoors.</p> <p>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.</p> <p>Press the “Tare” or “Zero” button to cancel automatically the weight of the container. The digital display will show zero again.</p> <p>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.</p>





			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		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.
13	Evaporating dish		Used to recover dissolved solids by evaporation.
14	Forceps		Used for picking up and moving small objects.
15	Glass funnel & Polypropylene funnel		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.
16	Glass stir rod		Used to stir liquids. They are usually made of glass.

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</p>






			<p>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		Used for moving small amounts of solid from place to place.
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		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.
40	Cardboard cover		
41	Rubber tube		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.

42	Borosilicate delivery tube		The delivery tube is particularly useful for bubbling a gas from a gas cylinder or stoppered vessel through a liquid.
43	Trough		The trough is used for collecting gases, such as hydrogen, oxygen and nitrogen. Troughs require a liquid such as water.
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.
53	Plastic balls		For designing the molecular model

B. List of chemicals

SN	Chemicals
1	Acetic acid, CH_3COOH (aq)
2	Alcohols (primary, secondary, and tertiary)
3	Aluminium oxide, Al_2O_3 (s)
4	Amino acid
5	Anhydrous zinc chloride (ZnCl_2)
6	Acetic anhydride, $\text{CH}_3\text{COOCH}_3$ (l)
7	Animal or plant oil
8	Anti-bumping granule
9	Benedict's solution
10	Bromine water
11	Calcium acetate, $(\text{CH}_3\text{COO})_2\text{Ca}$ (s)
12	Calcium carbide
13	Calcium oxide, CaO (s)
14	Chloroform, CHCl_3
15	Phosphoric acid, H_3PO_4
16	Copper (II) sulphate
17	Distilled water
18	Ethanal
19	Ethanoic acid
20	Ethanol
21	Fehling's solution
22	Hydrated oxalic acid, $(\text{COOH})_2 \cdot n\text{H}_2\text{O}$ (s)
23	Hydrochloric acid
24	Sodium hydroxide, NaOH (s)
25	Iodine solution
26	Lime water
27	Litmus paper
28	Magnesium oxide
29	Methylated alcohol (R-CH(OH)-CH_3) and non-methylated alcohols

30	Ninhydrin (0.2%)
31	Primary, secondary, and tertiary amines
32	Phenolphthalein indicator
33	Potassium hydrogen sulphate, $\text{KHSO}_4(\text{s})$
34	Potassium hydroxide, KOH
35	Potassium iodate (KIO_3)
36	Potassium iodide, KI
37	Potassium permanganate, KMnO_4
38	Primary, secondary, and tertiary alcohols
39	Propanal, $\text{CH}_3\text{CH}_2\text{CHO}$
40	Propanone, CH_3COCH_3
41	Salicylic acid, $\text{C}_6\text{H}_4\text{OHCOOH}(\text{s})$
42	Sodium chloride, NaCl
43	Sodium acetate, $\text{CH}_3\text{COONa}(\text{s})$
44	Sodium bicarbonate, $\text{NaHCO}_3(\text{s})$
45	Sodium carbonate, $\text{Na}_2\text{CO}_3(\text{s})$
46	Sodium dichromate, $\text{Na}_2\text{Cr}_2\text{O}_7(\text{s})$
47	Sodium hydroxide
48	Sodium nitrite, NaNO_2
49	Sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$
50	Sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$
51	Starch
52	Sugar
53	Sulfuric acid, H_2SO_4
54	Silver nitrate
55	Ammonia solution
56	2,4-dinitrophenylhydrazine
57	Methanol

**PART 3: DETAILED EXPERIMENTS PER
GRADE OF O'LEVEL**

EXPERIMENTS FOR SENIOR FIVE

EXPERIMENT 2.1: Laboratory preparation of methane gas**Rationale**

Methane is the simplest hydrocarbon which can be formed by the decay of natural organic materials. It is used primarily as fuel to produce heat and light. It is also used to manufacture some other organic substances. In this experiment, methane will be representing other alkanes.

Objective

Learners will be able to prepare and collect methane gas.

**Required materials****Apparatus**

- Stand and accessories
- Delivery tube
- Gas jar
- Boiling tube
- Stopper with one hole
- Bunsen burner
- Matchbox (or lighter)
- Electronic balance

Chemicals

- Sodium hydroxide, NaOH(s)
- Sodium acetate, CH₃COONa(s)
- Calcium oxide, CaO(s)
- Water

Experimental set-up

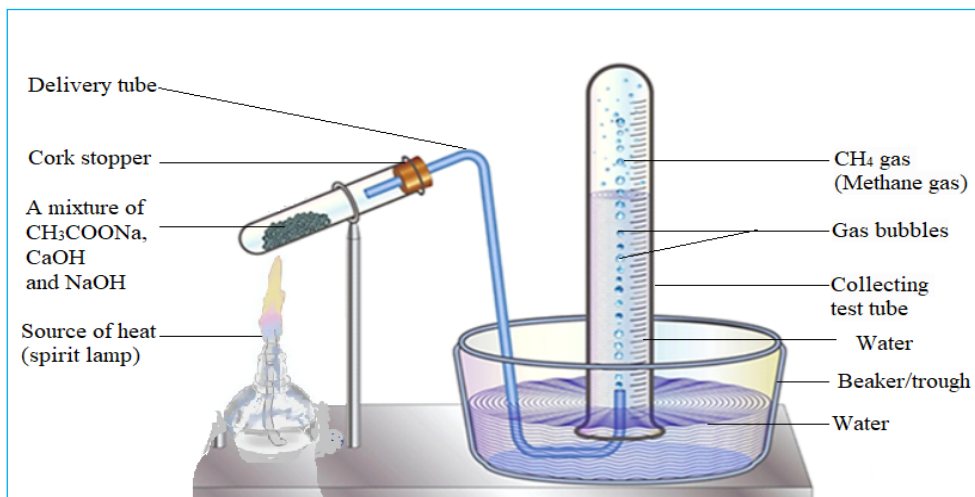


Figure 2.1: Laboratory preparation of Methane gas



Procedure

1. Mix 2 g of sodium acetate, 2 g of calcium oxide and 2 g of sodium hydroxide in a beaker.
2. Transfer the mixture into a boiling tube.
3. Seal the boiling tube with a stopper with a gas-delivery tube.
4. Fix the apparatus as shown in figure 2.1.
5. Heat the boiling tube gently with the cold part of the flame to avoid local overheating and keep the flame in motion. Note your observation.

Answer: After one minute the colorless gas is liberated.

Interpretation of results and conclusion

Guiding questions

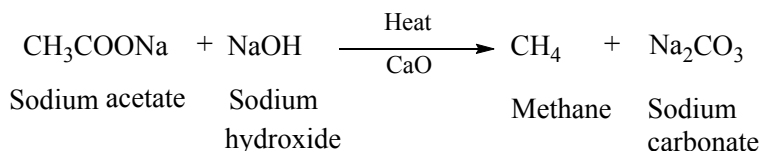
1. How is methane gas prepared in the laboratory? Support your answer with chemical equations.

2. What is the role of calcium oxide in the above preparation?

Answer to guiding questions

When the mixture of sodium acetate and soda lime (mixture of sodium hydroxide and calcium oxide) is heated, there is a production of methane gas and sodium carbonate. Calcium oxide is used to keep sodium hydroxide dry because it is highly hygroscopic.

Chemical equation of the reaction:



Evaluation

1. Why is the methane gas collected by downward displacement of water during its preparation?

Answer: The gas is insoluble and less dense than water.

2. Describe how is methane gas prepared in the laboratory?

Answer: The methane gas is the product of a chemical reaction between sodium acetate and sodium hydroxide by heating.

3. List at least two characteristics of methane gas.

Answer: Methane gas is lighter (less dense) than air.
Methane gas is combustible and flammable

EXPERIMENT 3.1:

Laboratory preparation of ethene gas and testing it with bromine water

Rationale

Ethene is mainly used as a major synthetic route for manufacturing many plastics and medicines. Beta-carotene (a polyalkene) gives an orange pigment responsible for the color of carrots used as a source of vitamin A. In this experiment, the students will explore the laboratory preparation of ethene gas and its chemical identification.

Objective

Learners will be able to prepare and test ethene gas.



Required materials

Apparatus

- Boiling tube(pyrex or borosilicate)
- Rubber stopper with hole
- Delivery tube
- Retort stand
- Bunsen burner
- Glass rod
- Trough

Chemicals

- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$
- Aluminium oxide, $\text{Al}_2\text{O}_3(\text{s})$
- Water
- Bromine water

Experimental set-up

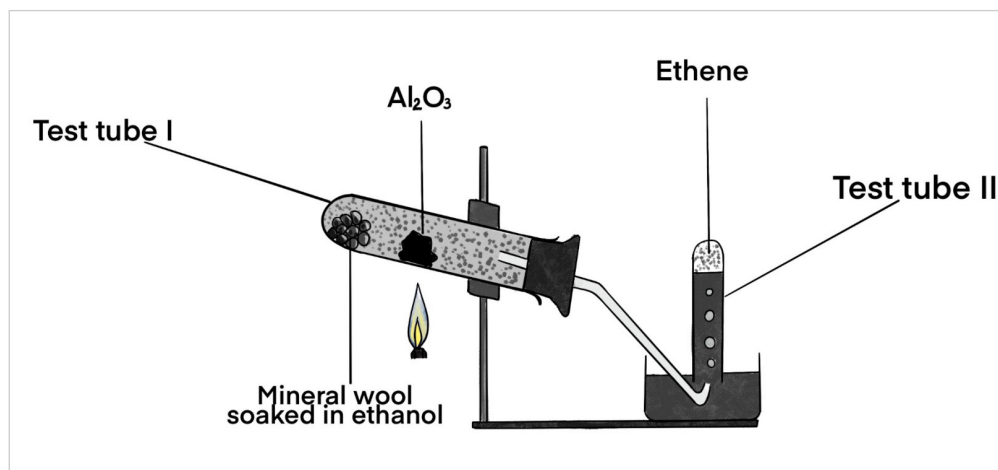


Figure 3.1: Preparation of ethene in laboratory



Procedure

1. Pour a small amount of ethanol into a test tube.
2. Add some cotton wool to soak up ethanol, using a glass rod to push the wool down the tube
3. Put a small amount of aluminum oxide about half-way along the test tube
4. Arrange the apparatus as shown in figure 3.1
5. Heat aluminium oxide and make sure that it is adjusted to a blue flame. What do you observe?

Answer: After some minutes the colorless gas is liberated.

6. Add brown bromine liquid (Bromine water) in a test tube containing ethene gas and shake gently. What do you observe?

Answer: The ethene gas turns brown bromine water into colorless.

Interpretation of results and conclusion

Guiding questions

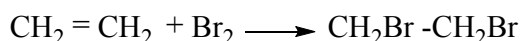
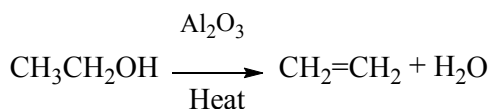
1. Describe briefly how is ethene gas prepared in the laboratory? Provide chemical equations of the reaction.
2. Explain briefly how is ethene gas tested in the laboratory using bromine water? Support your answer with chemical equation.

Answer to guiding questions:

When ethanol vapor passes over hot aluminum oxide, it is dehydrated to produce ethene gas which decolorizes brown bromine water.

To test ethene gas, bromine water is used. Ethene turns brown bromine water into colorless as it reacts with double bonds.

The chemical equations of reactions:



Evaluation

1. Why is ethene gas collected by downward displacement of water?
Answer: The ethene gas is insoluble and less dense than water.
2. Write down a chemical equation that should be used to prepare and test ethene gas.

EXPERIMENT 3.2:

Laboratory preparation of ethyne and test using bromine water

Rationale

Ethyne, also known as acetylene, is an important organic compound in our daily life as a raw material in the production of other essential organic compounds such as alcohol, vinegar, and plastic materials. In addition, ethyne can be used in the welding of metals when combined with oxygen gas. Ethyne gas is used

to fill rubber balloons enabling them to fly high during celebrations. In this experiment, the students will explore the laboratory preparation of ethyne gas and its chemical identification.

Objective

Learners will be able to prepare and test ethyne gas.



Required materials

Apparatus

- Dropping funnel
- Delivery tube
- Retort stand
- Glass rod
- Trough
- Conical flask

Chemicals

- Calcium carbide
- Water
- Bromine water

Experimental set-up

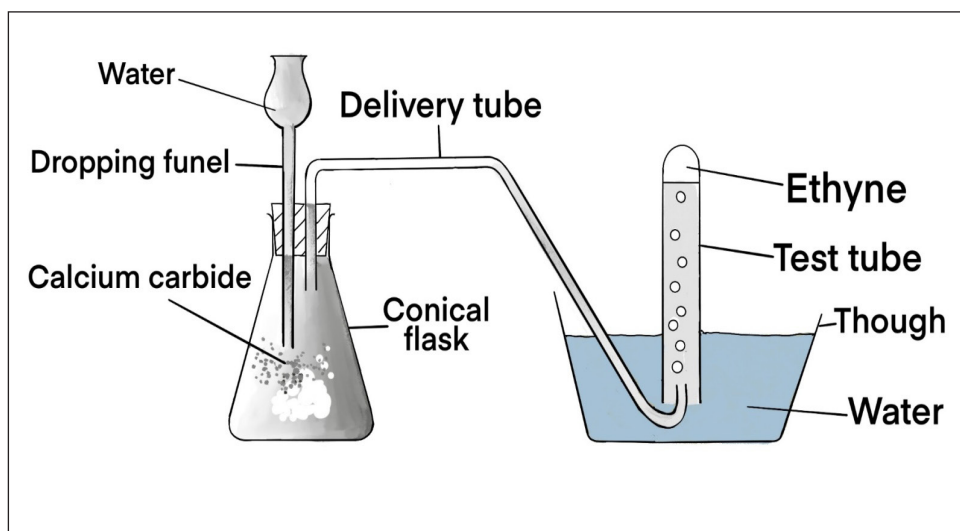


Figure 3.2: Preparation of ethyne in lab



Procedure

1. Place 2 g of calcium carbide in a conical flask.
2. Arrange the apparatus as displayed in figure 3.2
3. Using the dropping funnel, add water dropwise.
4. Collect the produced gas in the test tube. What do you observe?

Answer: After a while the colorless gas evolves, and it displaces water from the test tube.

5. Add bromine water in a beaker containing ethyne gas. What do you observe?

Answer: The gas decolorizes bromine water

Interpretation of results and conclusion

Guiding questions

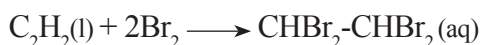
1. Explain how is ethyne gas prepared in the laboratory?
2. How is ethyne gas identified in the laboratory? Explain.

Answer to guiding questions

Water passes over calcium carbide to produce ethyne gas. The reaction is represented by the following chemical equation:



When ethyne gas passes through bromine water, the bromine reacts with ethyne producing a colorless substance.



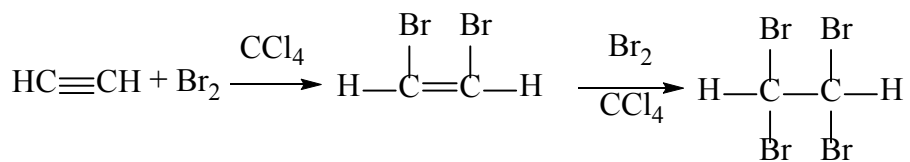
Evaluation

1. Why is ethyne gas collected by downward displacement of air during its laboratory preparation?

Answer: The gas is insoluble in water and less dense than water.

2. Write chemical equations to show what happens when ethyne gas reacts with bromine water

Answer: When ethyne gas reacts with bromine water, it first forms 1,2-dibromoethene followed by the formation of 1,1,2,2-tetrabromoethane.



Ethyne

1,2-Dibromoethene

1,1,2,2-Tetrabromoethane

3. Write a chemical equation to show how ethyne is prepared from calcium carbide.

Answer: $2\text{CaC}_2(\text{s}) + 2\text{H}_2\text{O}(\text{l}) \longrightarrow \text{H}-\text{C}\equiv\text{C}-\text{H} + \text{Ca}(\text{OH})_2(\text{aq})$

EXPERIMENT 5.1:

Comparison of oxidation reactions of primary, secondary and tertiary alcohols.

Rationale

Alcohol is a class of compounds that contain hydroxyl (-OH) functional group. Alcohols are recognized to be one of the most often occurring organic compounds. They are used in the manufacturing of perfumes, and in the process of synthesizing other organic compounds like aldehydes, ketones, and carboxylic acids. In this experiment, the students will explore the oxidation reaction of different classes of alcohols.

Objective

Learners will be able to compare oxidation reactions of primary, secondary and tertiary alcohols.



Required materials

Apparatus

- Test tubes
- Water bath
- Test tube rack
- Cork

Chemicals

- Primary, secondary, and tertiary alcohols
- KMnO_4
- Concentrated sulphuric acid



Procedure

1. Put three test tubes in a test tube rack labeled A, B and C (primary, secondary, and tertiary respectively).
2. Add 3-4 mL of alcohols to be tested in each test tube.
3. Pour 2 mL of concentrated sulphuric acid approximately 1M to each of the above test tubes.
4. Add one drop of saturated KMnO_4 solution to each test tube. Shake vigorously to mix.
5. Record your observations.

Answer: In a test tube containing primary and secondary alcohols, a brownish color appears while the purple KMnO_4 color disappears. With the tertiary alcohol no color changes since the purple color remains.

Note: Potassium dichromate may also be used to distinguish classes of alcohols. It changes from orange to green for primary and secondary alcohols and there is no change of color for tertiary alcohols.

Interpretation of results and conclusion

Guiding question

1. What are the chemical effects of oxidizing agents (KMnO_4)/ H^+ on primary, secondary and tertiary alcohols.

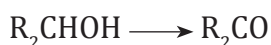
Answer to guiding questions

Primary and secondary alcohols are easily oxidized by acidified permanganate (vii) ions whereas tertiary alcohols are not easily oxidized.

Primary alcohols are oxidized by KMnO_4 in acidic medium to aldehyde and carboxylic acid while in the same conditions,

secondary alcohols are oxidized to ketones whereas tertiary alcohols, in contrast, are not oxidized.





Evaluation

1. You are provided the following alcohols: A, B, C, and D. Carry out the experiment to identify which one is primary, secondary or tertiary alcohol.

EXPERIMENT 5.2:

Distinction of primary, secondary and tertiary alcohols by Lucas test

Rationale

Alcohols are classified into primary, secondary, and tertiary. They can also be classified based on the number of hydroxyl groups attached to the longest chain of carbon atoms. Glycerin, also known as glycerol (tri-alcohol) is used as an ingredient in a variety of food and beverage products. Ethanediol is mainly used in the manufacture of polyester fibers and for antifreeze formulations. This experiment aims to differentiate primary, secondary, and tertiary alcohols using Lucas test.

Objective

Learners will be able to distinguish between primary, secondary and tertiary alcohols by using Lucas test.



Required materials

Apparatus

- Test tubes
- Water bath

Chemicals

- Hydrochloric acid (HCl.conc)
- Anhydrous zinc chloride ($ZnCl_2$)
- Alcohols (primary, secondary, and tertiary)



Procedure

1. Take equimolar quantities of zinc chloride and concentrated HCl and make a solution (Lucas reagent).
2. Put a very small amount of the given alcohols in test tubes (primary in the first test tube, secondary in the second test tube and tertiary in the third test tube).
3. Add 2mL of the Lucas reagent in each of the test tubes containing the given samples and mix them.
4. Record the time until the solution becomes cloudy. Note your observations.

Answer: In the first test tube containing primary, no turbid or cloudy solution is observed at room temperature. The turbidity is observed after heating for about 30-45 minutes. In the second test tube containing secondary alcohol, cloudy solution is observed at room temperature about after 3-5 minutes. In the third test tube containing tertiary alcohol, turbid or cloudy solution is instantly observed at room temperature.

Interpretation of results and conclusion

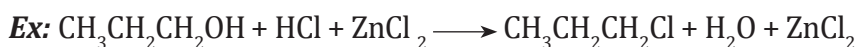
Guiding questions

1. What is the effect of Lucas reagent on primary, secondary and tertiary alcohols?
2. Write down chemical equations for reactions that occur when Lucas reagent reacts with primary, tertiary and secondary alcohols.
3. In alcohol testing by Lucas reagent, what is the role of zinc chloride?

Answer to guiding questions

Lucas test is used to distinguish between primary, secondary and tertiary alcohols. If turbidity appears immediately, the alcohol is tertiary. If it appears after about 5 minutes the alcohol is secondary. For the primary alcohol, turbidity should appear after more than 30 minutes or on heating. Thus, the primary, secondary and tertiary alcohols can be differentiated based on the rate at which they turn the solution turbid when reacted with Lucas reagent.

The chemical reactions involved are as follow:



The alkyl chloride formed is responsible for the turbidity of the solution.

Evaluation

1. What does Lucas test mean?

Answer: The Lucas test is a test intended to distinguish between primary, secondary, and tertiary alcohols.

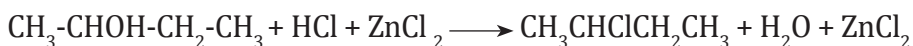
2. Which alcohol does not react readily with Lucas reagent?

Answer: Primary alcohols do not react readily at room temperature with the Lucas reagent, whereas tertiary alcohols react immediately.

3. What is catalyst used in Lucas test?

Answer: Zinc chloride is used as a catalyst

4. Write a balanced equation of the reaction when butan-2-ol is being tested in Lucas test



EXPERIMENT 5.3:

Experiment to distinguish between methyl and non-methyl alcohols

Rationale

Iodoform (CHI_3) is a pale yellow volatile crystalline substance with a penetrating and distinctive odor, with a sweetish taste. This organo-iodine compound has an antimicrobial effect and is commonly applied on open wounds to exert a wide spectrum of non-selective antibacterial action and used as a disinfectant for the medical and hygiene purposes. Haloform (iodoform) test helps to differentiate methyl alcohol and non-methyl alcohols. It is a qualitative test.

Objective

Learners will be able to distinguish between methyl and non-methyl alcohols.



Required materials

Apparatus

- 2 test tubes
- Water bath
- Droppers

Chemicals

- Iodine solution (I_2 , 10%)
- Sodium hydroxide solution (NaOH, 10 %)
- Methylated alcohol ($R-CH(OH)-CH_3$) and non-methylated alcohols



Procedure

1. Add 5 droplets of the non-methylated alcohol in the first test tube and a methylated one in the second test tube.
2. Add 10 droplets of iodine solution in each test tube, and shake to mix. Heat in a water bath. Record your observation.
3. Add enough droplets of 10% NaOH and shake each test tube side to side until the iodine color is discharged

What do you observe?

Answer: A yellow precipitate is observed in methylated alcohol $R-CH(OH)-CH_3$ and no observable change in non-methylated alcohol.

Note: if the precipitate is not formed at this temperature, it may be necessary to increase the temperature of the mixture.

Interpretation of results and conclusion

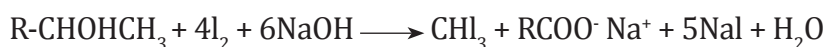
Guiding questions

1. How are methylated and non-methylated alcohols distinguished by iodoform test? Explain.
2. What are the chemical equations occurring in the iodoform test of methylated alcohols?

Answer to guiding questions:

Secondary alcohols that have a methyl group linked with carbon bonded to hydroxyl group (-OH) are called methylated alcohols. The general formula is R-CH(OH)-CH₃ including ethanol. The methylated alcohols react with I₂ in NaOH to give a yellow precipitate of triiodomethane (CHI₃ commonly known as iodoform) and sodium carboxylates. This test is called iodoform test. Other alcohols do not give a positive test.

The chemical equation of the reaction that occurs during iodoform test:



Evaluation

1. Which primary alcohol gives a positive test with iodoform?
Answer: The primary alcohol is ethanol (CH₃CH₂OH).
2. Why do methanol and tertiary alcohols do not give positive results on iodoform tests?
Answer: Methanol and tertiary alcohols do not give positive results on iodoform test because they do not have methyl group CH₃ bonded to the C-OH.
3. Which alcohols in the list below give or do not give a positive test on iodoform test? CH₃CHOHCH₃, CH₃CH₂CH₂OH, CH₃CHOHCH₂CH₃
Answer: CH₃CHOHCH₃ and CH₃CHOHCH₂CH₃ give positive tests, while CH₃CH₂CH₂OH do not give iodoform positive test.

EXPERIMENT 6.1:

Differentiation between carbonyl compounds and other organic compounds by Brady's test

Rationale

Carbonyl compounds (aldehydes and ketones) are of great importance both in biological and synthetic chemistry. However, the high reactivity of the carbonyl group among other organic compounds enables them to function more as intermediate in metabolism or in synthesis as end product. Their identification and distinction from other organic compounds is very important.

Objective

Learners will be able to distinguish carbonyl compounds from other organic compounds.



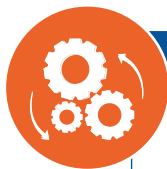
Required materials

Apparatus

- Test tubes
- Test tube rack
- Water bath

Chemicals

- 2,4-dinitrophenylhydrazine (Brady's reagent)
- Ethanal, CH_3CHO
- Propanone (acetone), CH_3COCH_3
- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$



Procedure

1. Arrange three test tubes in a test tube rack, labeled A, B and C.
2. Put 2mL of ethanal, acetone and ethanol in test tube A, B and C respectively.
3. Add 6 drops of 2,4-dinitrophenylhydrazine reagent to each of the three test tubes. Place the test tubes in the water bath for a few minutes. What do you observe?

Answer: A deep orange /yellow precipitate is formed in test tube A, and B containing carbonyl compounds. In the third test tube (C) there is no observable change.

Note: In laboratory, Brady's reagent (2,4 - dinitrophenylhydrazine) is prepared in the following way:

Add 1.0 g of solid to 50 mL of methanol. Stir well, and add 2 mL of concentrated sulphuric acid dropwise. Filter off any remaining undissolved solid.

Interpretation of results and conclusion

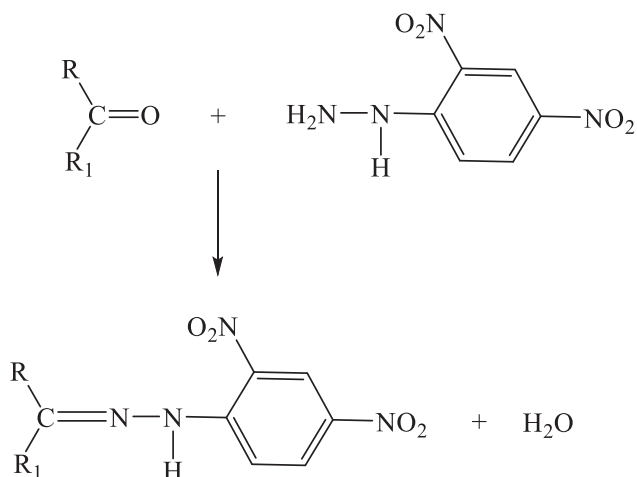
Guiding questions

1. How are carbonyl compounds distinguished from other organic compounds? Explain your answer using chemical equation.

Answer to guiding questions

The presence of carbonyl group in an aldehyde and a ketone can be easily tested by using a solution of 2,4-dinitrophenyl hydrazine (often abbreviated to 2,4-DNPH), also called Brady's reagent. If an aldehyde or ketone is present, a deep orange / yellow precipitate is formed.

The chemical equation of the reaction:



Orange or yellow precipitate formed
as positive test of aldehydes or ketones

Evaluation

1. What does Brady's reagent mean?

Answer: Brady's reagent is the chemical compound $C_6H_3(NO_2)_2NHNH_2$ is known as 2,4-Dinitrophenylhydrazine (DNPH, Brady's reagent). Dinitrophenylhydrazine is a red solid. It is mostly used for the qualitative research of aldehyde ketone and other related carbonyl group.

2. Describe how are carbonyl-based compounds (aldehydes and ketones) tested?

Answer: see interpretation.

EXPERIMENT 6.2: Preparation of ethanal

Rationale

Ethanal is one of the most important aldehydes, occurring widely in nature and being prepared in the laboratory by oxidation of ethanol. Ethanal occurs naturally in coffee, bread, and ripe fruit. It is used in the manufacture of acetic acid, perfumes, dyes, and drugs. This experiment reveals the laboratory preparation method of ethanal.

Objective

Learners will be able to prepare and collect ethanal.



Required materials

Apparatus

- Beakers
- Tap funnel
- Condenser
- Round bottomed flask
- Iced water bath
- Bunsen burner

Chemicals

- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}(\text{l})$
- Concentrated Sulfuric acid, H_2SO_4 cc
- Sodium dichromate, $\text{Na}_2\text{CrO}_7(\text{s})$
- Distilled water
- Anti-bumping granule

Experimental set-up

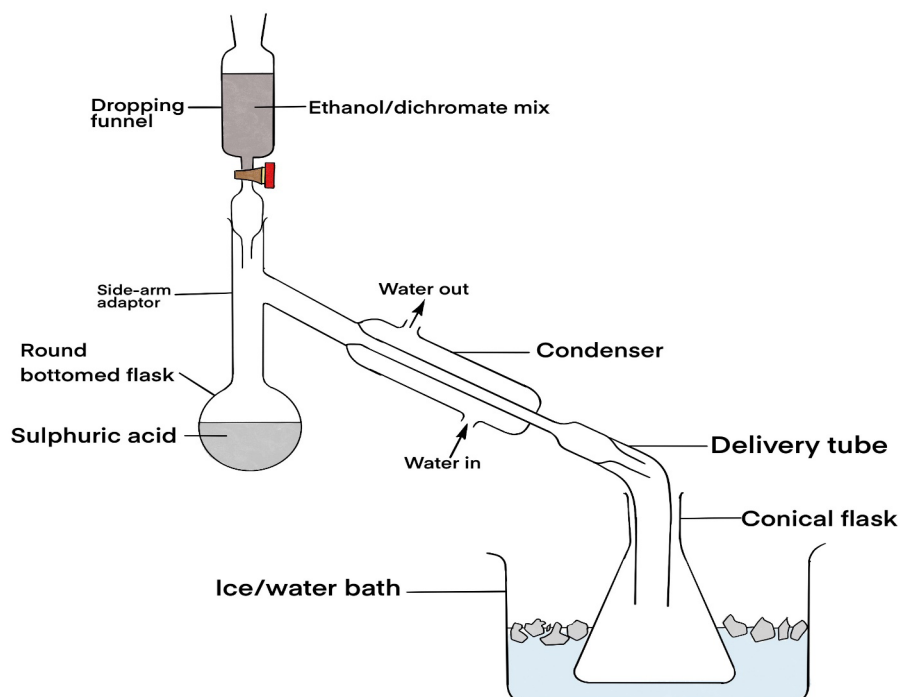


Figure 6.1: Preparation of ethanal



Procedure

1. Place 50 mL of water in a round-bottomed flask, add 17 mL of concentrated sulphuric acid and slowly swirl the content.
2. Add some anti-bumping granules to the solution
3. Dissolve 50 g of sodium dichromate (VI) in 50 mL of water contained in a small beaker.
4. Add 40 mL of ethanol into this beaker and stir thoroughly, then place this mixture in the tap funnel.
5. Heat the dilute acid in the flask until it begins to boil gently and then remove the flame
6. Run the alcohol/dichromate (VI) solution very slowly into the flask. What do you observe?

Answer: The distilled off aqueous solution smells like rotting apples and the mixture becomes green.

Interpretation of results and conclusion

Guiding questions

1. What is that compound that smells like rotting apples? Describe how ethanal is prepared in laboratory? Support your answer by providing chemical equation.
2. Which smell can help you to confirm that ethanal is obtained?
3. What are the required conditions to prevent further oxidation of ethanal to ethanoic acid?

Answer to guiding questions

Potassium dichromate (VI) acidified with dilute sulphuric acid is used as an oxidizing agent during the preparation of aldehyde or ketone. In this experiment ethanol is oxidized to ethanal. The solution of dichromate (VI) ions, $\text{Cr}_2\text{O}_7^{2-}$, is orange, during chemical reaction dichromate (VI) ions are reduced to chromium (III) ions, Cr^{3+} which is green. Further unwanted oxidation of the ethanol to ethanoic acid can be minimized by using more alcohol than oxidizing agent (i.e. the sodium dichromate is the limiting reagent) and distilling off the

ethanal as soon as it is made. The obtained ethanal is characterized by smells like rotting apples.



Evaluation

1. Briefly discuss the process of ethanal preparation from ethanol in the laboratory and give the corresponding balanced chemical equation.
2. What is likely to happen if excess sodium dichromate is used by mistake?
Answer: If excess sodium dichromate is used by mistake, further oxidation occurs which leads to the unwanted product ethanoic acid.
3. Why is ethanal collected in a vessel embedded in a beaker of ice water?
Answer: Ethanal boils at 21°C and must be kept cold to prevent losses through evaporation.

EXPERIMENT 6.3: Preparation of propanone

Rationale

Acetone, also known as propanone, is the simplest ketone. It is one of the organic substances mostly used by human beings due to its multiple industrial and domestic applications. People often use household acetone to remove stains on clothing that cannot be removed by the application of soap or regular detergents. Acetone is also commonly used as a solvent for many cosmetic products such as nail polish and nail paint. This experiment highlights the laboratory preparation method of propanone.

Objective

Learners will be able to prepare and collect propanone.



Required materials

Apparatus

- Beakers
- Condenser
- Round bottomed flask
- Bunsen burner
- Conical flask
- Thermometer

Chemicals

- Calcium acetate, $(\text{CH}_3\text{COO})_2\text{Ca}(\text{s})$

Experimental set-up

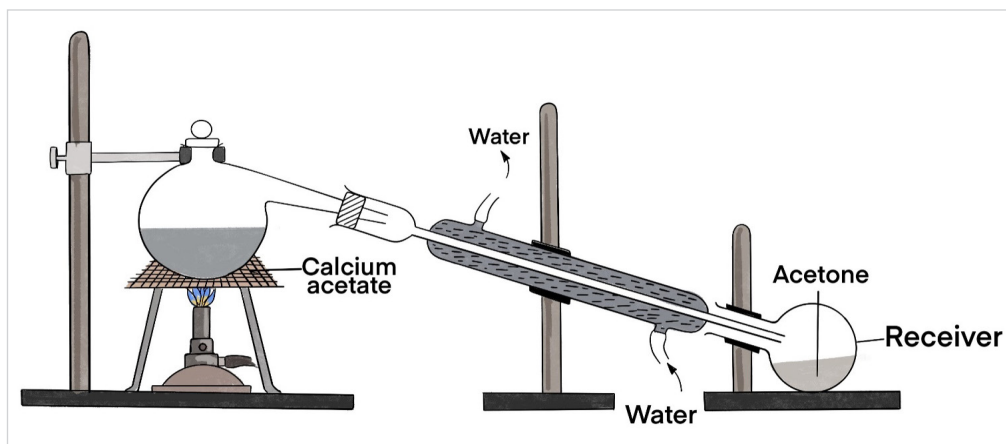


Figure 6.2: Preparation of propanone



Procedure

1. Put 15 g of calcium acetate in a 50 mL round bottom flask fixed on a retort stand.
2. Fix the apparatus as shown by the above figure 6.2.
3. Heat the flask gently. What do you observe?

Answer: Evolved water vapors are condensed to give liquid propanone in the receiver flask.

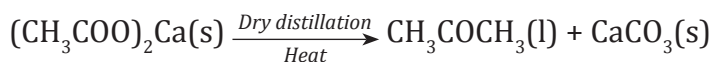
Interpretation of results and conclusion

Guiding questions

1. Describe how is propanone prepared in the laboratory from calcium acetate? Give the chemical equation of such synthesis.

Answer to guiding questions:

In the laboratory propanone is obtained by dry distillation of calcium acetate. The chemical equation of the reaction is:



Note: The obtained acetone is not pure. To purify this, it is shaken with a saturated solution of sodium bisulfite (NaHSO_3) then crystals of acetone sodium bisulfite salt separated out. The crystal is washed and heated with sodium carbonate and then dried over anhydrous CaCl_2 and then distilled at 56°C to get pure propanone.

Evaluation

1. Explain briefly how propanone is prepared in the laboratory and list chemicals that are used in propanone purification.

Answer: In the laboratory, propanone is obtained by dry distillation of calcium acetate and chemicals that are used in propanone purification are saturated solution of sodium bisulphite, sodium carbonate and anhydrous calcium chloride

EXPERIMENT 6.4: Distinction between ketones and aldehydes using Fehling's reagent or Benedict's solution

Rationale

Aldehydes and ketones are organic compounds containing a carbonyl group. These compounds are very essential in everyday life as they are used to synthesize plastics, drugs, textiles, paints removal, perfumes, cosmetics products, and dyes. This experiment will deal with identification of aldehydes, and ketones by using Fehling's reagent and Benedict's solution.

Objective

Learners will be able to distinguish between ketones and aldehydes using Fehling's or Benedict's solution.



Required materials

Apparatus

- Test tubes
- Test tubes holder
- Test tube racks
- Droppers
- Beakers
- Water bath

Chemicals

- Ethanal, CH_3CHO
- Propanone, CH_3COCH_3
- Fehling's solution
- Benedict's solution



Procedure

1. Take two clean test tubes.
2. Pour 2 mL of ethanal solution in the first test tube and 2 mL of propanone solution in the second test tube.
3. Add 6 drops of the Fehling's solution or Benedict's solution to each of the two test tubes.
4. Warm gently the mixture in a hot water bath for a few minutes. What do you observe?

Answer: The blue Fehling's solution produces a dark red/orange precipitate in the first test tube containing ethanal and no change is observed in the second test tube that contains propanone, where the Fehling's solution remains blue.

Note: Fehling's reagent is prepared as follow:

Fehling A

Dissolve 70 g of copper (II) sulphate pentahydrate in 800 mL of water and make up to 1 litre of solution; add a few drops of concentrated sulphuric acid.

Fehling B

Dissolve 350 g of sodium potassium tartrate and 100 g of sodium hydroxide in 800 mL of water. Add water to make up to 1 litre of solution.

Mix 500 mL of Fehling A with 500 mL of Fehling B.

Interpretation of results and conclusion

Guiding questions

1. What are the balanced chemical equations occurring when aldehydes react with Benedict's solution or Fehling's solution?
2. Explain briefly why aldehydes and ketones behave differently when Fehling's solution is used to test them.

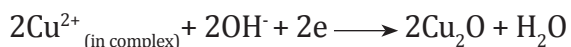
Answer to guiding questions:

Fehling's and Benedict's solution are oxidizing agents. They oxidize aldehydes to carboxylic acid. The presence of hydrogen atoms attached to the carbon-oxygen double bond in aldehydes makes them very oxidized. Ketones do not have that particular hydrogen atom, they resist to oxidation reaction.

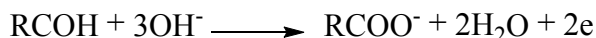
Fehling's solution is an alkaline solution containing copper (II) ions. When warmed with an aldehyde, the Cu^{2+} ions act as an oxidizing agent. The aldehyde is oxidized to a carboxylate ion while the Cu^{2+} are reduced to Cu^+ ions. The red/orange formed product is copper (I) oxide (Cu_2O).

The blue Fehling's solution produces a dark red precipitate in the test tube containing ethanal and no change observed in the test tube containing propanone. Thus, Benedict's solution/ Fehling's solution is the testing reagent to distinguish aldehydes from ketones.

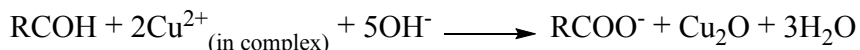
The electron-half-equations for both Fehling's and Benedict's solution can be written as:



The half-equation for the oxidation of an aldehyde under alkaline conditions is written as:



The overall equation:



Evaluation

1. List four similarities that enable Fehling's and benedict's solution to be used as testing reagents to identify aldehydes.

Answer:

- Both solutions are used to identify aldehydes.
 - Both are blue colored solutions.
 - The tests done using both solutions give a dark red/orange precipitate at the end.
 - Both tests need to heat the reaction mixture.
2. Summarize how Fehling's solution is used to distinguish ethanal from propanone.

Answer: See interpretation.

EXPERIMENT 6.5:

Distinction between ketones and aldehydes using Tollens's reagent

Rationale

Tollens' test also known as silver-mirror test is a qualitative laboratory test used to distinguish between an aldehyde and a ketone. It exploits the fact that aldehydes are readily oxidized, whereas ketones are not.

Objective

Learners will be able to distinguish between ketones and aldehydes using Tollens' reagent.



Required materials

Apparatus

- Test tubes
- Test tubes holder
- Test tube racks
- Count droppers
- Beakers
- Bunsen burner

Chemicals

- Propanal, $\text{CH}_3\text{CH}_2\text{CHO}$
- Propanone, CH_3COCH_3
- Tollens' reagent



Procedure

1. Take two clean test tubes and label them as test tube N° 1 and N° 2.
2. Add 2 mL of propanal solution in test tube N° 1 and 2 mL of propanone solution in test tube N° 2.
3. Add 6 drops of the Tollens' reagent to each of the two test tubes.
4. Warm gently the mixture in a hot water bath for a few minutes. What do you observe?

Answer: The colorless solution produces a grey precipitate of silver, or a silver mirror on the test tube containing propanal, while the colorless solution doesn't change on the test tube containing propanone (no observable change).

Note: As Tollens' reagent is not commercially available; it is hence freshly prepared in the laboratory. It is prepared as follow:

- 2 M sodium hydroxide is added to aqueous 0.2 M silver nitrate dropwise until a light brown precipitate is obtained.
- Next, 2 M ammonia solution is added dropwise until the brown precipitate of Ag_2O dissolves completely. **Never store the reagent.**

Interpretation of results and conclusion

Guidance questions

1. What is the balanced chemical equation of propanal oxidation into propanoic acid using Tollens' reagent?
2. Why doesn't propanone produce a positive result when it reacts with Tollens' reagent?

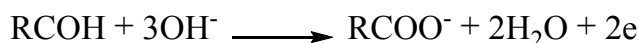
Answer to guiding questions

Aldehydes reduce the diamine silver (I) ion to metallic silver. Because the solution is alkaline, the aldehyde itself is oxidized to a salt of its corresponding carboxylic acid and the silver ions of Tollens' solution are reduced to solid silver. The latter coats the bottom of the test tube with a "silver mirror." Ketones are not oxidized, thus no silver mirror forms. The colourless solution produces a grey precipitate of silver, or a silver mirror on the test tube N° 1 containing propanal, while colourless solution doesn't change on test tube N° 2 containing propanone (no reaction). Therefore, Tollens' solution is the testing reagent to distinguish aldehydes from ketones.

The electron-half-equation for the reduction of the diamine silver (I) ions to silver is:



The half-equation for the oxidation of an aldehyde under alkaline conditions:



The overall equation:



Evaluation

1. You are given the following ketones and aldehydes: A, B, C, D, E and F. Conduct the experiment to identify which ones are ketones and aldehydes using Tollens' reagent.
2. Briefly describe the principle of Tollens' test for identification of aldehydes.

Answer:

- The Tollens' reagent is the solution of alkaline silver nitrate (AgNO_3) combined with aqueous ammonia solution (NH_4OH), leading to the development of a complex.
 - Silver nitrate in water produces a silver-aqua complex, in which water acts as a ligand.
 - Hydroxide ions convert the aqua complexes into silver oxides (Ag_2O).
 - The $[\text{Ag}(\text{NH}_3)_2]^+$ complex is formed when silver oxide develops a brown precipitate, which would be subsequently dissolved by aqueous ammonia.
 - The complex subsequently forms a carboxylic acid by oxidizing the aldehyde group in a given compound.
 - The silver ions in the reagent will be converted to metallic silver.
 - A silver mirror forms on the bottom side of a test tube as silver ions are reduced to metallic silver.
3. Why do ketones not give positive results with Tollens' test?

Answer: The main importance of Tollens' reagent is used to distinguish aldehydes and ketones. The reaction of aldehydes with Tollens' reagent is an oxidation reaction which converts aldehydes to carboxylic acid. Tollens' reagent is a mild oxidizing agent which can oxidize aldehydes but not ketones. Because aldehydes have the aldehydic-H which can be oxidized under mild oxidizing agents like Tollens' reagent but ketones do not have the aldehydic-H.

EXPERIMENT 6.6:

Distinction of methyl ketones from aldehydes and other ketones by Iodoform test

Rationale

Iodoform test is used to check the presence of carbonyl compounds with the structure R-CO-CH_3 . Iodoform test is used to test the presence of carbonyl compounds with the structure R-CO-CH_3 or alcohols with the structure R-CH(OH)-CH_3 in a given unknown substance.

Objective

Learners will be able to distinguish methyl ketones from aldehydes and other ketones by using iodoform test.



Required materials

Apparatus

- Test tubes
- Test tubes holder
- Test tube racks
- Count droppers
- Beakers
- Water bath

Chemicals

- Propanone, CH_3COCH_3
- Propanal, $\text{CH}_3\text{CH}_2\text{CHO}$
- 6M NaOH solution
- I_2 solution (or KI / I_2 solution)
- Distilled water



Procedure

1. Take two clean test tubes and label them as test tube N° 1 and N° 2.
2. Add 4 drops of propanone solution in test tube N° 1 and 4 drops of propanal solution in test tube N° 2.
3. Add 1 mL distilled water to each test tube.
4. Add 0.5 mL 6M NaOH and 0.5 mL of water to each test tube.
5. Add 6 drops of I_2 solution to each test tube.
6. If no precipitate forms immediately, warm the mixture very gently. What do you observe?

Answer: Yellow precipitate is formed in test tube No 1 containing propanone, whereas there is no change in test tube No 2 containing propanal.

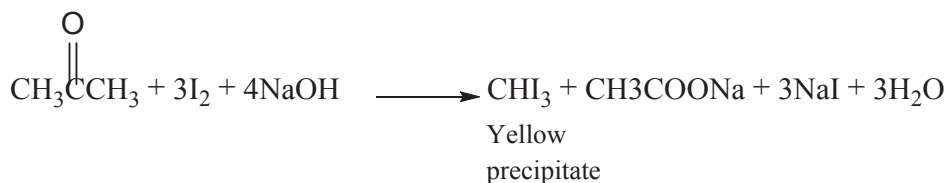
Interpretation of results and conclusion

Guidance questions

1. Why does the Iodoform test lead to a positive result for propanone while there is no observable change for propanal?
2. What is the balanced chemical equation for identifying propanone using Iodoform test?

Answer to guiding questions:

Iodoform test is used to confirm the presence of a methyl group aldehyde ($\text{CH}_3\text{C}=\text{O}$; ethanal or acetaldehyde) and ketones (ketone where CH_3- is directly attached to the carbonyl group $\text{C}=\text{O}$). In this case, a bright yellow color is observed. Generally, when methyl ketones and ethanal are treated with iodine in basic solution, hydrogens of the methyl group are replaced by iodine followed by cleavage of the methyl group. The products are the salt of carboxylic acid and triiodomethane. The reaction is fast until the 3 hydrogens at the methyl group have been replaced by iodine.



Evaluation

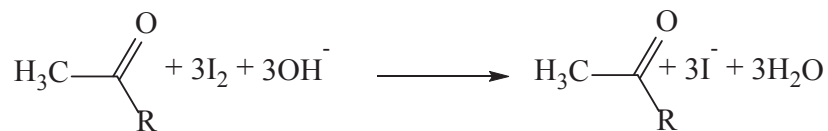
1. You are provided with the following methyl ketones, aldehydes, and other ketones: A, B, C, D, E and F.

Perform the experiment to identify which ones are methyl ketones, aldehydes and other ketones using Iodoform test.

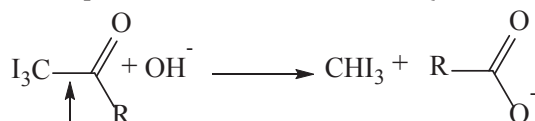
2. Explain the process iodoform formation from ketones using iodine and sodium hydroxide solution.

Answer: Iodine solution is added to a small amount of ketone, followed by just enough sodium hydroxide solution to remove the color of the iodine. If nothing happens in the cold, it may be necessary to warm the mixture very gently. A positive result is the appearance of a yellow precipitate of triiodomethane (previously known as iodoform) - CHI_3 . Apart from its color, this can be recognised by its faintly "medical" smell.

The first stage involves substitution of all three hydrogens in the methyl group by iodine atoms.

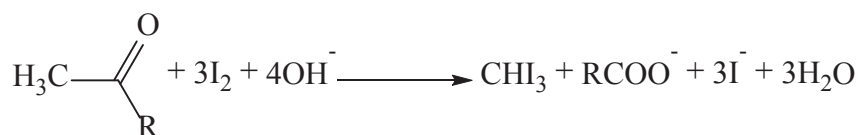


In the second stage, the bond between the CI_3 and the rest of the molecule is broken to produce triiodomethane (iodoform) and the salt of an acid.



This bond is broken

The overall chemical equation for the reaction:



3. What does an iodoform test indicate?

Answer: The iodoform test indicates the presence of a ketone in which a methyl group is one of the groups immediately bonded to the carbonyl carbon. Such a ketone is referred to as methyl ketone.

EXPERIMENT 7.1:

Experiment to distinguish the carboxylic acids from other organic compounds using sodium carbonate

Rationale

The main characteristic of the carboxylic acids is their acidity. They are generally more acidic than other organic compounds containing hydroxyl groups but are generally weaker than the familiar mineral acids (e.g., hydrochloric acid, HCl, sulfuric acid, H₂SO₄, etc.). Carboxylic acids and their derivatives are used in the production of polymers, biopolymers, coatings, adhesives, and pharmaceutical drugs. They can be also used as solvents, food additives, antimicrobials, and flavorings. This experiment distinguishes the presence of carboxylic acids by using sodium carbonate.

Objective

Learners will be able to distinguish the carboxylic acids from other organic compounds using sodium carbonate.



Required materials

Apparatus

- Test tubes
- Test tube rack
- Test tube holder

Chemicals

- Acetic acid, CH₃COOH (aq)
- Sodium carbonate, Na₂CO₃(aq)
- Ethanal
- Lime water



Procedure

1. Take two clean test tubes and label them as test tube N° 1 and N° 2.
2. Add 1 mL of acetic acid, CH_3COOH (aq) solution in test tube N° 1 and 1 mL of ethanol solution in test tube N° 2
3. To each test tube add 1 mL of sodium carbonate solution. What do you observe?

Answer: There is an effervescence within test tube N° 2 and no observable change in tube N°1.

4. Test the gas which is responsible of effervescence by using lime water solution. What do you observe?

Answer: The solution becomes milky.

Interpretation of results and conclusion

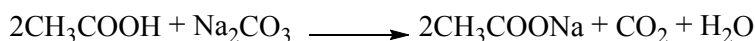
Guidance questions

1. What is the chemical test used to distinguish the carboxylic acids from other organic compounds? Justify your answer by writing the balanced chemical equations.

Answer to guiding questions

Carboxylic acids are weak acids, but generally acidic enough to react with both sodium carbonate and sodium hydrogen carbonate. Effervescence is observed due to evolved carbon dioxide gas. Therefore, ethanoic acid (acetic acid) gives positive results while ethanol does not. Thus, this test helps to distinguish carboxylic acids from other organic compounds.

The chemical equation of the reaction:



The carboxylic acid also turns the blue litmus paper red. It is an acid.

Evaluation

1. What causes effervescence in sodium bicarbonate test?

Answer: The effervescence is caused by carbon dioxide gas produced.

2. Write a general chemical equation of the reaction when any carboxylic acid reacts with sodium hydrogen carbonate (NaHCO_3).

Answer: $\text{RCOOH}(\text{aq}) + \text{NaHCO}_3(\text{aq}) \longrightarrow \text{RCOONa}(\text{aq}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})$

EXPERIMENT 7.2:

Preparation of carboxylic acid by oxidation of primary alcohol by using acidified potassium permanganate (VII)

Rationale

Carboxylic acids occur widely in nature. Vinegar is generally 5-8% acetic acid by volume. The carboxylic acids are used as food additives, to prepare sorbic acid, and benzoic acid, in elaboration of cheese and other milk products (lactic acid). Acetic acid is scientifically known as ethanoic acid and is one of the most common carboxylic acids.

Objective

Learners will be able to prepare carboxylic acid by oxidation of primary alcohols using acidified potassium permanganate (VII).



Required materials

Apparatus

- Bunsen burner
- Tripod stand
- Round bottom flask
- Borosilicate Beaker
- Condenser
- Retort stand and accessories
- Match box

Chemicals

- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$ (l)
- Acidified potassium permanganate (VII), KMnO_4
- Water

Experimental set-up

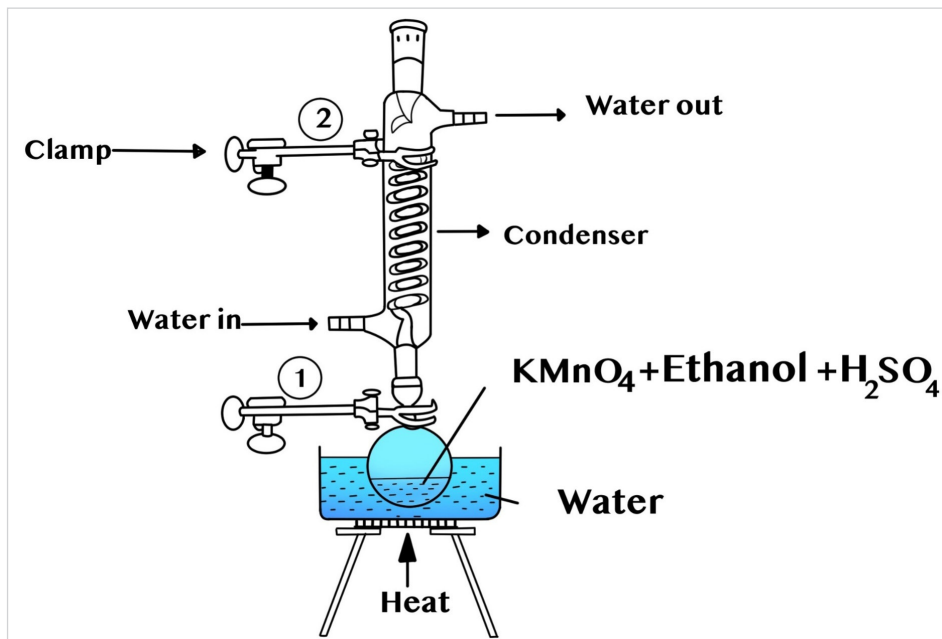


Figure 7.2: Preparation of carboxylic acid

Procedure

1. Pour 50 mL of ethanol in a 500 mL round bottom flask.
2. Add anti-bumping granules.
3. Add 85 mL of acidified potassium permanganate solution.
4. Add blue and red litmus papers in the mixture. What do you observe?

Answer: No observable change

5. Set the apparatus as indicated in the figure 7.2 above.
6. Heat the flask in water as shown in the setup. What do you observe?

Answer: The purple color of potassium permanganate (VII) turns colorless.

7. Test the colorless solution in the flask using blue and red litmus paper. What do you observe?

Answer: Blue litmus paper turns red.

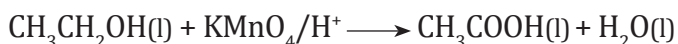
Interpretation of results and conclusion

Guiding questions

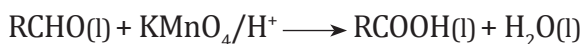
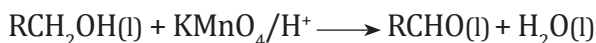
1. What happens when primary alcohol reacts with KMnO_4 ?
2. Why blue litmus paper turns red?
3. Does aldehyde oxidize to carboxylic acid?

Answer to guiding questions:

Ethanol is oxidized by permanganate (VII) solution in the presence of dilute sulphuric acid to form ethanoic acid. The produced acid is obtained from the mixture by distillation at its boiling point.

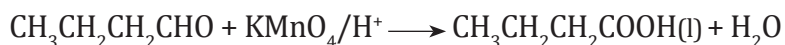


In general, carboxylic acids can be prepared by oxidation of alcohols or aldehydes using potassium permanganate (VII) solution in the presence of dilute sulphuric acid. KMnO_4 is slightly stronger oxidizing agent than $\text{K}_2\text{Cr}_2\text{O}_7$ in acidic medium due to its higher reduction potential. The reduction product is MnO_2 , observable change in color is from deep purple to a brown suspension, or even to colorless Mn^{2+} ion.



Evaluation

Write abalanced chemical equation of the reaction between butanol, butanal, and KMnO_4 in the presence of diluted sulphuric acid.



UNIT: 8

ESTERS, ACID ANHYDRIDES, AMIDES AND NITRILES

EXPERIMENT 8.1: Esterification reaction

Rationale

Natural esters are found in pheromones. Naturally occurring fats and oils are fatty acid esters of glycerol. Esters that have fragrant odors are used as a constituent of perfumes, essential oils, food flavorings, cosmetics. It is also used as an organic solvent. This experiment focusses on the laboratory preparation of esters.

Objective

Learners will be able to prepare esters.



Required materials

Apparatus

- Test tubes
- Droppers
- Beakers
- Stirring rod
- Water bath

Chemicals

- Concentrated sulphuric acid
- Sodium bicarbonate
- Ethanol
- Ethanoic acid



Procedure

1. Pour 3 mL of ethanoic acid in a test tube.
2. Add 3 mL of ethanol in the test tube.
3. Add 3 or 4 drops of concentrated sulphuric acid.
4. Heat the mixture in water bath about 2 minutes and a half.
5. Pour the mixture into the beaker containing about 20 mL of sodium bicarbonate, NaHCO_3 .
6. By wafting, test the smell of the product. What do you smell?

Answer: Fruity smell

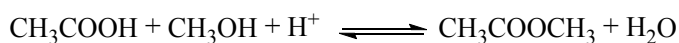
Interpretation of results and conclusion

Guidance questions

1. What is the characteristic odor of ester products?
2. Why is esterification reversible?

Answer to guiding questions:

Esters with sweet fruity smell can be prepared by a reaction between alcohols and carboxylic acids in strong acidic medium acting as a catalyst. The commonly used acid is a concentrated sulphuric acid. The reaction is reversible since ester and water can react to form the carboxylic acid and alcohol.



Ester is relatively soluble in water. Ester consists of an oxygen atom which forms hydrogen bonding with water making it slightly soluble in water. However, esters of high molar mass are insoluble in water.

Evaluation

1. Describe how are esters prepared in a laboratory?

Answer: See procedures and interpretation.

2. Write the chemical equation of esterification where propylpropanoate is prepared



EXPERIMENT 8.2: Saponification reaction

Rationale

Soap is very important to modern life. Hand soaps allow us to keep clean and remove dirt from our hands before eating. Washing powder is a type of soap used to clean clothes. Soaps save lives by stopping the spread of harmful bacteria in dirt. Soap is an emulsifier meaning it allows water and oil to mix. This allows it to remove dirt, making it a useful aid in maintaining good health every day. This experiment emphasizes on soap making.

Objective

Learners will be able to make a soap



Required materials

Apparatus

- Beakers
- Electronic balance
- Magnetic stirrer bar (if any)
- Magnetic stirrer hot plate (if any)
- Chronometer
- Stirring rod
- Ice water bath

Chemicals

- Animal or plant oil
- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$ (l)
- Sodium hydroxide, NaOH (20%)
- Saturated sodium chloride, NaCl (30 g in 100mL)

Experimental set-up

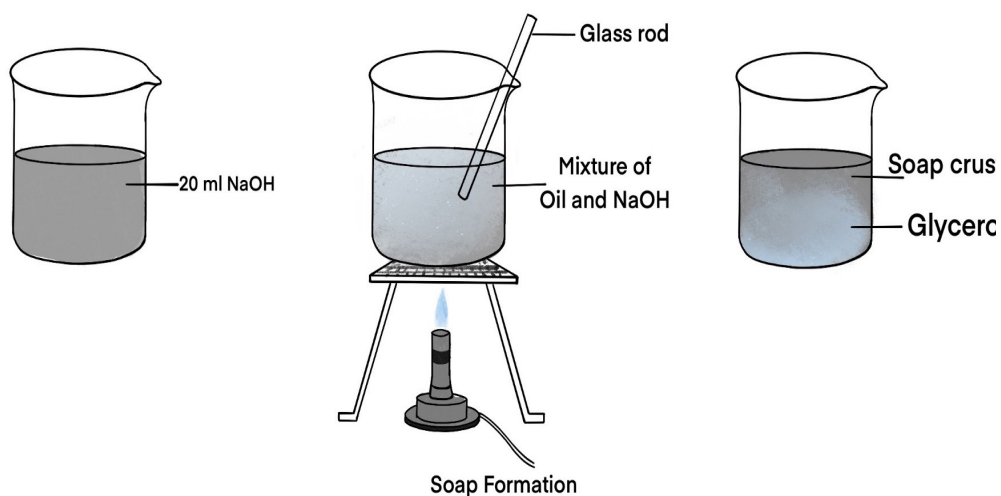


Figure 8.2: Preparation of soap in the laboratory



Procedure

1. Add about 20 mL of oil in a pyrex beaker and boil it at 45°C
2. Prepare 6M NaOH solution by dissolving 2.4 g in 10 mL of water
3. Add 10 mL of ethanol to 15 mL 6M NaOH at about 35°C
4. Pour the alkaline solution onto the beaker containing oil and stir to mix
5. Mix until thick substance is formed
6. Allow the mixture to set for 1-3 days.

Caution: Be very careful when pouring the NaOH solution, and don't let it splatter/to scatter and very hot mixture of oil and ethanol may splatter or catch fire. Wear gloves and goggles at all times because NaOH can cause permanent eye damage.

Interpretation of results and conclusion

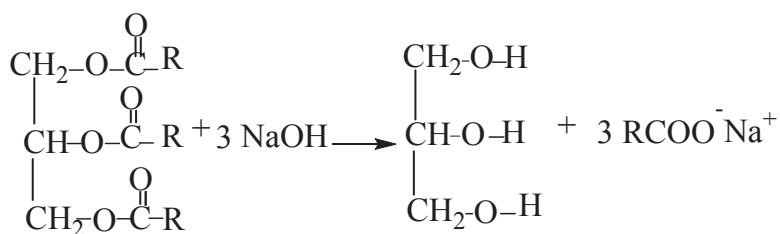
Guidance questions

1. What are the main ingredients in soap making?
2. Write down the chemical equation for soap making.

Answer to guiding questions:

Saponification is a process of converting esters into soap and alcohol by the action of aqueous alkali (for example, sodium hydroxide). The reaction requires a solution of sodium hydroxide in water and triglycerides (esters) in oil and heat. The obtained products are soap and glycerol.

The chemical equation of the saponification reaction:



Evaluation

1. Describe the process of soap making

Answer: See procedure and interpretation.

2. What type of reaction is used in making soap?

Answer: Saponification is the hydrolysis of an ester to form an alcohol and the salt of a carboxylic acid in acidic or essential conditions. Saponification is usually used to refer to the soap-forming reaction of a metallic alkali (base) with fat.

EXPERIMENT 8.3: Experiment to prepare aspirin using acetic anhydride and salicylic acid

Rationale

Aspirin also known as acetylsalicylic acid has been shown to be helpful when used daily to lower the risk of heart attack, clot-related strokes and other blood flow problems in patients who have cardiovascular disease or who have already had a heart attack or stroke. Aspirin relieving headache, reducing swelling, and reducing a fever. This experiment aims at the preparation of aspirin in laboratory.

Objective

Learners will be able to prepare aspirin using acetic anhydride and salicylic acid.



Required materials

Apparatus

- Conical flasks
- Water bath
- Filter paper
- Filter funnel
- Watch glass

Chemicals

- Salicylic acid, $C_6H_4OHCOOH(s)$
- Acetic anhydride, $CH_3COOCH_3(l)$
- Concentrated phosphoric acid, H_3PO_4 (85%)
- Distilled water



Procedure

1. Weigh accurately 2 g of salicylic acid and put it into a 250 mL conical flask.
2. Add 5.0 mL of distilled acetic anhydride and 5 drops of phosphoric acid into the flask.
3. Keep the flask in the hot water bath for 45 minutes.

- Pour 2 mL of distilled water to the flask and warm the mixture.
- Remove the flask from the water bath and bring the reaction mixture to room temperature.
- Add 20 mL of distilled water to the flask and wait for 2 minutes for decantation. What do you observe?
Answer: White crystals are formed.
- Remove the solid by filtration.
- Put the solid to watch glass and dry the crystal.

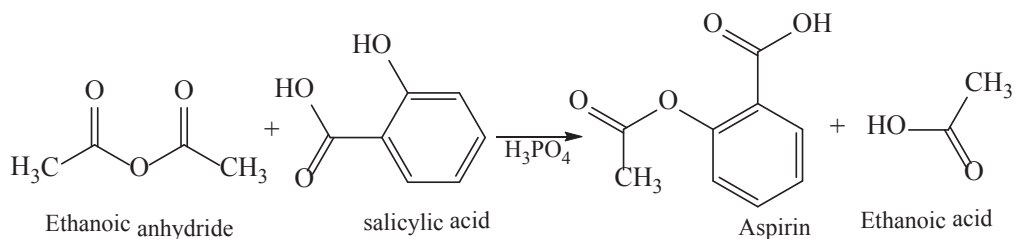
Interpretation of results and conclusion

Guidance questions

- Describe how is aspirin prepared in the laboratory?
- What is the effect of phosphoric acid in the above preparation?

Answer to guiding questions

In this experiment, salicylic acid reacts with an excess of acetic anhydride. A small amount of a strong acid, concentrated phosphoric acid is used as a catalyst which speeds up the reaction to produce aspirin. The excess acetic acid will be reduced by the addition of water. The aspirin product is not very soluble in water, thus the aspirin product will precipitate when water is added. The synthesis reaction of aspirin is shown below:



Evaluation

1. What are the reagents used in aspirin preparation?

Answer: To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction.

2. Which is the limiting reagent in the preparation of aspirin?

Answer: The salicylic acid is the limiting reagent.

3. What is the catalyst used in aspirin synthesis?

Answer: To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. In this experiment, sulfuric acid or phosphoric acid can be used as the catalyst.

EXPERIMENT 9.1

Distinction of primary, secondary, and tertiary amines in a solution

Rationale

Amines are basic organic compounds that are considered as derivatives of ammonia. They are classified into primary, secondary, and tertiary amines based on the number of groups attached to the nitrogen atom and noted as RNH_2 , R_2NH , or R_3N respectively, where R is any alkyl or aryl group. Amines are used to remove carbon dioxide (CO_2) from gases combustion, preparedyes, inhibited the corrosion in boilers, produce pharmaceuticals, as photographs agents, and emulsifiers. This experiment targets the differentiation of primary, secondary, and tertiary amines.

Objective

Learners will be able to test for the presence of the amines in a solution and distinguish primary, secondary, and tertiary amines in a solution.



Required materials

Apparatus

- Test tubes
- Water bath
- Iced bath

Chemicals

- Organic compounds to be tested (primary, secondary, and tertiary amines)
- Potassium hydroxide, KOH
- Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$
- Chloroform, CHCl_3
- Distilled water

- Sodium nitrite solution, NaNO_2
- Concentrated hydrochloric acid, HCl
- Sodium hydroxide, NaOH
- Litmus paper



Procedure

1. Take three clean test tubes and label them as test tube N° 1, N° 2 and N° 3.
2. Dissolve primary amine in the test tube N° 1, the secondary amine in the test tube N° 2 and the tertiary amine in the test tube N° 3 in concentrated hydrochloric acid.
3. Cool the solution in iced bath at $0-5^\circ\text{C}$.
4. Add sodium nitrite dropwise to the cooled solution and mix well.
5. Test the solution by litmus paper for the presence of free nitrous acid.
6. Add excess of nitrous acid solution if there is an insufficient amount of nitrous acid. What do you observe?

Answer: Appearance of bubbles (Nitrogen gas) in test tube N° 1 indicates the presence of primary amines; yellow oily liquid in test tube N° 2 proves the presence of secondary amines and formation of nitrite salts in the test tube N° 3 confirms the presence of tertiary amines.

Interpretation of results and conclusion

Guidance questions

1. Explain how primary, secondary and tertiary amines can be differentiated in the laboratory?

Answer to guiding questions:

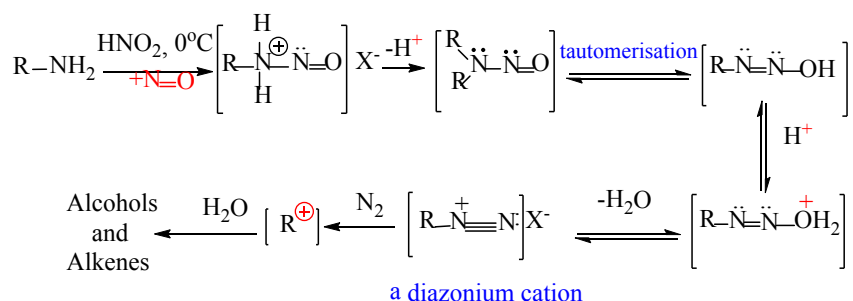
Amines like ammonia are bases. Being basic in nature, they turn red litmus paper to blue, and react with acids to form salts.



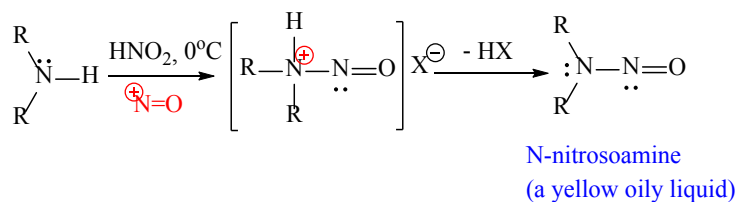
Primary aliphatic amines react with nitrous acid to produce a very unstable diazonium salt which spontaneously decomposes by losing nitrogen gas (N_2).

Nitrous acid in cold acidic solution reacts with amines to produce nitrogen gas, an insoluble oil (N-nitrosamine) and a clear solution (Ammonium salt)

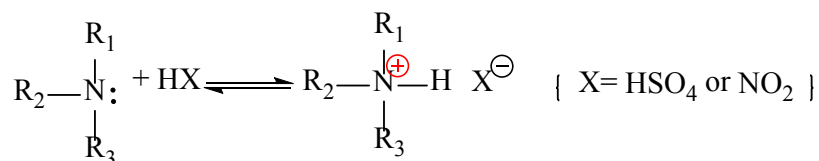
Primary amine



Secondary amine



Tertiary amine



Evaluation

1. How does methyl amine behave with litmus paper?

Answer: Methyl amine changes red litmus blue, this litmus paper test shows basic nature of methyl amine

2. Arrange the following compounds in increasing order of basicity strengths in their aqueous solutions: NH_3 , CH_3NH_2 , $(\text{CH}_3)_2\text{NH}$, $(\text{CH}_3)_3\text{N}$

Answer: Basicity order (due to stability of ammonium cation) $(\text{CH}_3)_2\text{NH} > \text{CH}_3\text{NH}_2 > (\text{CH}_3)_3\text{N} > \text{NH}_3$

3. State the reason why primary amines have higher boiling points than tertiary amines.

Answer: Primary amines undergo extensive intermolecular H-bonding due to the presence of two H-atoms on the N-atom, whereas tertiary amines do not undergo H-bonding due to the absence of an H-atom on the N-atom. As a result, primary amines have greater boiling points than tertiary amines.

EXPERIMENT 9.2

Experiment to test for the presence of amino acid in a solution

Rationale

Amino acids are the basic units of proteins and the primary substance supporting biological life activities. Amino acids have many special physiological functions, playing roles in protein synthesis, metabolism, body development, osmotic pressure stability, and neurotransmission. In addition to these functions, amino acids are widely used in the food industry. This experiment helps to test the presence of amino acid in a solution.

Objective

Learners will be able to test for the presence of amino acid in a solution.



Required materials

Apparatus

- Test tubes
- Water bath

Chemicals

- Amino acid to be tested
- Ninhydrin (0.2%)



Procedure

1. Put 1 mL amino acid solution in a test tube.
2. Add 5 drops of 0.2% ninhydrin solution in acetone.
3. Boil over a water bath for 2 minutes. What do you observe?

Answer: A deep blue/violet color is observed.

Caution: Ninhydrin is a strong oxidizing agent. It should be handled with care and avoid its contact with skin or eyes. Wear gloves and mask and hood is a must.

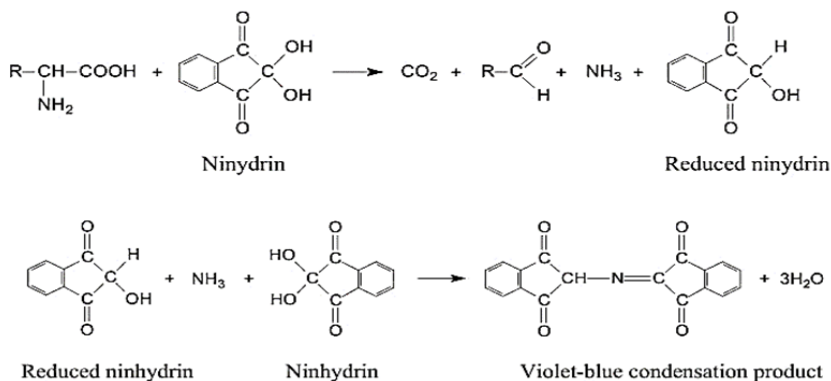
Interpretation of results and conclusion

Guidance questions

1. How do you confirm the presence of an amino acid in a given solution?
2. What is the color change to confirm the present of amino acid in solution by using Ninhydrin reagent?

Answer to guiding questions

Amino acids also react with ninhydrin at pH=4. The product obtained from an amino acid and ninhydrin excess (triketohydrindene hydrate) is the reduced ninhydrin which further reacts with another molecule of ninhydrin to yield a blue colored substance. This chemical reaction provides an extremely sensitive test for amino acids.



Evaluation

Explain how you can test in a laboratory the presence of amino acid in a given solution.

Answer: See interpretation

UNIT: 11

SOLUTIONS AND TITRATION

EXPERIMENT 11.1

Preparation of solutions of different concentrations

Rationale

In chemistry when the concentration of a solution is accurately known it is called a standard solution. Standard solutions are needed for carrying out chemical experiments in the laboratory. Standard solutions are used in analytical chemistry to determine the unknown concentrations of substances such as solutions in titration. Furthermore, they are needed to calibrate the accuracy and precision of chemical monitoring instruments. They have several scientific, medical and industrial applications (quality control and environmental safety). This experiment provides the guidance for preparing the solutions of different concentrations.

Objective

Learners will be able to prepare standard solutions.



Required materials

Apparatus

- Electronic balance
- Stirring rod
- Erlenmeyer
- Graduated cylinder
- Volumetric flask
- Pipette and dropper
- Filter funnel
- Beakers
- Labels

Chemicals

- Solid sodium carbonate, Na_2CO_3 (s)
- Sulfuric acid, H_2SO_4 (98%, $d=1.84\text{g/mL}$)
- Distilled water

Caution: Always add acid to water, never add water to acid



Procedure

A. Preparation of 250 mL of 0.5M of sodium carbonate solution

Number of moles = $C \times V$

Calculation of number of moles in 250 mL of Na_2CO_3 $0.5 \text{ M} = 0.5 \times 250 \times 10^{-3} = 0.125$

Mass = Molar mass \times moles

Mass of Na_2CO_3 in 250 mL = $0.125 \text{ mol} \times 106 \text{ g/mol} = 13.25 \text{ g}$

1. Weight 13.25 g of Na_2CO_3 using a balance.
2. Put the weighted Na_2CO_3 into a beaker.
3. Add about 50 mL of distilled water, stir with a glass rod until all solids are dissolved.
4. Pour all the solution carefully through a funnel into a volumetric flask.
5. Wash the beaker and the glass rod with distilled water, then transfer the washings into the volumetric flask.
6. Add distilled water carefully using a glass tube nearly to 250 mL mark, and then adjust the final volume using a dropper.
7. Label the solution. On the label indicate the name of the solution, the concentration of the solution and the date that the solution was prepared.

B. Preparation of 250 mL 1M of sulphuric acid

Calculations involved in preparation of 250 mL of 1M H_2SO_4 from concentrated H_2SO_4 (98% and density of 1.835 g/mL)

Calculate the mass of H_2SO_4 contained in one liter: $1.835 \text{ g/mL} \times 1000 \text{ mL} = 1835 \text{ g}$

Calculate the mass of 98% concentrated H_2SO_4 in one liter: $1798.3 \text{ g} / (98 \text{ g/mol}) = 18.35 \text{ mol}$

Calculate the number of moles of H_2SO_4 in one liter: $1798.3 \text{ g} / (98 \text{ g/mol}) = 18.35 \text{ mol}$

The molarity of concentrated sulfuric acid is 18.35M

The following chemical formula can also be used to determine the molar concentration of the stock solution of sulphuric acid:

$$\text{Molarity} = \frac{n}{V}$$

where D: Density

P: Percentage

Mm: Molar mass

Molar concentration of the stock solution of sulphuric acid:

$$\text{Molarity} = \frac{1.835 \times 1000 \times 0.98}{98} = 18.35\text{M}$$

The volume of the concentrated sulphuric acid needed (V_1) is calculated by using dilution formula: $M_1V_1 = M_2V_2$

Then $18.35 \text{ M} \times V_1 = 1\text{M} \times 250 \text{ mL}$

$$V_1 = 13.6 \text{ mL}$$

1. Pour 150 mL of water into a 250 mL volumetric flask.
2. Add 13.6 mL of concentrated sulphuric acid carefully and shake the solution.
3. Add distilled water carefully using a glass tube nearly to 250 mL mark, and then adjust the final volume using a dropper.
4. Label the solution. On the label indicate the name of the solution, the concentration of the solution and the date that the solution was prepared.

Evaluation

1. a. Describe the preparation of 500 mL of a 0.5 M of sodium chloride solution.
b. What volume of 0.5 M sodium chloride solution is needed to prepare 100 mL of 0.1 M sodium chloride solution.
2. Describe the preparation of 500 mL of 2 M ammonia from 33% concentrated ammonia with density 0.88 g/mL
3. Give a description, including calculations involved for the preparation of 250 mL of 1 M HNO_3 from 70% concentrated nitric acid with specific gravity 1.42

EXPERIMENT 11.2:

Titration of sodium hydroxide solution by standard solution of hydrochloric acid

Rationale

Titration is a quantitative chemical analysis used in a laboratory to determine the unknown concentration of an analyte. Titration finds many applications in the food industry, medicine, wastewater, and acid rain analysis, among others. In this experiment, the molarity of NaOH is determined by titrating a certain volume of HCl.

Objective

Learners will be able to determine the unknown concentration of NaOH solution by titrating it with HCl.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (50 mL)
- Conical flask (100 mL)
- Beaker (100mL)
- Retort stand
- Funnel
- Glazed tile (white)
- Measuring cylinder (100 mL)
- Volumetric flask (1 L)
- Volumetric flask (500 mL)

Chemicals

- Hydrochloric acid, HCl(s)
- Sodium hydroxide solution, NaOH(s)
- Phenolphthalein indicator

Experimental set-up

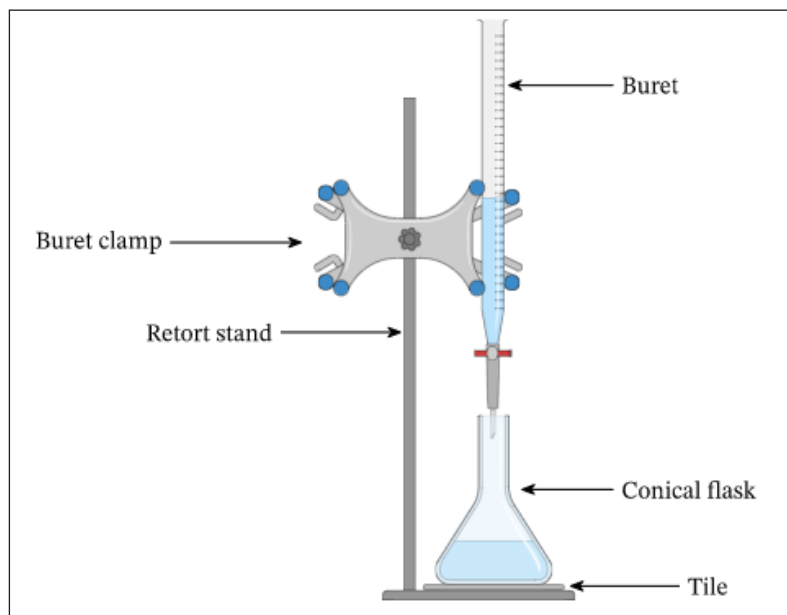


Figure 11.2: *Titration of sodium hydroxide solution by standard solution of hydrochloric acid*



Procedure

1. Prepare 250 mL of 0.2M standard solution of hydrochloric acid.
2. Dissolve 2.4 g of solid sodium hydroxide in 500 mL distilled water and stir.
3. Clean the burette thoroughly, wash it with distilled water.
4. Clamp the burette to the retort stand and place the small funnel on top.
5. Make sure the burette is closed, then carefully add the hydrochloric acid to the burette until the burette is full.
6. Place the clean beaker below the burette and carefully let some of the hydrochloric acid solution run into it. This will ensure that there are no air bubbles in the burette.
7. Pipette out 25 mL of sodium hydroxide solution in a washed and dried conical flask.

8. Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as shown in the experiment set up above.
9. Do a rough titration experiment by adding the hydrochloric acid to the conical flask dropwise while shaking. Stop as soon as the colour of the solution changes (end point). What do you observe?

Answer: Colourless/clear solution.

10. Repeat the experiment starting from step 3 to 9 at least 2 times.

Note: For accuracy you should repeat this experiment until you have two readings with a difference of no more than 0.1 mL.

Data recording

Trials	1	2	3
Final burette reading	22.60	22.90	42.80
Initial burette reading	0.00	0.00	20.00
Volume of hydrochloric acid	22.60	22.90	22.80

Notice: Burette readings should be written to two decimal places (for burette having precision up to hundredth)

Interpretation of results and conclusion

Guiding questions

1. What was the color of sodium hydroxide solution when the phenolphthalein was added?
2. What was the color when enough acid was added?

Answer to guiding questions

When phenolphthalein is added to a solution of sodium hydroxide, there is a change of color from colorless to pink. When hydrochloric acid solution is added, sodium hydroxide is completely reacted and the pink color of phenolphthalein fades until the solution becomes colorless. This means that the equivalence point is reached.

The average volume used to reach the equivalence point is 22.85 mL.

Note: The average should be calculated using values that differ by not more than ± 0.10 mL.

Calculations:

Average volume = 22.85 mL

The equation of reaction is



Calculating the number of moles of HCl used in the titration;

$$\text{Molarity} = \frac{n}{V}$$

$n = \text{Molarity} \times \text{Volume}$

$$n = 0.2 \text{ mol/L} \times 22.85 \times 10^{-3} \text{ L}$$

$$= 0.00457 \text{ moles of HCl}$$

From a balanced equation, one mole of NaOH is needed to neutralize one mole of HCl. The amount of HCl in the titration must be 3×10^{-3} moles in 25 mL of NaOH.

Molarity of NaOH is calculated as $\frac{0.00457}{0.025} = 0.182\text{M}$

Evaluation

1. Vinegar is a solution of acetic acid (CH_3COOH) in water. For quality control purposes, it can be titrated using sodium hydroxide to assure a specific percentage composition. If 25.00 mL of acetic acid is titrated with 9.08 mL of a standardized 2.293 M sodium hydroxide solution, what is the molarity of the vinegar?

EXPERIMENT 11.3:

Determination of moles of water of crystallization in oxalic acid by titration

Rationale

Oxalic acid is the simplest dicarboxylic acid with a white crystalline form which turns colorless in water. It is used as bleach, metal polish, mineral processing mechanisms. This experiment deals with the determination of the unknown moles of water of crystallization in oxalic acid by titration.

Objective

Learners will be able to determine the number of moles of water of crystallization in a given sample by titration.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (10 mL/25mL)
- Conical flask (100 mL)
- Burette stand
- Funnel
- Glazed tile (white)
- Measuring flask (100 mL)

Chemicals

- Hydrated oxalic acid, $(\text{COOH})_2 \cdot n\text{H}_2\text{O}(\text{s})$
- Sodium hydroxide solution, $\text{NaOH}(\text{aq})$
- Phenolphthalein indicator

Experimental set-up

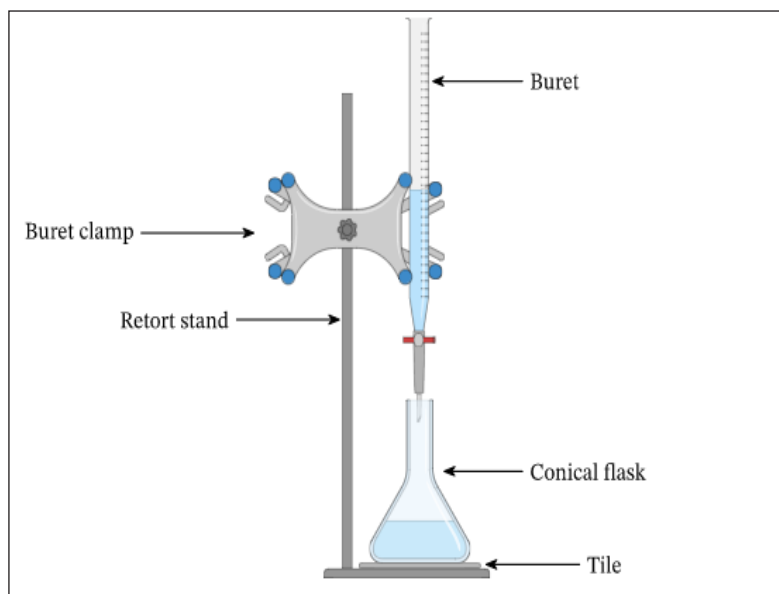


Figure 11.3: Determination of moles of water of crystallization of oxalic acid by titration



Procedure

1. Prepare 250 mL of 0.1M standard solution of sodium hydroxide.
2. In a volumetric flask, dissolve 1.6 g of oxalic acid by using distilled water to give the final volume of 250 mL.
3. Clamp the burette to the retort stand, and place the small funnel on top.
4. Make sure the burette is closed, carefully add the sodium hydroxide standard solution to the burette until the burette is full.
5. Pipette out 15 mL of oxalic acid solution in a washed, and dried conical flask.
6. Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as shown in figure 11.3. What do you observe?

Answer: Phenolphthalein remains colorless.

7. Add sodium hydroxide solution to the conical flask dropwise while shaking. Stop as soon as the colour of the solution changes (End point). What do you observe?

Answer: Phenolphthalein turns pink.

Note: For accuracy you should repeat this experiment until you have two readings with a difference of no more than 0.1 mL.

Data recording

Titration	Titration 1	Titration 2	Titration 3
Final burette reading	15.06	30.07	45.12
Initial burette reading	0.00	15.06	30.07
Volume of hydrochloric acid	15.06	15.01	15.05

Notice: Burette readings should be written to two decimal places (for burette having precision up to hundredth)

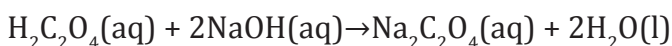
Interpretation of results and conclusion

Calculations:

Title values used for calculating average volume of NaOH are 15.06 mL and 15.05 mL

$$\text{Average value of NaOH} = \frac{(15.06 + 15.05)}{2} = 15.055 \text{ mL}$$

The neutralization occurs by the following reaction.



From the chemical equation of reaction, mole ratio of $(\text{COOH})_2 \cdot n \text{H}_2\text{O} / \text{NaOH} = 1:2$

$$\text{Moles of NaOH} = \frac{0.1 \times 15.055}{1000} = 0.0015055 \text{ moles}$$

$$\text{Moles of oxalic acid reacted in 15 mL by from the balanced chemical equation} \\ = \frac{0.0015055}{2} = 0.0007527$$

$$\text{Moles of acid in 1000 mL} = \frac{0.0007527 \times 1000}{15} = 0.05 \text{ moles}$$

The molarity of the acid is 0.05 M

$$\text{Concentration in g/L of } (\text{COOH})_2 \cdot n \text{H}_2\text{O} = \frac{(1.6 \times 1000)}{250} = 6.4 \text{ g / mol}$$

$$\text{The molecular mass of the acid} = \frac{\text{concentration in g/l}}{\text{molarity}}$$

$$\frac{6.4 \text{ g/mole}}{0.05 \text{ mol/l}} = 128 \text{ g/mole}$$

$$\text{Molecular mass} = 128 \text{ g/mole}$$

$$\text{Molecular mass of } \text{C}_2\text{H}_2\text{O}_4 \cdot n\text{H}_2\text{O} = 128$$

$$\text{That is: } (2 \times 12) + (2 \times 1) + (4 \times 16) + n((2 \times 1) + 16) = 128$$

$$90 + 18n = 128$$

$$18n = 128 - 90 = 38$$

$$n = 38/18 = 2.1 \approx 2$$

Formula of the hydrated oxalic acid is $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$

Evaluation

Sodium carbonate crystals (27.8230 g) were dissolved in water and made up to 1.00 dm³. 25 mL⁻¹ of the solution were neutralized by 48.8 mL of hydrochloric acid of concentration 0.100 molL⁻¹. Find n in the formula Na₂CO₃·nH₂O.

EXPERIMENT 11.4:

Determination of the percentage purity of an impure sample

Rationale

Pure substances are made up of a singular type of atom or a singular grouping of molecules. Impurities are any components not defined as active substances, they arise from the sources of starting materials and their contaminants. The determination of the purity of a substance is one of the many applications of titrations. The importance of purity in chemistry is to bring assured quality to life. The purity percentage determination is widely used in food processing, drug analysis, water quality assessment, mineral processing. In this experiment, the percentage purity of an impure sample of sodium hydroxide is estimated by titration.

Objective

Learners will be able to determine the percentage of purity of an impure sample.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (50 mL)
- Conical flask (100 mL)
- Burette stand
- Funnel
- Glazed tile (white)
- Measuring flask (100 mL)

Chemicals

- Impure sodium hydroxide, NaOH(s)
- Phenolphthalein indicator
- Hydrochloric acid, HCl(aq)
- Distilled water

Experimental set-up

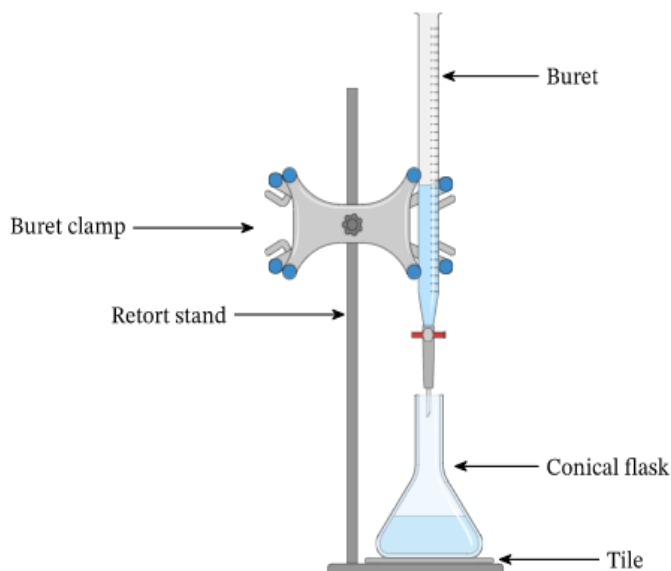


Figure 11.4: Determination of the percentage of purity of an impure sample

Procedure

1. Dissolve 5 g of impure sodium hydroxide in distilled water to make 1L solution.
2. Prepare 250 mL of 0.1M standard solution of hydrochloric acid.
3. Clamp the burette to the retort stand and place the small funnel on top.
4. Make sure the burette is closed, carefully fill the burette with hydrochloric acid standard solution.
5. Pipette out 25 mL of impure sodium hydroxide solution in a washed, and dried conical flask.
6. Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as indicated in figure 11.4. What do you observe?

Answer: Colorless phenolphthalein turns pink.

7. Add the hydrochloric acid solution to the conical flask dropwise while shaking. Stop as soon as the colour of the solution changes (end point). What do you observe?

Answer: Pink phenolphthalein turns colorless.

8. Repeat the experiment at least two times.

Note: For accuracy you should repeat this step until you have at least two readings with a difference of no more than 0.1mL.

Interpretation of results and conclusion

Data recording:

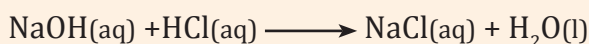
Trials	1	2	3
Final burette reading/mL	25.00	24.56	25.00
Initial burette reading/mL	0.00	0.00	0.00
Volume of hydrochloric acid/mL	25.00	24.56	25.00

Note: Burette readings should be written to two decimal places (for burette having precision up to hundredths)

Average titre should be obtained using values which differ not by more $\pm 0.10\text{mL}$.

$$\frac{25 + 25}{2} = 25$$

The chemical equation of reaction:



$$\text{Moles of HCl reacted} = \frac{0.1 \times 25}{1000} = 0.0025 \text{ moles}$$

Mole ratio is 1:1, thus $n \text{ HCl} = n \text{ NaOH}$

$$\text{Moles of NaOH in 25mL} = 0.0025 \text{ mol}$$

$$\text{Moles of NaOH in 1000mL} = \frac{0.0025 \times 1000}{25} = 0.1 \text{ moles}$$

$$\text{Mass of NaOH} = 0.1 \text{ moles} \times 40 \text{ gram/mol} = 4 \text{ grams}$$

$$\text{Mass of pure NaOH} = 4 \text{ g}$$

$$\text{Percentage purity} = \frac{4 \times 100}{5} = 80 \%$$

Evaluation

1. A sample of lead (II) bromide was made and weighed 15 g. After titration, the sample was found to be impure and only contained 13.5 g of lead (II) bromide. Calculate the percentage purity of the lead (II) bromide.
2. A 1.62 g sample of impure sodium carbonate was dissolved in distilled water, and then made up to 250 mL. 25.0 mL of this solution was put into a conical flask and three drops of methyl orange indicator added. This was titrated with a 0.105 mol/L solution of hydrochloric acid until the end point was reached. The titration was repeated three more times. The results are presented in the table below.

Trials	1	2	3	4
Final burette reading/mL	25.25	25.30	25.85	25.95
Initial burette reading/mL	0.00	0.50	0.75	1.25
Volume/mL	25.25	24.80	25.10	24.70

The chemical equation of the reaction is: $\text{Na}_2\text{CO}_3 + 2\text{HCl} \rightarrow 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2$

- a) Describe the colour change that tells when the end point has been reached.
- b)
 - (i) Select the appropriate volumes and calculate their average.
 - (ii) Calculate the amount (in moles) of hydrochloric acid solution in the average volume.
 - (iii) Calculate the amount (in moles) of pure sodium carbonate in 25.0 mL of solution.
 - (iv) Calculate the amount (in moles) of pure sodium carbonate in 250 mL of solution.
 - (v) Calculate the mass of pure sodium carbonate, Na_2CO_3 , taken.
 - (vi) Calculate the percentage purity of the sample of sodium carbonate.

EXPERIMENT 11.5:

Determination of the relative atomic mass of the metal M in a metal hydrogen sulphate ($MHSO_4$)

Rationale

Relative atomic mass, also known as atomic weight, is a dimensionless physical quantity defined as the ratio of the average mass of atoms of a chemical element in a given sample to the atomic mass constant. The determination of relative atomic mass helps to identify the unknown element in a given compound. Knowing the relative atomic mass of an element in a given compound can also help to determine the amount of that compound which can be involved in a given reaction. This method is more helpful as it implies the determination of the amount of a metal element in a given drug, fertilizer, soil, water, oil, paint. This experiment demonstrates how to calculate the relative atomic mass of a metal in a metal hydrogen sulphate by titration.

Objective

Learners will be able to determine the relative atomic mass of the metal M in a metal hydrogen sulphate ($MHSO_4$) by titration.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (50 mL)
- Conical flasks (100 mL)
- Burette stands
- Funnel
- Glazed tile (white)
- Measuring flask (100 mL)

Chemicals

- Potassium hydrogen sulphate, $KHSO_4(s)$ ($MHSO_4$)
- Phenolphthalein indicator
- Sodium hydroxide, $NaOH(aq)$
- Distilled water

Experimental set-up

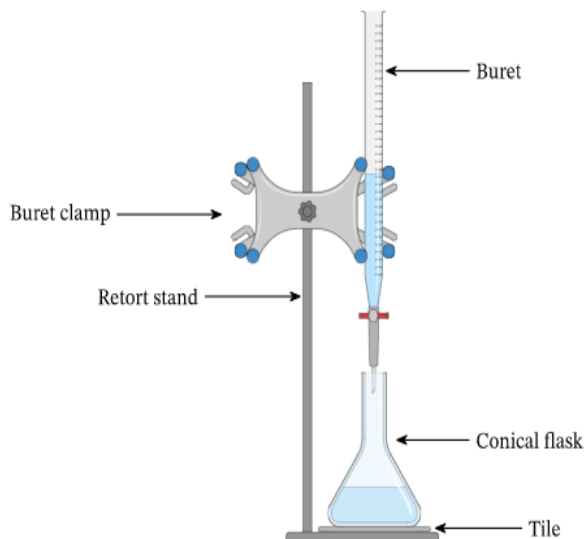


Figure 11.5: Determination of the relative atomic mass of the metal M in a metal hydrogen sulphate ($MHSO_4$)



Procedure

In a volumetric flask, dissolve 3.4 g of metal hydrogen sulphate in distilled water and make up the solution to 250 mL.

1. Prepare 250 mL of 0.1M standard solution of sodium hydroxide.
2. Clamp the burette to the retort stand, and place the small funnel on top. Fill the burette with sodium hydroxide standard solution.
3. Pipette out 20 mL of the metal hydrogen sulphate solution in a washed, and dried conical flask.
4. Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as demonstrated in figure 11.5. What do you observe?

Answer: Phenolphthalein remains colorless.

5. Add sodium hydroxide solution to the conical flask dropwise while shaking. Stop as soon as the colour of the solution changes (end point). What do you observe?

Answer: Phenolphthalein turns pink.

6. Repeat the titration at least three times.

Note: For accuracy you should repeat this step until you have three readings with a difference of no more than 0.1 mL.

Interpretation of results and conclusion

Data recording

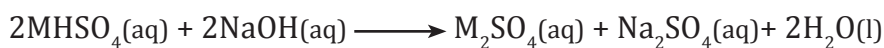
Trial	1	2	3
Final burette reading/mL	20.50	20.51	20.49
Initial burette reading/mL	0.00	0.00	0.00
Volume of Sodium hydroxide used/ mL	20.50	20.51	20.49

Notice: Burette readings should be written to two decimal places (for burette having precision up to hundredth)

Average titre should be obtained using values which differ not by more than ± 0.10 mL.

$$\frac{20.51+20.50+20.49}{3} = 20.50$$

The chemical equation of reaction:



From the chemical equation of reaction, the mole ratio of M_2SO_4 : NaOH is 1:1

$$\text{Moles of NaOH reacted} = \frac{0.1 \times 20.50}{20} = 2.05 \times 10^{-3} \text{ moles}$$

$$\text{Moles of MHSO}_4 \text{ in } 1000\text{mL} = \frac{0.00205 \times 1000}{20} = 0.1 \text{ moles/L}$$

$$\text{The concentration of MHSO}_4 \text{ (g/L)} = \frac{3.4 \times 1000}{250} = 13.6$$

$$\text{Molar mass of MSO}_4 = \frac{13.6}{0.1} = 136 \text{ g/mol}$$

$$136 = M + 1 + 32 + (16 \times 4)$$

$$136 = M + 1 + 32 + 64$$

$$M = 136 - 97$$

$$M = 39$$

Therefore, the relative atomic mass of the metal M is 39. Thus, the element is Potassium (K)

Evaluation

- 1.7 g of a sample of a metal sulphate ($MHSO_4$) was dissolved in distilled water and the volume was made to 250 mL. 20 mL of the solution was titrated using 0.05M sodium hydroxide. The average volume used to get a complete reaction of the metal sulphate is 20 mL. Calculate the relative atomic mass M.

EXPERIMENT 11.6:

Back titration of magnesium oxide

Rationale

A back titration also known as indirect titration is a titration method used to determine the concentration of an analyte by reacting it with a known amount of excess reagent. The remaining reagent is then titrated using another reagent. The second titration's results indicate how much of the excess reagent is used in the first titration, hence enabling the concentration of the original analyte to be determined. Back titrations are suitable when insoluble or volatile substances are analysed. This experiment demonstrates how to calculate the percentage purity of magnesium oxide using a back titration method.

Objective

Learners will be able to determine the percentage purity of magnesium oxide using a back titration.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (25 mL)
- Conical flask (100 mL)
- Burette stand
- Funnel
- Glazed tile (white)
- Measuring flask (100 mL)

Chemicals

- Magnesium oxide(s)
- Hydrochloric acid (1M)
- Sodium hydroxide solution, NaOH (0.2M)
- Phenolphthalein indicator
- Distilled water



Procedure

1. Weigh 2.0 g of magnesium oxide and transfer it in a 250 mL conical flask.
2. Add 100 mL of a 1M HCl to the conical flask containing magnesium oxide. What do you observe?
Answer: Magnesium oxide is dissolved.
3. Fill the burette with 0.2M sodium hydroxide.
4. Add 2-3 drops of phenolphthalein to the content of the conical flask.
5. Titrate the content of the conical flask using 0.2M sodium hydroxide. What do you observe?
Answer: phenolphthalein turns pink when the reaction is complete.
6. Repeat the experiment at least two times.

Data recording

Trials	1	2	3
Final burette reading/mL	13.90	27.85	41.80
Initial burette reading/mL	0.00	14.00	28.00
Volume of Sodium hydroxide used /mL	13.90	13.85	13.80

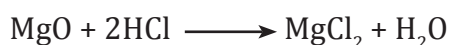
$$\text{Average volume: } \frac{13.85+13.80}{2} = 13.825$$

Interpretation of results and conclusion

Guiding questions

Write balanced equations of the reactions involved in this titration.

Answer to guiding questions



Number of moles of HCl added to the magnesium oxide = $5 \text{ mol/L} \times 0.100 \text{ L} = 0.5 \text{ mol}$

Number of moles of NaOH used to titrate excess HCl = $0.2 \text{ mol/L} \times 0.0138 \text{ L} = 0.00276 \text{ mol}$

Number of moles of excess HCl titrated = 0.00276 mol

Number of moles of HCl reacting with MgO = $0.5 \text{ mol} - 0.00276 \text{ mol} = 0.049724 \text{ mol}$

Number of moles of MgO reacted = $0.049724 \text{ mol} / 2 = 0.02486 \text{ mol}$

Mass of MgO reacting with the acid = $0.02486 \text{ mol} \times 40 \text{ g/mol} = 0.99448 \text{ g}$

$$\% \text{ purity of MgO} = \frac{0.99448 \text{ g}}{2.00 \text{ g}} \times 100\% = 49.72\%$$

Evaluation

1. A student was asked to determine the mass, in grams of calcium carbonate present in 0.134 g sample of chalk. The student placed the chalk sample in a 250 mL conical flask and added 49.5 mL of 0.2 mol/l hydrochloric acid using a pipette. The excess hydrochloric acid was then titrated with 0.250 mol/l sodium hydroxide. The average sodium hydroxide used was

found to be 32.12 mL. Calculate the mass of calcium carbonate in grams present in the chalk sample.

EXPERIMENT 11.7:

Redox titration using potassium permanganate

Rationale

Redox titration refers to a laboratory method of determining the concentration of a given analyte by carrying out a redox reaction between the titrant and the analyte. Redox titrations often require the use of a redox indicator or a potentiometer. It is one of the most common laboratory methods to identify the concentration of unknown analytes.

This experiment demonstrates how to calculate the concentration of Potassium permanganate using redox titration method.

Objective

Learners will be able to determine the average molarity of the potassium permanganate by titrating sodium oxalate solution of known molarity.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (50 mL)
- Conical flasks (100 mL)
- Burette stand
- Funnel
- Glazed tile (white)
- Measuring flask (100 mL)

Chemicals

- Sulfuric acid, H_2SO_4
- Sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$
- Potassium permanganate, KMnO_4

Experimental set-up

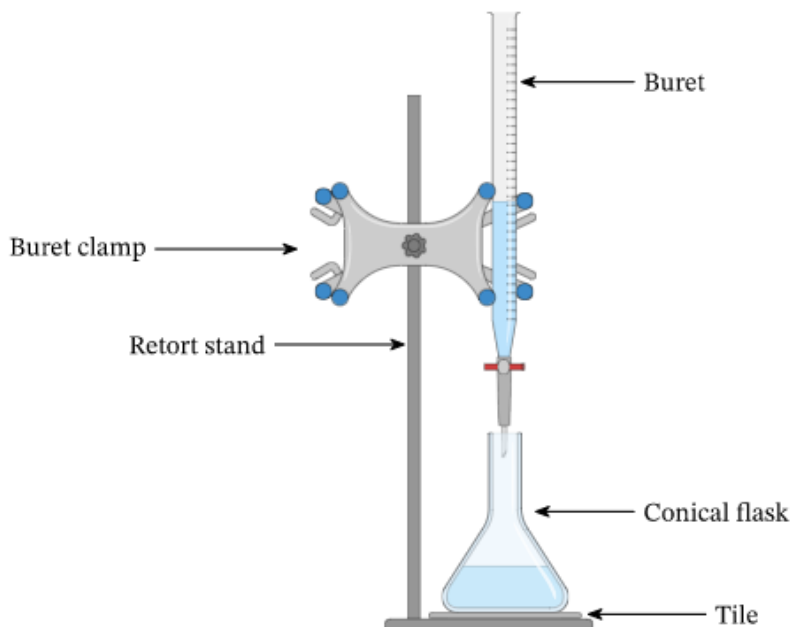


Figure 11.7: Redox titration using potassium permanganate

Procedure

1. Prepare a solution of sodium oxalate using 0.25 g of sodium oxalate in 250 mL of distilled water and pour it in a conical flask.
2. Pipette 25 mL of sodium oxalate and add 3 mL of 3M sulfuric acid in a conical flask of 250 mL.
3. Clamp the burette to the retort stand and place the small funnel on top. Making sure the burette is closed, carefully pour a potassium permanganate standard solution to the burette until the burette is full.
4. Add 0.02 M of potassium permanganate solution to the conical flask dropwise while shaking. Stop as soon as the colour of the solution changes (end point). What do you observe?
Answer: A colorless solution turns light pink.
5. Repeat the experiment at least four times.

Interpretation of results and conclusion

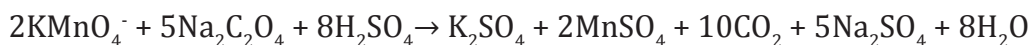
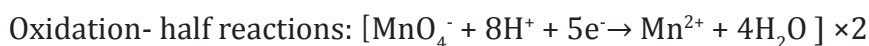
Data recording

Trials	1	2	3	4
Final burette reading/mL	43.00	36.00	40.10	40.20
Initial burette reading/mL	0.00	0.00	0.00	0.00
Volume of KMnO_4 / mL	43.00	36.00	40.10	40.20

Average titre should be obtained using values which differ not by more $\pm 0.10\text{mL}$.

$$\frac{40.10+40.20}{2} = 40.15$$

Write the balanced chemical equation for the reactions



The above equation shows that the mole ratio potassium permanganate: sodium oxalate is 2:5.

1. Calculate the moles of sodium oxalate that reacted in the titration 0

$$\text{Moles of sodium oxalate} = \frac{\text{Mass}}{\text{Molar mass}} = \frac{0.25 \text{ g}}{40.15 \text{ L}} = 0.00186 \text{ mol}$$

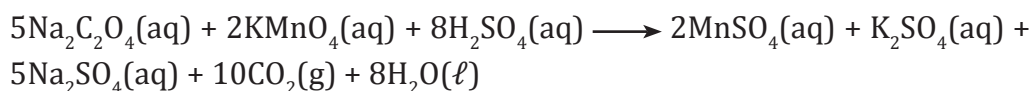
2. Moles of $\text{KMnO}_4 = 0.00186 \times \frac{2}{5} \text{ mol} = 0.000744 \text{ mol}$

3. Molarity (C) of $\text{KMnO}_4 = 0.000744 \text{ mol} \times \frac{1000}{134 \text{ g/mol}} = 0.0185 \text{ mol/L}$

Evaluation

0.2640 g of sodium oxalate is dissolved in a flask and requires 30.74 mL of potassium permanganate (from a buret) to titrate it and cause it to turn pink (the end point).

The equation for this reaction is:



- How many moles of sodium oxalate are present in the flask?
- How many moles of potassium permanganate have been titrated into the flask to reach the end point?
- What is the molarity of the potassium permanganate?

EXPERIMENT 11.8:

Redox titration of iodine using sodium thiosulfate

Rationale

Iodometric titration is a volumetric analysis technique that uses iodine and its oxidation/reduction as an indicator mechanism. Iodine readily reacts with many substances. It is used both to determine the amount of reducing agents (by direct titration with iodine) and of oxidizing agents (by titration of iodine with thiosulfate). This experiment demonstrates how to calculate the concentration of potassium permanganate using redox titration method.

Objective

Learners will be able to determine the percentage of purity of KIO_3 to titrate iodine using sodium thiosulphate.



Required materials

Apparatus

- Burette (50 mL)
- Pipette (50 mL)
- Conical flasks (100 mL)
- Burette stand
- Funnel
- Measuring flask (250 mL)
- Dropper

Chemicals

- Hydrochloric acid, HCl
- Sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$
- Starch solution
- Potassium iodate, (KIO_3)
- Potassium iodide, KI
- Sodium carbonate, Na_2CO_3

Experimental set-up

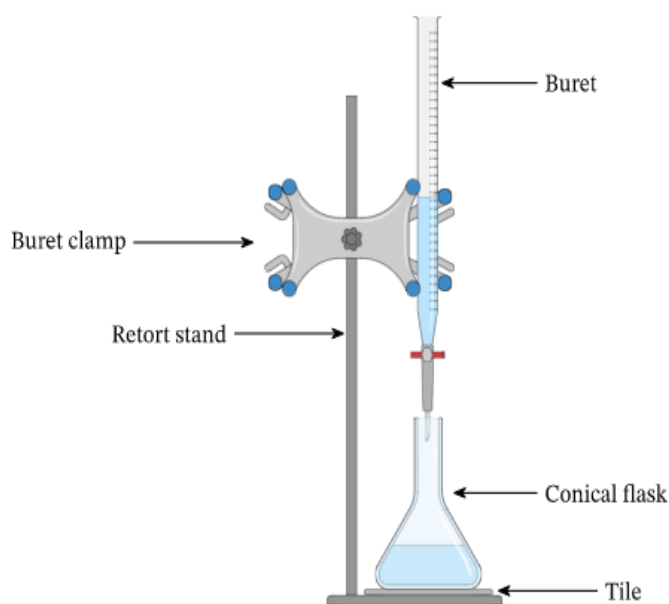


Figure 11.8: Redox titration using Thiosulfate

Procedure

1. Prepare a paste by adding about 0.5 g of soluble starch to 10 mL of deionized water. The starch serves as the indicator for titrations involving iodine.
2. Boil about 300 mL of water in a 1 L beaker and add the paste. Stir the solution while continuing to heat until the solution is completely transparent.
3. Cool the starch indicator solution and place it in a clean storage bottle and label it.
4. Pour 100 mL of distilled water in a 250 mL beaker and add 3.5 g of potassium iodate. Stir to dissolve and make up to 250 mL.
5. Pipette 25 mL of potassium iodate solution and put it in a conical flask of 250 mL, then add 1 g of potassium iodide. Stir the solution to dissolve potassium iodide.

6. Add 5mL of 1M HCl. What do you observe?

Answer: A brown colour is observed.

7. Pour 100 mL of distilled water in a 250 mL beaker and add 7.5 g of sodium thiosulphate pentahydrate. Stir to dissolve and add 0.3 g of sodium carbonate; Stir to dissolve and make up to 250 mL.

8. Clamp the burette to the retort stand and place the small funnel on top.

9. Fill a 50 mL burette with the sodium thiosulphate solution prepared in step 7.

10. Titrate the content of the conical flask (step 5 and 6) with sodium thiosulfate solution dropwise while shaking. Stop as soon as the colour of the solution changes (end point). What do you observe?

Answer: The brown color changes to light yellow.

11. Add 2-3 drops of starch. What do you observe?

Answer: The light yellow colour changes to Blue-black colors.

12. Continue the titration with sodium thiosulfate solution to the conical flask dropwise while shaking. What do you observe?

Answer: Blue-black color changes to colorless.

Interpretation of results and conclusion

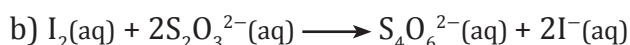
Guiding questions

Determine the percentage purity of iodate.

Calculations

After the end point, the average volume of sodium thiosulfate used is 20.3 mL.

Iodine which is titrated was produced from excess of potassium iodide, and oxidized by potassium iodate as follow:



Molar concentration of the thiosulphate used = $\frac{7.5}{248.18 \times 0.25} = 0.12 \text{ mol/L}$

Number of moles of thiosulphate = $V \times C = \frac{0.1 \times 20.3}{1000} = 2.0310^{-3} \text{ mol}$

The equation (2) shows that the mole ratio iodine: thiosulphate is 1:2.

Number of moles of iodine used in (2) = $\frac{0.00203}{2} = 1.015 \times 10^{-3} \text{ mol}$

Number of moles of iodine produced in (1) = $1.015 \times 10^{-3} \text{ mol} \times 3 = 3.0410^{-3} \text{ mol}$

The equation (1) shows that the mole ratio of iodate: iodine is 1:3.

Number of moles of iodate used = $\frac{0.0030452}{3} = 1.015 \times 10^{-3} \text{ mol}$ in 25 mL

Number of moles of iodate in 250 mL = $\frac{0.001015 \text{ l} \times 250 \text{ mL}}{25 \text{ L}} = 10.15 \times 10^{-3} \text{ mol}$

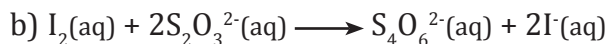
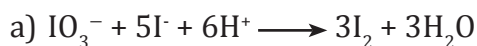
Mass of pure iodate = $M_m \times n = 214 \text{ g/mol} \times 10.15 \times 10^{-3} \text{ mol} = 2.172 \text{ g}$

Percentage of purity = $\frac{2.54 \text{ g}}{3.5 \text{ g}} \times 100 = 62.0 \%$

Evaluation

1. A 0.6125 g sample of potassium iodate (V) KIO_3 is dissolved in water made up 250 mL. A 25.0 mL portion of the solution is added in acid solution. The formed iodine requires 22.5 mL of sodium thiosulphate solution for titration. What is the concentration of the thiosulphate solution? (Answer: $7.63 \times 10^{-2} \text{ M}$).

Use:



2. Calculate the percentage of impure samples of sodium thiosulphate from the following data: a 0.2368 g sample of the sodium thiosulphate was added to 25 mL of 0.0400 mol/L iodine solution. The excess of iodine that remained after reaction needed 27.8 mL of 0.0400 mol dm^{-3} thiosulphate solution in a titration.

EXPERIMENT 12.1: Verification of the conductivity of solutions**Rationale**

Conductivity is a measure of how well a solution conducts electricity. To carry a current a solution must contain charged particles or ions. Most conductivity measurements are made in aqueous solutions, and the ions responsible for the conductivity come from electrolytes dissolved in water. Conductivity of solution is therefore an appropriate measure for routine testing of solutions like in water treatment and in agricultural industries to determine the quality of water and the composition of solutions. This experiment helps to verify whether a solution conduct electricity or not.

Objective

Learners will be able to explain the conductivity of solutions.

**Required materials****Apparatus**

- Batteries
- Conducting wires
- Light bulb
- Graphite electrodes
- Beakers
- Washing bottle

Chemicals

- Sodium chloride solution, NaCl (aq)
- Sugar solution
- Distilled water

Experimental set-up

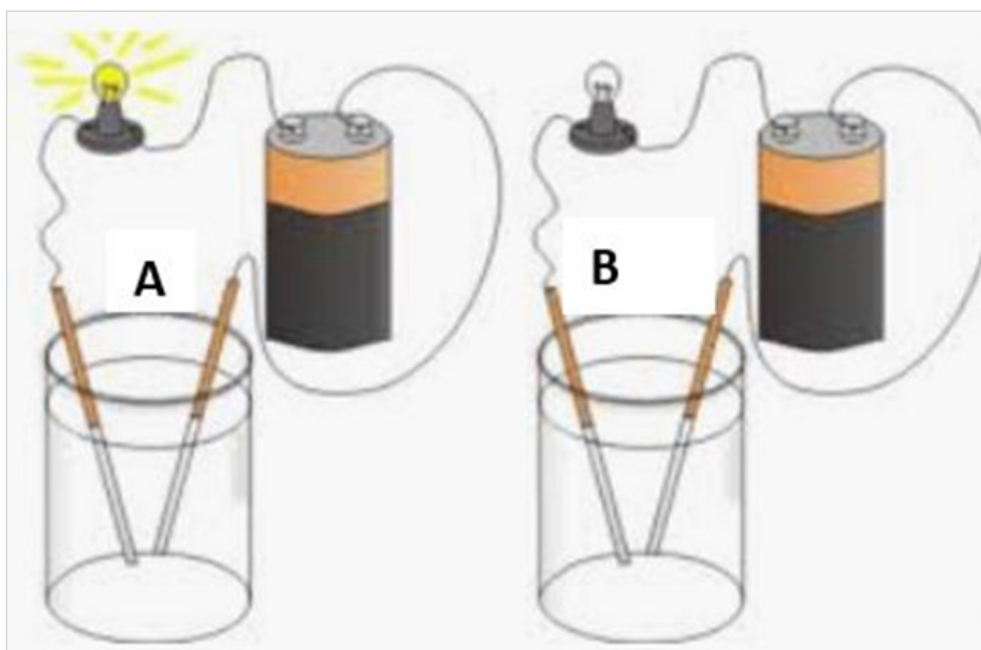


Figure 12.1: Verification of the conductivity of solutions



Procedure

1. Take two beakers and label them A and B.
2. Half-fill the beaker A with sodium chloride solution and beaker B with sugar solution.
3. Put the graphite electrodes in the solution of sodium chloride as shown in Figure 12.1. What do you observe?
Answer: The bulb lights up
4. Wash the electrodes and then dry them with a piece of tissue.
5. Repeat step 3 using the sugar solution. What do you observe?
Answer: The bulb does not light up

Interpretation of results and conclusion

Guiding questions

Explain why does sodium chloride solution conduct electricity whereas sugar solution does not?

Answer to guiding questions:

Sodium chloride solution contains charged ions and ionizes into Na^+ and Cl^- in water that are freely moving about and responsible for the conductivity of electricity. Sugar solution does not contain any ions. Therefore, it does not conduct electricity. The solution which conducts electricity is electrolyte while the one which does not conduct electricity is a non-electrolyte. The non-electrolyte does not ionize into ions (eg: sugar, ethanol, benzene). Most acids, bases and salts are examples of electrolytes as they ionize in water. Electrolytes are further classified into strong and weak electrolytes. Strong electrolytes completely ionize into ions (eg: NaCl , HCl , NaOH) while weak electrolyte is partially ionized into ions (Ammonia, acetic acid, magnesium hydroxide).

Evaluation

1. What do all the solutions that conduct electricity have in common? Explain your answer by using sodium chloride solution.

Answer: All the solutions that conduct electricity contain ions. For example, NaCl solutions conduct electricity because the NaCl solution is a strong electrolyte in which the constituent ions dissociate entirely into Na^+ and Cl^- ions.

2. Describe the 2 types of electrolytes by using some examples.

Answer: See interpretation

EXPERIMENT 12.2:

Comparison of electrolytic conductivity and metallic conductivity

Rationale

Substances conduct electricity due to presence of mobile electrons or presence of free ions. Some common conductors are metals. Electrolytes can also be used to make a circuit. Strong electrolytes conduct electricity as metals do due to the mobility of the positive and negative ions. However, the conductivity of metals is made possible by the movement of free electrons. This differentiates it from electrolyte in which free ions are responsible of the conduction of electricity. Knowledge in the difference in conductivity helps to predict which substances are good or bad conductor of electricity hence care is taken while working with them to avoid accident (electric shock). Therefore, this experiment helps to compare the conductivity of metal and electrolyte.

Objective

Learners will be able to compare the electrolytic conductivity and metallic conductivity.



Required materials

Apparatus

- Batteries
- Conducting wires (copper and iron)
- bulbs
- Iron nails
- Graphite electrodes
- Beakers

Chemicals

- Sodium hydroxide solution, NaOH (aq)

Experimental set-up

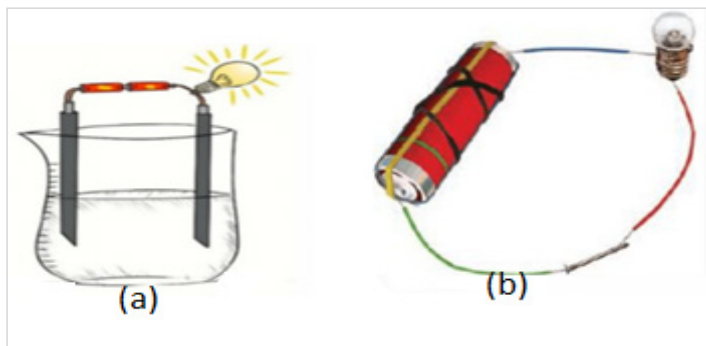


Figure 12.2: *Electrolytic and metallic conductivity*



Procedure

1. Prepare a clean beaker and half-fill it with sodium hydroxide solution.
2. Put the graphite electrodes in the sodium hydroxide solution as displayed in Figure 12.2. (a) What do you observe?

Answer: The bulb lights up

3. Connect a bulb to the batteries by using electric wires as shown in Figure 12.2. (b). What do you observe?

Answer: The bulb lights up

Interpretation of results and conclusion

Guiding questions

How are the intensities of lights in the experiment (a) and (b)?

Answer to guiding questions

Metals conduct electricity due to the flow of electrons and there is no decomposition of the substance while the conductivity of electrolytes is caused by free movement of ions generated from solution. Both metals and electrolytes light up the bulb which demonstrates that they conduct electricity.

Evaluation

1. What is the difference between electrolytic conduction and metallic conduction?

Answer:

Electrolytic conduction	Metallic conduction
It is due to the flow of ions.	It is due to the flow of electrons.
It is accompanied by decomposition of the substance. (Physical as well as chemical change occur)	It is not accompanied by decomposition of the substance. (Only physical changes occurs)
It involves transfer of matter in the form of ions.	It does not involve transfer of matter.
Conductivity increases with increases in temperature and degree of hydration due to decreases in viscosity of medium.	Conductivity decreases with increase in temperature.

2. Mercury is a liquid at room temperature and a good conductor of electricity. Explain why is not mercury considered as an electrolyte?

Answer: Tough, mercury is a liquid at room temperature and is a metal element also known as liquid metal not a compound. It does not ionize or dissociate in solvent that is the reason why it is not considered as an electrolyte.

EXPERIMENT 13.1:

Electrolysis of water

Rationale

Electrolysis is a technique used by scientists to decompose a compound or molecule into its components by passing an electric current. One of the many applications of electrolysis is the production of substances. By passing electricity to acidified water, hydrogen and oxygen gases are produced. Hydrogen gas is produced on large scale during electrolysis of water and is used to manufacture fertilizers, drugs, fuel, paints, and other useful chemicals. This experiment demonstrates how the electrolysis can be used to prepare different substances such as metals and non-metals, and their applications in our daily life.

Objective

Learners will be able to explain the electrolysis of water.



Required materials

Apparatus

- Plastic mug
- Rubber stopper
- Carbon electrodes (anode & cathode)
- Battery
- Test tube

Chemicals

- Water, H_2O
- Dilute sulphuric acid, H_2SO_4

Experimental set-up

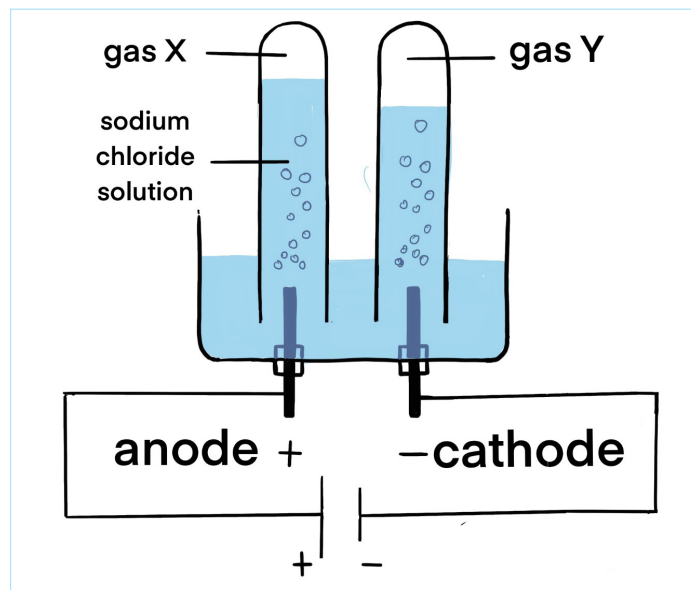


Figure 13.1: *Electrolysis of water*



Procedure

1. Drill two holes in the bottom of the plastic mug.
2. Insert the two rubber stoppers into the corresponding holes.
3. Insert the anode and cathode carbon electrodes into the rubber stoppers.
4. Connect a 6-volt battery to these carbon electrodes.
5. Fill the plastic mug half-way with water so that the carbon electrodes are submerged.
6. Add a few drops of sulphuric acid.
7. Fill two test tubes with water, then invert the test tubes onto the carbon electrodes.
8. Turn on the current from the 6-volt battery.
9. Wait a while for the reaction to proceed. What do you observe?

Answer: There will be bubble formation at both electrodes in the test tubes which means gas is forming or liberating from water.

10. Carefully remove the test tubes from the mug once they have been filled with the gases.

11. Place a lit candle near the mouth of the test tubes to determine which gas is present. What do you observe?

Answer: The gas formed at the cathode produces a pop sound while the one at the anode produces a bright flame.

Interpretation of results and conclusion

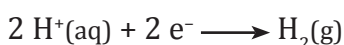
Guiding question

1. How does electrolysis of water proceed? write down the balance chemical equation for the process.

Answer to guiding questions:

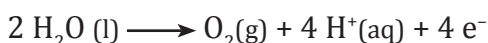
Electrolysis of water takes place in an electrolytic cell which consists of a positively charged anode and a negatively charged cathode, and both are typically made of carbon. The chemical reaction that occurs at electrodes are the following:

At the cathode, hydrogen ions acquire electrons and are converted into hydrogen gas in a reduction reaction.



The hydrogen gas formed at the cathode is characterized by a pop sound during its testing.

At the anode, water molecules are oxidized to oxygen gas in an oxidation reaction.



The oxygen gas formed at the anode produces a bright flame.

Overall, the chemical equation of water electrolysis is



The small amount of an electrolyte such as H_2SO_4 is required because pure water cannot carry enough charge due to a lack of ions.

Evaluation

1. What are electrodes used in the electrolysis of water?
Answer: carbons are electrodes used in the electrolysis of water.
2. What are the products formed during the electrolysis of acidified water?
Answer : See interpretation

EXPERIMENT 13.2:

Electrolysis of concentrated sodium chloride solution

Rationale

Sodium chloride is a common salt which is used for food seasoning as well as to produce chemicals for a range of uses like soaps making, and as a natural preservative. Electrolysis of concentrated sodium chloride produces hydrogen gas, chlorine gas and sodium hydroxide. Hydrogen is used as a fuel and for making ammonia. Chlorine is used to sterilize water supplies, to make bleach, and hydrochloric acid. Sodium hydroxide is used to make soap, paper, and bleaches. This experiment deals with the electrolysis of concentrated sodium chloride solution.

Objective

Learners will be able to perform the experiment of electrolysis of concentrated sodium chloride solution.



Required materials

Apparatus

- Connecting wire
- test tubes
- Dry cell
- Candle
- March box
- Switch
- Carbon or graphite rods

Chemicals

- Concentrated sodium chloride, NaCl
- Litmus paper

Experimental set-up

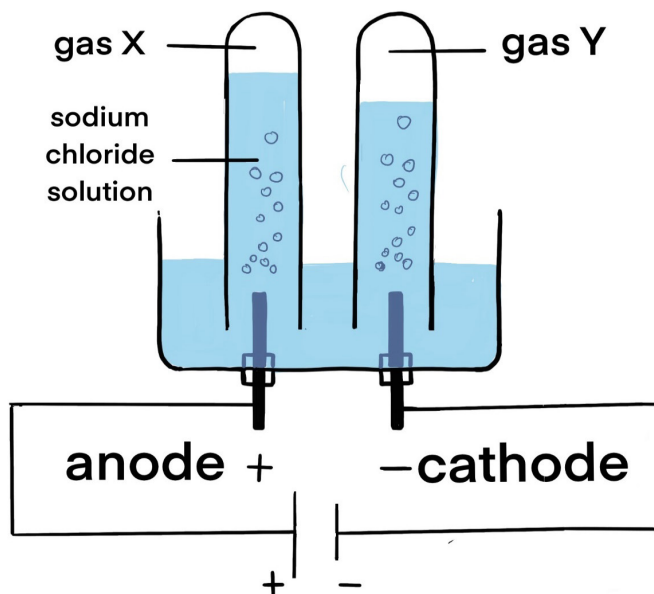


Figure 13.2: Electrolysis of concentrated sodium chloride solution



Procedure

1. Clamp the electrolytic cell to the stand and half-fill with concentrated sodium chloride solution.
2. Fill the two small test tubes with concentrated sodium chloride solution and invert them over the electrodes as presented in figure 13.2.
3. Connect the electrodes to the power supply using the wires and clips.
4. Allow the gases to be collect in the test tubes.
5. Disconnect the circuit when test tubes have filled with gas.
6. Carry out the test of the gas produced on the anode. What do you observe?

Answer: A pale green gas that turns blue litmus paper red and bleach, is observed.

7. Place a lit candle near the mouth of the test tubes to determine the gas generated on cathode. What do you observe?

Answer: The gas burns with a pop sound.

Interpretation of results and conclusion

Guiding questions

1. How does electrolysis of concentrated sodium chloride happen? Explain your answer by providing balanced chemical equations of the reactions taking place at each electrode.

Answer to guiding questions

When an electric current is passed through concentrated sodium chloride solution, hydrogen gas is formed at the negative electrode while chlorine gas is formed at the positive electrode, and a solution of sodium hydroxide is also formed.

At the cathode (negative electrode), the H^+ cations are reduced when they gain electrons.

The chemical equations of the reduction reaction: $2\text{H}^+(\text{aq}) + 2\text{e}^- \longrightarrow \text{H}_2(\text{g})$

The gas which burns with a pop sound is hydrogen

At the anode (positive electrode), the Cl^- anions are oxidized when they lose electrons.

The chemical equation of the oxidation reaction: $2\text{Cl}^-(\text{aq}) \longrightarrow \text{Cl}_2(\text{g}) + 2\text{e}^-$

The pale green gas that turns blue litmus paper red and bleaches is chlorine.

The products of electrolysis of concentrated aqueous sodium chloride are sodium hydroxide, hydrogen gas, and chlorine gas.

Evaluation

1. What are the products formed when concentrated sodium chloride solution is electrolyzed?

Answer: When a concentrated solution of sodium chloride (brine) is electrolyzed, it gets decomposed, and the products of this reaction are sodium hydroxide, chlorine gas and hydrogen gas.

2. Suggest how can the produced gases during the electrolysis of concentrated sodium chloride solution be tested?

Answer: See interpretation

EXPERIMENT 13.3:

Electrolysis of dilute sodium chloride solution

Rationale

The electrolysis of dilute sodium chloride produces hydrogen and oxygen. The hydrogen gas is used in fuel cells to generate electricity, in ammonia preparation, and in margarine making. Oxygen is used at hospitals more especially in treatment of patient having breathing problems, in production of steel, in welding and cutting of steel and other metals, in rocket propellant. In addition, oxygen is also used as therapy and life support systems in aircraft, submarines, spaceflight and diving. This experiment highlights the electrolysis of dilute sodium chloride solution.

Objective

Learners will be able to conduct the experiment of electrolysis of dilute sodium chloride solution.



Required materials

Apparatus

- Connecting wire
- test tube
- Dry cell
- Switch
- Carbon or graphite rods

Chemicals

- water
- Salt

Experimental set-up

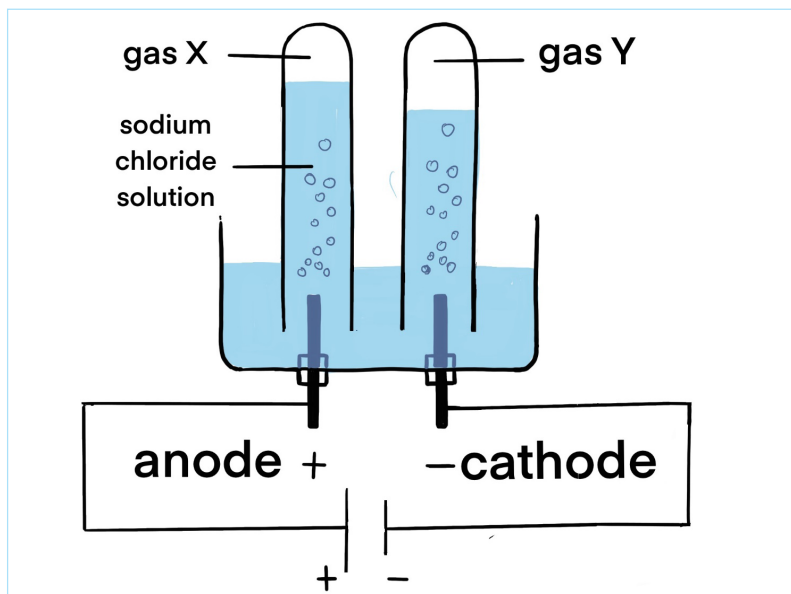


Figure 13.3: Electrolysis of dilute sodium chloride solution



Procedure

1. Clamp the electrolytic cell to the stand and half-fill with dilute sodium chloride solution.
2. Fill the two small test tubes with dilute sodium chloride solution and invert them over the electrodes as shown in the diagram.
3. Connect the electrodes to the power supply using the wires and clips.
4. Allow the gases to collect in the test tubes.
5. Disconnect the circuit when the test tubes have filled with gas.
6. Place a lit candle near the mouth of the test tubes to determine the gas generated on anode. What do you observe?
Answer: Bright flame is observed.
7. Place a lit candle near the mouth of the test tubes to determine the gas is present on the cathode. What do you observe?
Answer: The gas burns with a pop sound.

Interpretation of results and conclusion

Guiding questions

1. How does electrolysis of dilute sodium chloride occur?
2. Write balanced chemical equations of the reactions occurring at each electrode

Answer to guiding questions

At the cathode

The H^+ and Na^+ ions are attracted to the cathode, H^+ ions gain electrons from the cathode to form hydrogen gas as a result, H^+ ions are discharged as hydrogen gas, which bubbles off.

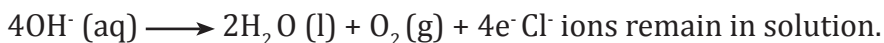
The chemical equation of reaction:



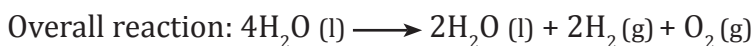
The gas produced which burns with a pop sound is hydrogen.

At the Anode

The OH^- and Cl^- are attracted to the anode and as Cl^- ions are present in small amount, OH^- ions are preferably discharged to give oxygen and water. The chemical equation of reaction:



The gas produced which gives a bright flame with a lit candle is oxygen.



The electrolysis of dilute sodium chloride solution is like that of water; thus, hydrogen and oxygen gas are produced.

Evaluation

1. Briefly explain the electrolysis of dilute sodium chloride solution by using inert carbon electrodes and write balanced half chemical equations at anode and cathode electrodes.
2. What is the effect of concentration on the electrolysis of sodium chloride?

EXPERIMENT 13.4:

Electrolysis of concentrated copper (II) sulphate solution using carbon electrode

Rationale

Some metals can be extracted from their corresponding salts. Electrolysis is one of the techniques that is used in this process. In this experiment, copper metal can be extracted by using the electrolysis of copper (II) sulfate solution. The outcomes of this experiment help to appreciate the use of inactive electrodes during electrolysis.

Objective

Learners will be able to carry out the experiment of electrolysis of concentrated copper (II) sulphate solution using carbon electrode



Required materials

Apparatus

- Connecting wire
- Carbon electrodes
- Dry cell
- Switch
- Beaker

Chemicals

- Concentrated copper (II) sulphate solution

Experimental set-up

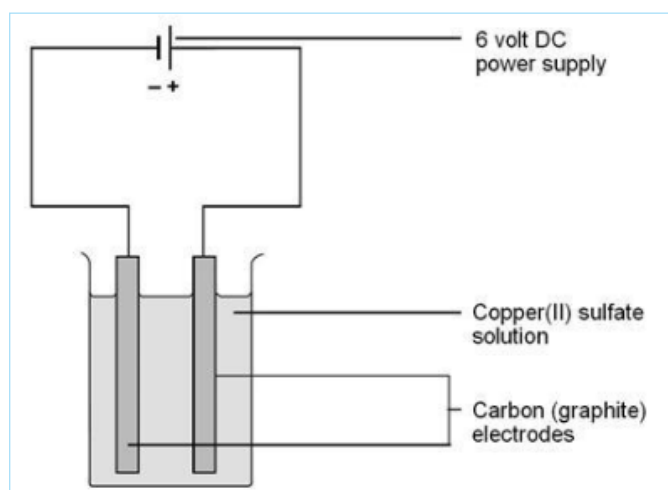


Figure 13.4: Electrolysis of concentrated copper (II) sulphate solution using carbon electrode



Procedure

1. Half-fill beaker with concentrated copper (II) sulphate solution.
2. Clamp the electrolytic cell to the stand.
3. Connect the electrodes to the power supply using the wires and clips. What do you observe?

Answer: On the cathode there is formation of bubbles of a colorless gas. On the anode, there is a deposition of a red solid and the blue color of electrolyte fades off.

Interpretation of results and conclusion

Guiding question

1. How does the electrolysis of concentrated copper (II) sulphate solution proceed by using carbon electrode? Support your answer by writing down the balanced chemical equations of reactions happening on each electrode.

Answer to guiding questions:

The less reactive a metal, the more readily its ion is reduced on the electrode surface but the sulphate ion to the side of anode is too stable and nothing happens. Instead, hydroxide ions are discharged and oxidized to form oxygen. The remaining solution is sulfuric acid.

At the cathode

The Cu^{2+} ions (from copper sulphate) and H^+ ions from water are attracted to the negative electrode. Copper ion is preferably discharged and being reduced into copper metal and deposited on electrode.

The chemical equation of the reduction reaction: $\text{Cu}^{2+}(\text{aq}) + 2\text{e}^- \rightarrow \text{Cu}(\text{s})$

The red solid deposited at the cathode is copper metal.

At the anode

The SO_4^{2-} (from copper sulphate) and OH^- from water are attracted to the positive electrode. The OH^- ions are preferably discharged as they are lower in electrochemical series (they are stronger reducing agents than sulphate ions). An oxygen colorless gas is liberated..

The chemical equation of the oxidation reaction: $4\text{OH}^-(\text{aq}) \rightarrow 2\text{H}_2\text{O}(\text{l}) + \text{O}_2(\text{g}) + 4\text{e}^-$

Copper metal is produced at cathode while oxygen gas is produced at anode, and blue color of solution turns colorless.

Evaluation

1. Briefly discuss the process of electrolysis of concentrated copper (II) sulphate solution.
2. Why do hydroxide ions are preferably discharged instead of sulphate ions?
3. Explain the reason why does the blue color of copper (II) sulphate solution fade off?
4. What will happen to the masses of the anode and the cathode during the purification of copper?

EXPERIMENT 13.5: Electroplating of graphite using copper (II) sulphate solution

Rationale

There are metals that are easily attacked by corrosion and others have an appearance that needs to be improved, thus electroplating is a durable solution to deal with the above-mentioned issue in non- electroplated materials. This experiment demonstrates the effect of graphite electroplating.

Objective

Learners will be able to perform and explain electroplating of graphite using copper (II) sulphate solution.



Required materials

Apparatus

- Two crocodile clips
- 1.5 volt DC battery with battery holder
- Copper electrode
- graphite
- Beaker

Chemicals

- Copper (II) sulphate solution

Experimental set-up

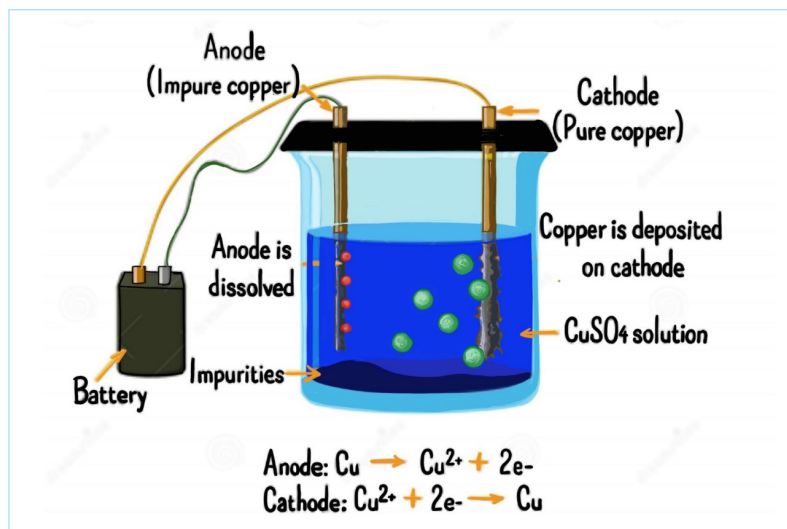


Figure 13. 5: Electroplating of graphite using copper



Procedure

1. Prepare the graphite for copper-plating by cleaning it with soap and water. Dry it off on a paper towel.
2. Dissolve copper sulphate into hot water in a beaker until no more dissolves.
3. Use one crocodile clip to attach the copper electrode to the positive terminal of the battery (anode) and the other to attach the graphite to the negative terminal (cathode).
4. Suspend the graphite in the solution of copper sulphate.
5. Place the copper electrode into the solution, making sure it does not touch the graphite.
6. Leave the circuit running for 20-30 minutes. What do you observe?

Answer: The red solid is deposited on graphite electrode, the blue color of copper sulphate solution does not fade off.

Interpretation of results and conclusion

Guiding questions

1. What are the chemical equations of the reactions occurring at electrodes during electroplating of graphite using copper.
2. What is the identity of product formed at the cathode?

Answer to guiding questions

The coating of a metal object with another metal object is called electroplating. The object to be plated is placed at the cathode in the cell so that metal ions move to it when inserted in an electrolyte.

At the cathode

Copper metal deposit at cathode and hence the size of the cathode increases.

The chemical equation of the reduction reaction: $\text{Cu}^{2+}(\text{aq}) + 2\text{e}^{-} \rightarrow \text{Cu}(\text{s})$

At the anode

Copper metal of anode will dissolve, and copper (II) ions are added in electrolyte. Hence the size of the anode decreases.

The chemical equation of the oxidation reaction: $\text{Cu}(\text{s}) \rightarrow \text{Cu}^{2+}(\text{aq}) + 2\text{e}^{-}$

The blue color of copper sulphate solution does not fade off because the copper ions discharged at the cathode are replaced by the ones from the anode.

Before the electrolysis, the graphite has dark-grey color, after the electrolysis a red-brown layer covers the graphite. The size of the graphite increased while that of copper electrode decreases.

Evaluation

1. Are electroplated objects alloys? Explain.
2. What is the difference between electrolytic extraction of a metal and electroplating?
3. Mention at least three applications of electroplating.

EXPERIMENT 14.1:

Determination of enthalpy change of combustion of ethanol

Rationale

Some organic chemical reactions release heat while others absorb it to form products. Ethanol is used as fuel for a range of applications ranging from cooking to fueling cars. It becomes a significant component for biofuels. However, a good fuel should be able to generate a high amount of heat. In this experiment through combustion of ethanol, heat, carbon dioxide, and water are produced.

Objective

Learners will be able to determine the enthalpy change of ethanol combustion.



Required materials

Apparatus

- Spirit burner
- Thermometer
- Calorimeter
- Measuring cylinder
- Retort stand and accessories
- Balance
- Breeze shield

Chemicals

- Ethanol
- Tap water

Experimental set-up

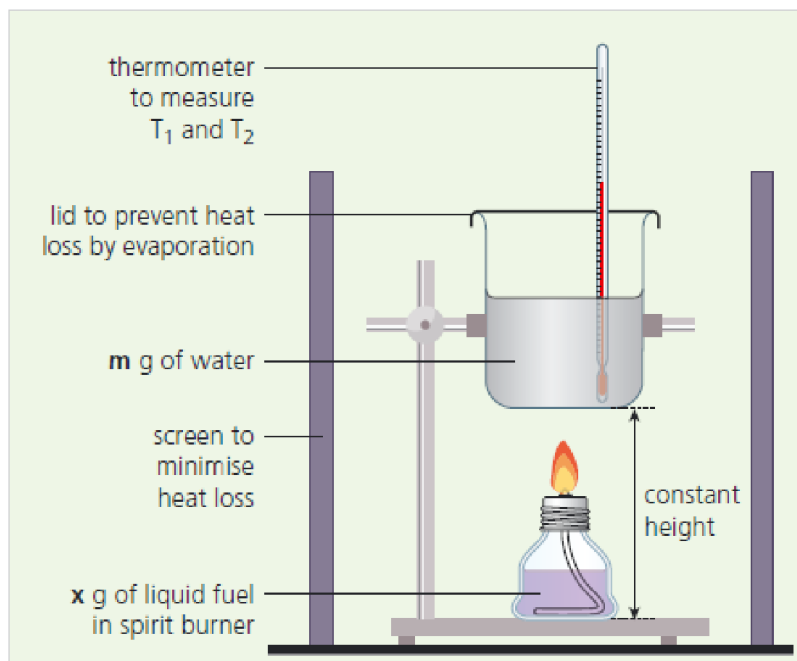


Figure 14.1: Determination of enthalpy change of combustion of ethanol



Procedure

1. Weigh the spirit burner containing ethanol with its cap and record X_1 g.
2. Using the measuring cylinder, measure 200 mL of water and pour it into the calorimeter.
3. Set up the apparatus as indicated in the figure 14.1.
4. Measure and record the temperature of water as T_1 .
5. Remove the cap from the spirit burner and immediately light the burner.
6. Measure the temperature of water using thermometer.
7. When the temperature has risen by about , recap the spirit burner, measure and record the maximum temperature of water as T_2 .
8. Reweigh the spirit burner and record X_2 g.

Caution:

1. Ethanol is highly flammable, and the main risk is from burns.
2. Wear eye protection.
3. Ensure the spirit burner is always placed in a stable position.
4. If you have to re-fill the spirit burner, allow it to cool and then fill it away from sources of ignition.

Interpretation of results and conclusion**Guiding question**

1. How can the enthalpy change of ethanol combustion be calculated?

Answer to guiding questions

The energy produced by the combustion of ethanol is used to heat a known mass (m) of water. Therefore, the heat provided by ethanol is equal to the heat received by water.

The heat amount can be calculated by using the following the relation:

$$q = m \times C_s \times \Delta T$$

Where m is the mass of water;

C_s is the specific heat capacity of water;

ΔT is the temperature change

Knowing the mass of ethanol used, the enthalpy change of combustion can be calculated.

Evaluation

1.200 mL of water were heated by burning ethanol in a spirit burner. The following mass measurements were recorded:

Mass of spirit burner and ethanol (before burning) = 58.25 g

Mass of spirit burner and ethanol (after burning) = 57.62 g

The initial temperature of the water was 20.7 °C and the highest temperature recorded was 41.0°C. The specific heat capacity of water is 4.18 J g⁻¹K⁻¹. Calculate the value of the standard enthalpy change of combustion of ethanol in kJ mol⁻¹.

Answer: The enthalpy of combustion of ethanol is 1238.7445 kJmol⁻¹.

EXPERIMENT 14.2:

Determination of enthalpy change of neutralization of hydrochloric acid with sodium hydroxide solution

Rationale

The amount of heat produced when an acid and a base combine to make salt and water is known as the enthalpy of neutralization (H). This concept helps in estimating how much heat is generated or absorbed during a neutralizing process. It also enables researchers to learn more about the strength of acids and bases. In this experiment, chemists estimate the amount of heat generated when sodium hydroxide neutralizes hydrochloric acid.

Objective

Learners will be able to determine the enthalpy change of neutralization of hydrochloric acid with sodium hydroxide solution.



Required materials

Apparatus

- Calorimeter
- Thermometer
- Scale
- Weigh boats

Chemicals

- 1 M HCl
- 1 M NaOH



Procedure

1. Weigh an empty plastic beaker. Record the mass.
2. With a graduated cylinder, measure 50 mL of 1 M HCl and pour it into the plastic beaker.
3. With a thermometer, measure the temperature (T_1) of the HCl.
4. Rinse the graduated cylinder and measure 50 mL of 1 M NaOH.
5. Measure the temperature (T_2) of the NaOH.
6. Pour the NaOH into the plastic beaker and stir gently with the thermometer.
7. Record the highest temperature (T_3) that the thermometer reaches.
8. Weigh the plastic beaker and the liquid.

Interpretation of results and conclusion

Guiding questions

Calorimetry is also used to determine the enthalpy change of a reaction taking place in solution. For an exothermic reaction, the heat energy released increases the temperature of the water in solution while for an endothermic reaction, the heat energy absorbed is derived from water in the solution and the temperature of the solution falls.

When hydrochloric acid reacts with sodium hydroxide, the temperature of the mixture increases and the heat is transferred to the plastic beaker, the reaction is exothermic.

Enthalpy change = mass of the solution \times specific heat capacity \times temperature rise

$$\Delta H = m \times C_s \times \Delta T$$

If the solutions used are more concentrated, the temperature increases, and the amount of heat exchanged also increases. However, the molar enthalpy of

neutralization remains the same because when the concentration increases, the density increases as well as the number of moles.

Evaluation

1. Perform an experiment to determine the enthalpy of neutralization of HClO_4 and NaOH by reacting 50.0 mL of 2.00 M HClO_4 with 50.0 mL of 2.00 M NaOH in a calorimeter and give a full interpretation of this experiment.
2. An experiment was conducted to find out the enthalpy of neutralization of a weak acid, HX . 30 mL of 1M HX solution were mixed with 40 mL of 1M KOH (in excess) in a polystyrene cup. The temperature in the reaction was 5.0 °C. Calculate the enthalpy change for the neutralization of the weak acid.

EXPERIMENT 14.3:

Determination of enthalpy change of dissolution of sodium hydroxide in water

Rationale

Determination of enthalpy change of dissolution allows scientists to determine the amount of a substance dissolved in water. It also helps to know whether the process is endothermic or exothermic.

Objective

Learners will be able to calculate the enthalpy change of dissolution of sodium hydroxide in water.



Required materials

Apparatus

- Calorimeter
- Thermometer
- Electronic balance
- Graduated cylinder

Chemicals

- Sodium hydroxide, NaOH(s)
- Water



Procedure

1. With a graduated cylinder, measure 100 mL of water and pour it into the calorimeter.
2. With a thermometer, measure the temperature (T_1) of water.
3. Weight X g sodium hydroxide.
4. Put the sodium hydroxide into the calorimeter containing water and stir gently.
5. Use a thermometer to record the highest temperature (T_2).

Interpretation of results and conclusion

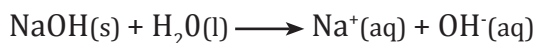
Guiding question

1. How is the dissolution enthalpy change of sodium hydroxide determined experimentally?

Answer to guiding questions

When a sodium hydroxide is dissolved in water, it is usually referred to as the solute and water as a solvent. For the dissolution of sodium hydroxide to take place, the water molecules must rearrange to allow room for the sodium hydroxide. All these interactions involve energy, often detected in the form of heat exchange. If the system releases heat during these reactions, it is known as exothermic, and if the system takes in heat, it is called endothermic.

The dissolution reaction of sodium hydroxide is illustrated by the following chemical equation



This reaction is an exothermic process so an increase in temperature of the solution is observed.

Evaluation

A student determined the enthalpy of solution of NaOH by dissolving 4.00 g of NaOH(s) in 180 mL of pure water (density of water is 0.999 g/mL) at an initial temperature of 19.5 °C. Temperature-time data collected after mixing was extrapolated back to the time of mixing to obtain a temperature change 5.5°C. The density of the final solution was 1.013 g/mL at 25 °C, and the solution specific heat was 4.08 J/g °C. Using the provided data, calculate the enthalpy change of dissolution

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