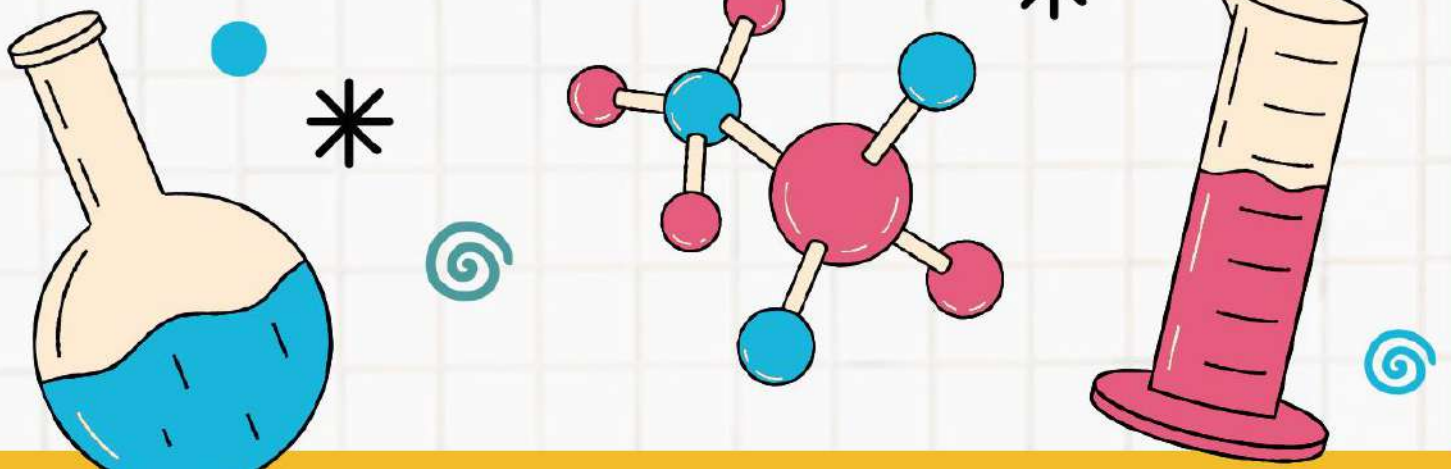
Pusat Asasi Sains dan Perubatan UniSZA
UniSZA Science and Medicine Foundation Centre

LABORATORY MANUAL
CHEMISTRY II

SEM II 2023/2024

1. Determination Heat of a Reaction
2. Functional Group Identification in Organic Molecules
3. Reaction of Aliphatic and Aromatic
4. Hydrocarbon
5. Classification of Alcohols Carboxylic acid and its Derivatives



LABORATORY RULES AND REGULATIONS

1. Attendance is **COMPULSORY**. If you are unable to attend any practical class, you should produce a medical certificate or a letter of exemption.

2. Read, understand and plan your experiment in a jotter book before entering practical class.

3. Wear shoes, lab coats and safety goggles at all times in the laboratory.

4. Tie long hair or tuck head scarf under your lab coat.

5. Do not wear contact lenses during experiments.

6. Foods and drinks are not allowed in the laboratory.

7. Do not perform any unauthorized experiments! Understand and follow the specified procedures for each experiment.

8. Do not waste chemicals. Take only sufficient amount of chemicals needed for your experiments.

9. Replace the lids or stoppers on the reagent bottles or containers immediately after use.

10. Do not remove chemicals from the laboratory.

11. Handle volatile and hazardous compounds in the fume cupboard. Avoid skin contact with all chemicals, wash off any spillages.

12. **Clean up spillages immediately. In case of a mercury spillage, do not touch the mercury. Notify your instructor immediately.**

13. Ensure there are no flames in the vicinity before working with flammable chemicals.

14. **NEVER** leave an ongoing experiment unattended.

15. Be aware or familiar with the location and proper way of handling safety equipment, including eyewash, safety shower, fire blanket, fire alarm and fire extinguisher.

16. Turn off Bunsen flames when not in use. Notify your instructor immediately of any injury, fire or explosion.

17. Do not throw any solid wastes into the sink. Dispose any organic substances in the waste bottles provided.

18. Wash all glassware after use and return the apparatus to its appropriate places.

19. Keep your work area clean and tidy.

20. Notify your instructor immediately of any injury, fire or explosion.

EXPERIMENT 1: DETERMINATION HEAT OF A REACTION

Learning Outcome

At the end of this practical, student should be able to:

1. Calculate the enthalpy change for a reaction from temperature data and specific heats.
2. Use Hess's Law to calculate the enthalpy change for a reaction.

Objective

1. To determine the heat capacity of a calorimeter
2. To determine the heat of neutralisation of HCI and NaOH.

Apparatus

Stopwatch, thermometer, beaker (100 ml), calorimeter or polystyrene cup, measuring cylinder (100 ml) and hot plate.

Chemicals

1M hydrochloric acid (HCI), 1 M sodium hydroxide (NaOH) and distilled water.

Introduction

Heat can be released or absorbed during chemical reactions. It can be measured by using a calorimeter. A calorimeter is a container that is thermally isolated from the environment. Heat released by the chemical reaction, $-q_{rxn}$, is absorbed by the solution and the calorimeter.

$$-q_{rxn} = q_s + q_c \quad \text{.... (1)}$$

where

q_s = heat absorbed by solution
q_c = heat absorbed by calorimeter

The heat absorbed is proportional to the change in temperature. The proportionality constant, C, is known as the heat capacity of a sample. Heat capacity is defined as the amount of heat required to increase the temperature by 1 °C.

$$q = C\Delta T \quad \text{....(2)}$$

For a solution, the heat absorbed is proportional to the mass of the solution and the change in temperature. Proportionality constant, c, is known as the specific heat capacity of solution per unit mass.

For example, the specific heat capacity for water is 4.1g JK^{-1} g^{-1}.The specific heat capacity of a very dilute solution is equivalent to the specific heat capacity of pure water.

Therefore, the value of the specific heat capacity of the solution used in this experiment will be 4.18 JK^{-1} g^{-1}.

Heat that is being released or absorbed q_{rxn}, can be determined by measuring the temperature
before and after the reaction.

$$-q_{rxn} = C_c\Delta T + m_s c_s \Delta T \qquad (3)$$

where

ΔT = final temperature of system - initial temperature of system
m_S = mass of solution
C_C = heat capacity of calorimeter
c_S = specific heat capacity of solution

For this experiment the density of the solution is assumed to be the same as the density of water. Therefore, the mass of the solution can be calculated.

Procedure

A. Determination of the heat capacity of a calorimeter

1. Measure the temperature, T, of an empty calorimeter.
2. Pour 50 ml of distilled water into a 100 ml beaker
3. Heat the beaker to a temperature between 50 – 60 °C.
4. Pour the hot water into the calorimeter. Immediately measure the temperature of the hot water, T_2.
5. Observe the decrease in temperature until it remains constant for one minute. Record the temperature, T_3.
6. Determine the heat capacity of the calorimeter.

B. Determination of the heat of neutralisation of 1 M HCl and 1 M NaOH

1. Place 25 ml of 1M NaOH solution into the calorimeter and 25 ml of 1 M HCl solution into a beaker. Record the initial temperature of each solution, T_0.
2. Without removing the thermometer, lift the lid slightly and quickly pour in HCl solution into the calorimeter.
3. Quickly replace the lid of the calorimeter.
4. Stir the solution and record the temperature every 15 seconds. Note the maximum temperature reached.

5. Repeat the experiment.
6. Calculate the heat of neutralisation.

C. Energy Values for Foods

Note: **Each group prepare one food product with nutrition data on label.**

1. Obtain a food product that has a Nutrition Facts label. Record the type of food.
2. Record the serving size.
3. List the grams of carbohydrate, fat, and protein in one serving of the food.
4. From the mass of each food type, calculate the Calories (kcal) of each food type in one serving using the accepted energy values. Round the answer to the tens place.
5. Determine the total Calories (kcal) in one serving.
6. Compare your total to the Calories listed on the label.

PRE-LAB STUDY QUESTION EXPERIMENT 1

DETERMINATION HEAT OF A REACTION

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

1. What is the equation for converting a Fahrenheit temperature to a Celsius temperature?

2. What is meant by the term *specific heat*?

3. Why is a measured amount of water needed to determine the specific heat of a metal object?

4. What are the units of specific heat?

5. How is the caloric value of a food sample determined?

DATASHEET EXPERIMENT 1
DETERMINATION HEAT OF A REACTION

NAME :_____ DATE EXPERIMENT :_____
STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

A. Determination of the heat capacity of a calorimeter

Temperature	Reading (°C)
T_1	
T_2	
T_3	

Q1: Calculate heat capacity of calorimeter?

B. Determination of the heat of neutralisation of 1 M HCl and 1 M NaOH

Temperature	Reading 1 (°C)	Reading 2 (°C)
T_0		
T_1		
T_2		
T_3		
T_4		

Q1: Calculate heat of neutralization

C. Energy values for foods

Name of food product	**:**
Serving size	**:**
Mass of food types in one serving	
Carbohydrate:	
Fat:	
Protein:	
Calculations for kcal per serving (s*how calculations*)	
Carbohydrate:	
Fat:	
Protein:	
Total Calories (kcal) per serving:	
Calories (for one serving) listed on the label	

Lecturer's signature,

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EXPERIMENT 2: FUNCTIONAL GROUP IDENTIFICATION IN ORGANIC MOLECULES

Learning Outcome

At the end of this practical, students are able to:

1. Recall reagents and predict products from a defined set of organic reactions test.
2. Draw organic structures consistent with the results of specific chemical tests.
3. Identify the functional group of organic molecules.

Introduction

Compounds that are easily oxidized react with a solution of chromium (VI) oxide in sulfuric acid, commonly referred to as "chromic acid." Primary and secondary alcohols react within 2-3 seconds to form an opaque blue-green suspension. Aldehydes give the same result but usually take 10 seconds or more to react.
Chemists have developed a number of simple chemical tests that are positive only for compounds having certain kinds of functional groups:

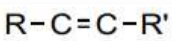
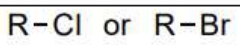
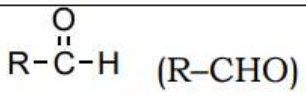
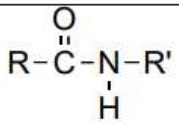
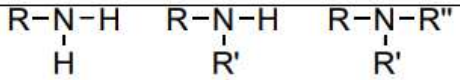
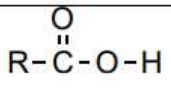
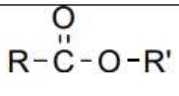
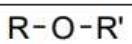
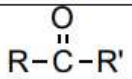
1. **Aldehydes and ketones** both react with 2, 4-dinitrophenyl hydrazine (DNPH) reagent to yield yellow or orange precipitates within a few minutes.

2. **Alkenes** react readily with dilute aqueous potassium permanganate to form a brown precipitate as the purple color of the permanganate disappears.

3. **Alkyl halides** give a green flame in the Beilstein test, which involves heating a copper wire moistened with the unknown in a burner flame.

Qualitative organic analysis, the identification of organic compounds based on their physical and chemical properties, is analogous in some ways to the identification of plants and animals according to their taxonomy, their structural features and presumed natural relationships. To classify an organic compound into a given family requires first detecting a specific *functional group* (characteristic set of atoms) in the molecules of organic compounds. Because functional groups influence the physical, chemical, and spectral properties of an organic compound. A chemist can identify a compound's functional groups by measuring certain physical properties, observing its chemical behavior with different classification reagents, and studying other spectral data. Some common organic functional groups are shown in the **Table 4.1**.

Table 4.1: Some common organic functional groups

Functional group name	General formula*
Alkene	
Alkyl halide	
Alcohol	
Aldehyde	
Amide	
Amine	
Carboxylic acid	
Ester	
Ether	
Ketone	

Note: R, R', and R'' are general hydrocarbon groups

Objective

1. To identify class of functional group of a series of organic compounds using specific chemical reactions.

Apparatus

Hot plate, glass rod, white tiles, measuring cylinder, analytical balance, crucible tong, disposable test tubes, stopper (rubber septum), blade, burner, test tube rack.

Chemicals

Hydrochloric acid (HCl), 96% sulphuric acid (H_2SO_4), ether, reagent-grade acetone (C_3H_6O), 2,4-dinitrophenylhydrazine (2,4-DNPH), ethanol (C_2H_5OH), chromic acid (H_2CrO_4) reagent, limonene ($C_{10}H_{16}$), potassium permanganate ($KMnO_4$), bromobenzene.

Procedure

First, you will be performing the functional group identification tests on known compounds (indicated in each of the test procedures) for which each of the tests should produce a positive result. Note your observations carefully in your laboratory report after performing each of these tests, as they will be helpful in identifying an unknown compound later.

Procedure A-C can be performed in any order. One of the following tests will be set up in one of three fume hoods in the lab. When you are finished with a test, move to another hood to perform another test.

A. 2, 4 -Dinitrophenyl hydrazine (2, 4-DNPH) test

In this case, reagent grade acetone will give a positive test). Add one drop of the compound to be tested (reagent-grade acetone) to 1 ml of DNPH reagent in a disposable test tube. Put a stopper (small rubber septum) to the tube and shake the test tube. Then, let the mixture stand for 15 minutes. Carefully record your observations.

B. Chromic acid test

In this case, ethanol will give a positive test. Dissolve one drop of the compound to be tested (ethanol) in 1 ml of reagent-grade acetone in a disposable test tube. Add one drop of the chromic acid reagent. Shake the mixture and observe it for at least 1 minute. Record the time required for positive test carefully.

C. Potassium permanganate test

In this case, limonene will give a positive test). Dissolve one drop of the compound to be tested (limonene) in 1 ml of 95% ethanol in a disposable test tube. Slowly add 5-10 drops of 0.1M potassium permanganate with shaking. Record your observation carefully, after each drop is added.

D. Beilstein's test

In this case, bromobenzene will give a positive test. Make a small loop in the end of a 10 cm length of copper wire. Heat the loop to redness in a burner flame. (Keep the unknown and any other flammable liquids at a safe distance). Dip the loop into a little bit of the compound to be tested (bromobenzene) and hold it in the lower outside part of the flame. Record the color of the flame produced

E. Test of unknown compound

Obtain the unknown compound from your instructor and record its number in your laboratory report.

Important: cap your unknown vial tightly after removing the required amount for the tests, as these compounds may have strong smells and will evaporate very quickly!

Perform each of the four functional group classification tests as in steps 3-6, but use your unknown as the test compound as opposed to the control compounds in the instructions above. Carefully observe and record the results of your tests. Decide whether they are positive or negative. (Positive tests should look similar to the tests with the control reagents in Procedure A-D).

Note: Some tests may sometimes provide ambiguous results. The purity of the test reagent and the unknown compound can have adverse effects on these tests, so it is especially important to prevent cross-contamination today. The 2, 4-DNPH reagent has been known to crystallize a small amount on its own, so you should look for heavy crystallization as a sign of a positive test. Consult with the instructor and/or run a test again if the result seems ambiguous.

Use your reasoning skills to deduce which functional group(s) may be present in your unknown compound. Based on the functional group tests, identify your unknown compound by choosing it from the following possible unknowns;

1-bromobutane, 2-butanone, cyclohexene or 1-butanol

Note: Waste disposable handling

- After of each chemical test is complete, dispose of the chemical and the disposable test tube in the waste container in the hood where the test was conducted.
- Dispose any of unused unknown chemical after you are sure of its identity in the waste container and place the empty vial in the collection bin provided.
- Do not mix up the used unknown vials with the new one

PRE-LAB STUDY QUESTION EXPERIMENT 2

FUNCTIONAL GROUP IDENTIFICATION IN ORGANIC MOLECULES

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

1. Draw the correct chemical structures as line-angle formulas for each of the standard and possible unknown chemicals in this experiment:
    a) Acetone.
    b) Ethanol.
    c) Limonene.
    d) Bromobenzene.
    e) 1- Bromobutane
    f) 2-butanone
    g) Cyclohexene
    h) 1-butanol

2. State the correct IUPAC names for acetone and limonene.

DATASHEET EXPERIMENT 2

FUNCTIONAL GROUP IDENTIFICATION IN ORGANIC MOLECULES

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

A. Test of known compound

Name of Test	Compound Tested	Observation	Functional Group
2,4-DNPH			
Chromic Acid			
Potassium Permanganate			
Beilstein's test			

B. Test of unknown compound

Name of Test	Observation	Functional Group
2,4-DNPH		
Chromic Acid		
Potassium Permanganate		
Beilstein's test		

Lecturer's signature,

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EXPERIMENT 3: REACTION OF ALIPHATIC AND AROMATIC HYDROCARBON

Learning Outcomes

At the end of this practical, students should be to:

1. Identified the chemical properties of aliphatic and aromatic hydrocarbons..
2. Distinguish an alkane from an alkene and an arene

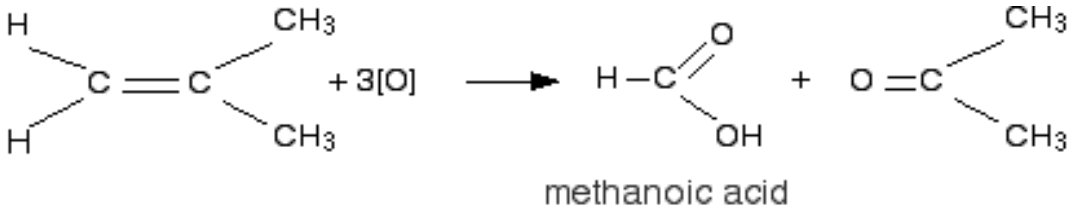
Introduction

Hydrocarbons are organic compounds that contain only carbon and hydrogen. Alkenes which are also known as paraffin are saturated hydrocarbons. They do not contain double or triple bonds. Due to this reason, alkanes are relatively inert to chemical reactions and undergo only free radical substitution reaction**.**

$$CH_4 + Br_2 \xrightarrow{uv} CH_3Br + HBr$$

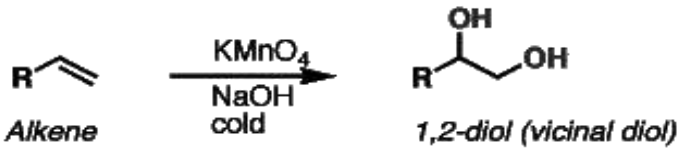
Alkenes are unsaturated hydrocarbons with at least one double bond between two carbon atoms. Alkenes can easily undergo addition reactions at the C=C bond. For example, alkenes undergo hydrogenation, halogenation to form alkanes and dihalides respectively. Alkene also react with potassium permanganate solution in two different condition:

a. In hot acidic medium to form carboxylic and ketone



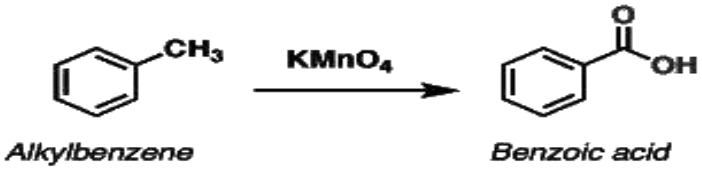
methanoic acid

b. In basic medium to form a diol.



Alkene *1,2-diol (vicinal diol)*

Arene are aromatic hydrocarbons with stable molecular structures. Although arenes have a very high degree of unsaturation, they are relatively inert towards all addition reactions except at a very high pressure and temperature.



Objective

1. To study the chemical properties of an alkane, an alkene and an arene.
2. To differentiate an alkane from an alkene and an arene.

Apparatus

White tile, dropper, parafilm, crucible, labelling paper, wood splint, water bath, burner/lighter, test tube rack, test tube.

Chemicals

Toluene, cyclohexane, cyclohexene, dichloromethane, 0.01 M acidified $KMNO_4$, 4% bromine in dichloromethane.

Procedure

A. Combustion test

1. This procedure will be performed in a fume hood.
2. Place 1 mL of cyclohexane and cyclohexene in two separate evaporating dishes.
3. Ignite both compound simultaneously with a burning wood splint
4. Compare the colour intensity of the flame and the soot given off.

> **Note**: This test is not carried out with toluene because of its carcinogenic properties.

B. Reaction with bromine in dichloromethane

1. This procedure will be performed in a fume hood.
2. Label 6 dry, clean test tubes, A to F.
3. Place 1 mL of cyclohexane in test tubes A and B, 1 mL of cyclohexene in test tube C and D, and 1 mL of toluene in test tubes E and F.
4. Wrap test tube A, C, and E with parafilm.
5. Add 4 to 5 drops of 4% bromine in dichloromethane into each test tube.
6. Keep test tubes A, C, and E in a dark place, and test tubes B, D and F in the sunlight. Leave for 10 minutes.
7. Record the observation.

C. Oxidation with acidified $KMnO_4$

1. Label 3 dry, clean test tubes, G, H and I.
2. Place 1 mL each of cyclohexane, cyclohexene and toluene in test tubes G, H and I respectively.
3. Add 5 drops of acidified $KMnO_4$ into each test tube.
4. Heat the test tube in a water bath at 70 to 80°C for 10-15 minutes.
5. Record the observations.

PRE-LAB STUDY QUESTION EXPERIMENT 3

REACTION OF ALIPHATIC AND AROMATIC HYDROCARBON

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

1. What is the functional group of all tested compounds?

2. Name the mechanism involve in experiment **B**.

3. Show the mechanism and name all the steps involve in experiment **B**.

4. Write the chemical equation involve in experiment **C**.

DATASHEET EXPERIMENT 3

REACTION OF ALIPHATIC AND AROMATIC HYDROCARBON

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

A. Combustion Test

Compound	Observation

B. Reaction with Bromine in dichloromethane

Compound	Observation	
	Dark Room	Sunlight

C. Oxidation with Acidified $KMnO_4$

Compound	Observation	
	Room (before place in water bath	Hot

Lecturer's signature,

_____

EXPERIMENT 4: CLASSIFICATION OF ALCOHOLS

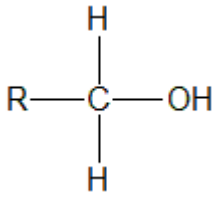
Learning Outcomes

At the end of this practical, students should be to:

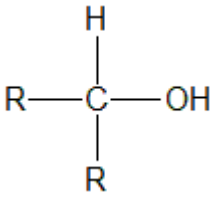
1. Describe the chemical and physical properties of alcohols.
2. Classify an alcohol as primary, secondary, or tertiary.
3. Perform a chemical test to distinguish between the classes of alcohols.
4. Draw the condensed structural formulas of the oxidation products of alcohols

Introduction

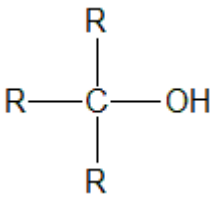
Alcohols are organic compounds containing hydroxyl group, -OH, as the functional group. Alcohols can be classified into:



Primary alcohol (1°)



Secondary alcohol (2°)



Tertiary alcohol (3°)

[R = alkyl group]

The simplest alcohol is methanol. Ethanol is found in alcoholic beverages and preservatives and is used as a solvent. 2-propanol, also known as rubbing alcohol or isopropyl alcohol, is found in astringents and perfumes. Hand sanitizers are used to kill bacteria and viruses that spread colds and flu. As a gel or liquid solution, many hand sanitizers use ethanol as their active ingredient. The amount of ethanol in an alcohol containing sanitizer is typically 60% (v/v), but can be as high as 85% (v/v). The high volume of ethanol can make hand sanitizers a fire hazard in the home because ethanol is highly flammable. When using an ethanol-containing sanitizer, it is important to rub your hands until they are completely dry. It is also recommended that sanitizers containing ethanol be stored in areas away from heat sources in the home.

Lucas reagent, a mixture of concentrated hydrochloric acid and anhydrous zinc chloride, can be used to differentiate the three classes of alcohols. Tertiary alcohols turn cloudy or appear in two layers almost immediately. Secondary alcohols turn cloudy within 5 to 10 minutes whereas primary alcohols do not show any changes.

Alcohol can be oxidised to aldehyde, ketone or carboxylic acid. The product formed depends on the class of alcohol used. Various oxidizing agent such as KMnO4, $Na_2Cr_2O_7$ and H_2CrO_4 can be used. The reaction between alcohols and carboxylic acids will produce esters.

Objective

1. To classify alcohols.
2. To study the chemical properties of alcohols.

Apparatus

Stopper, dropper, test tube, stopwatch, water bath and measuring cylinder (10 ml).

Chemicals

Ethanol, 1-butanol, 2-butanol, alcohol X, Lucas reagent, 2-methyl-2-propanol, cyclohexanol, concentrated H_2SO_4, 0.04 M $Na_2Cr_2O_7$, glacial acetic acid, phenol, bromine water.

Procedure

A. Properties of alcohols

1. Place 10 drops of butanol, 2-butanol, 2-methyl-2-propanol, cyclohexanol, 20% phenol, and the alcohol X into six separate test tubes.
2. Obtain some pH paper.
3. Use a stirring rod to place a drop of each on a piece of pH paper
4. Clean the stirring rod between applications.
5. Compare the color of the pH paper with the color chart on the container to determine the pH. Record the observations.
6. Add 2 ml of water to each test tube.
7. Shake and determine the solubility in water of each alcohol.
8. If the compound is soluble in water, a clear solution appear with no separate layers. If it is insoluble, a cloudy mixture or separate layer will form. Record the observations.

B. Lucas test

1. Place 1 ml of 2-methyl-2-propanol in a test tube.
2. Add 2 ml of Lucas reagent into the test tube.
3. Stopper and shake the test tube.
4. Record the observation and the time taken for the reaction to occur.
5. Repeat the above steps using 2-butanol, 1-butanol and alcohol X. If no change occurs within 10 minutes, place the test tube in a water bath at 70 – 80 °C for 10 minutes.

C. Oxidation

1. Place 5 ml of 0.04 M $Na_2Cr_2O_7$ solution in a test tube.
2. Add 2 to 3 drops of concentrated H_2SO_4 to the solution in the fume cupboard.
3. Add 3 drops of 1-butanol to the mixture and heat it in a water bath at 70 – 80 °C for 10 minutes.
4. Repeat the above steps using 2-butanol, 2-methyl-2-propanol and alcohol X.
5. Record the colour change.

D. Esterification

1. Place 2 ml of ethanol in a dry test tube.
2. Add 1 ml of glacial acetic acid and 3 drops of concentrated H_2SO_4 to the ethanol inthe fume cupboard.
3. Shake the mixture and warm it in a water bath at 60 °C for 5 to 10 minutes.
4. Add 3 ml of distilled water and take a whiff of the vapour released. Describe the smell.

PRE-LAB STUDY QUESTION EXPERIMENT 4

CLASSIFICATION OF ALCOHOLS

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

5. What is the functional group of an alcohol and a phenol?

6. Why are some alcohols soluble in water?

7. Classify each of the following alcohols as primary, secondary or tertiary.

   a. 3-pentanol :_____
   b. 2-methyl-2-butanol :_____
   c. 1-propanol :_____

8. If you add chromate, an oxidizing agent, to each of the following, would a green Cr^{3+} solution be formed?

   a. 3-pentanol :_____
   b. 2-methyl-2-butanol :_____
   c. 1-propanol :_____

9. If an alcohol solution has a pH of 5, would it be a primary alcohol, a secondary alcohol, a tertiary alcohol, or a phenol?

DATASHEET EXPERIMENT 4

CHEMICAL EQUILIBRIA AND LE CHATELIER'S PRINCIPLE

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

A. Properties of alcohols

Alcohol	pH	Solubility in water
1-butanol		
2-butanol		
2-methyl-2-propanol		
Cyclohexanol		
Alcohol X		

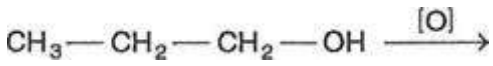
B. Lucas test

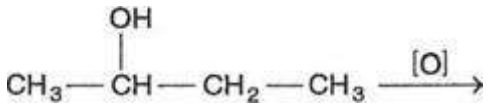
Alcohol	Color change	Time taken (s)	Condensed Structural Formula
Butanol			
2-butanol			
2-methyl-2-propanol			
Cyclohexanol			
Alcohol X			

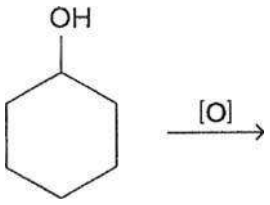
C. Oxidation

Alcohol	Color change	Classification of alcohol	Condensed structural formula of oxidation product
Butanol			
2-butanol			
2-methyl-2-propanol			
Alcohol X			

Q1: Draw the condensed structural formula of the product of the following reactions (if no reaction, write NR):

a. 

b. 

c. 

Q2: Deduce the alcohol X and write its condensed structural formula.

Q3: Describe the smell of esterification process (Part D)

Lecturer's signature,

_____

EXPERIMENT 5: CARBOXYLIC ACID AND ITS DERIVATIVES

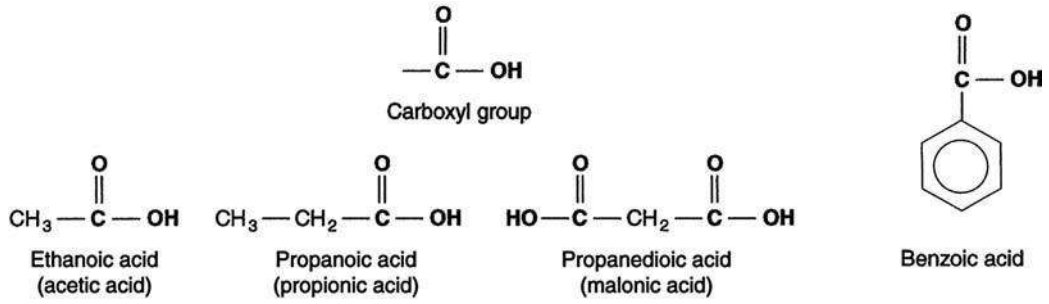
Learning Outcomes

At the end of this practical, students should be to:

1. Write the condensed structural formulas of carboxylic acids and their derivatives.
2. Determine the solubility and acidity of carboxylic acids and their derivatives.
3. Prepare esters and identify their characteristic odors.

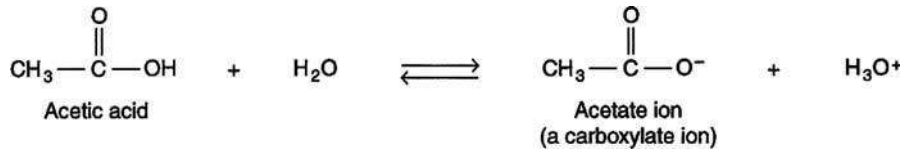
Introduction

A salad dressing made of oil and vinegar tastes sour due to the vinegar, which contains acetic acid (ethanoic acid). The sour taste of fruits such as lemons is due to acids such as citric acid. Some face creams contain alpha hydroxy acids such as glycolic acid. All these acids just described are carboxylic acids, which contain the carboxyl group: a carbonyl group attached to a hydroxyl group. A dicarboxylic acid, such as malonic acid, has two carboxylic acid functional groups. The carboxylic acid of benzene is called benzoic acid.



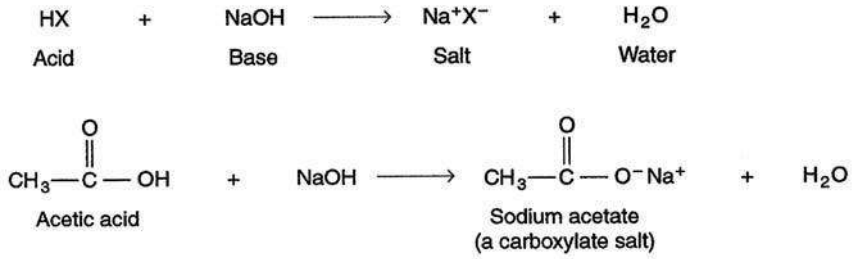
Ionization of Carboxylic Acids in Water

Carboxylic acids are weak acids because the carboxylic acid group ionizes slightly in water to give a hydrogen ion (H^+) and a carboxylate ion as shown in the equation below. The polarity of the carboxylic acid group makes acids with one to four carbon atoms soluble in water.



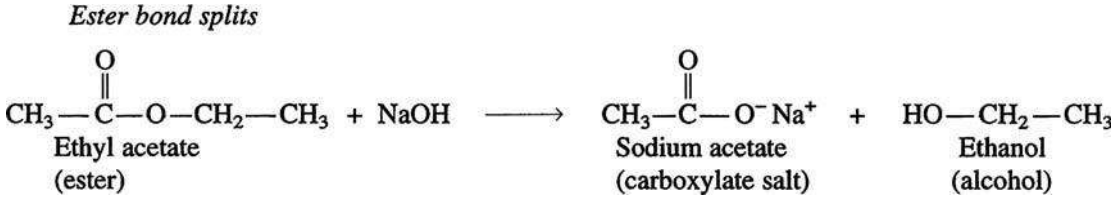
Neutralization of Carboxylic Acids

An important feature of carboxylic acids is their neutralization by bases such as sodium hydroxide to form carboxylate salts and water. Neutralization is the reaction of an acid with a base to give a salt and water. Even insoluble carboxylic acids with five or more carbon atoms can be neutralized to give salts that are usually soluble in water. For this reason, acids used in food products or medications are in their soluble carboxylate salt form rather than the acid.



Saponification

When an ester is hydrolyzed in the presence of a base, the reaction is called *saponification*. The products are the salt of the carboxylic acid and the alcohol. Although the ester is usually insoluble in water, the salt and alcohol (if short-chain) are soluble.



Objective

1. To study the chemical properties of carboxylic acid and its derivatives.

Apparatus

Beaker, dropper, scissor, test tube, heating mantle, thermometer, measuring cylinder, round bottom flask (100 ml), Liebig's condenser, Hirsch funnel and suction pump.

Chemicals

Ice, diethyl ether, boiling chips, ethyl benzoate, blue litmus paper, propanoyl chloride, 5.0 M HCl, 2.5 M NaOH, 5 M NaOH, 0.6 M AgNO3, glacial acetic acid, benzoic acid, stearic acid.

Procedure

A. Solubility of carboxylic acid

1. Label 9 dry test tubes
2. Place:
    i. 3 ml of diethyl ether into test tube 1,2 and 3
    ii. 3 ml of water into test tubes 4, 5 and 6.
    iii. 3 ml of 2.5 M NaOH into test tubes 7, 8 and 9.
3. Add 6 drop of glacial acetic acid to test tubes 1, 4 and 7.
4. Shakes the tubes. If the acid dissolves, continue adding glacial acetic acid one drop at time, until the volume of solution reaches 6 ml.
5. Observe and record the solubility as soluble, slightly soluble or insoluble.
6. Add benzoic acid in small portions into test tubes 2,5 and 8. Shake the test tubes

after each addition until each solution is saturated.

7. Record the observation
8. Repeat step 5 using stearic acid in test tube 3, 6 and 9.

B. Hydrolysis of acid chloride

1. Carefully add 5-10 drops of propanoyl chloride (a drop at time) into 5 ml of water in a fume cupboard. Observation: Exothermic reaction, steamy acidic gas release.
2. Record the temperature of the reaction.
3. After the reaction has completed, test the solution with pH paper.
4. Add 1 ml of 0.6 M $AgNO_3$.
5. Record the observation.

C. Saponification of ester

1. In a 100 ml round bottom flask add mix 5 ml of ethyl benzoate. Record the appearance and odor of the ester.
2. Mix ethyl benzoate with 15 ml of 5 M NaOH. Add 3-4 boiling chips into the flask. Record the observation.
3. Fit the flask with Liebig's condenser as shown in Figure 5.1.
4. Reflux the mixture for 30 minutes.
5. Cool the flask to room temperature before placing it in ice bath.
6. Dilute the contents with 5 ml of water. Record any changes in the odor of the ester.
7. Add 5.0 M HCl in small volumes until the blue litmus paper changes to red.
8. Filter the product through a Hirsch funnel.
9. Recrystallize by dissolving it in 5 ml of hot water. Cool it in an ice bath.
10. Collect the product by filtering through a Hirsch funnel. Record the observation.

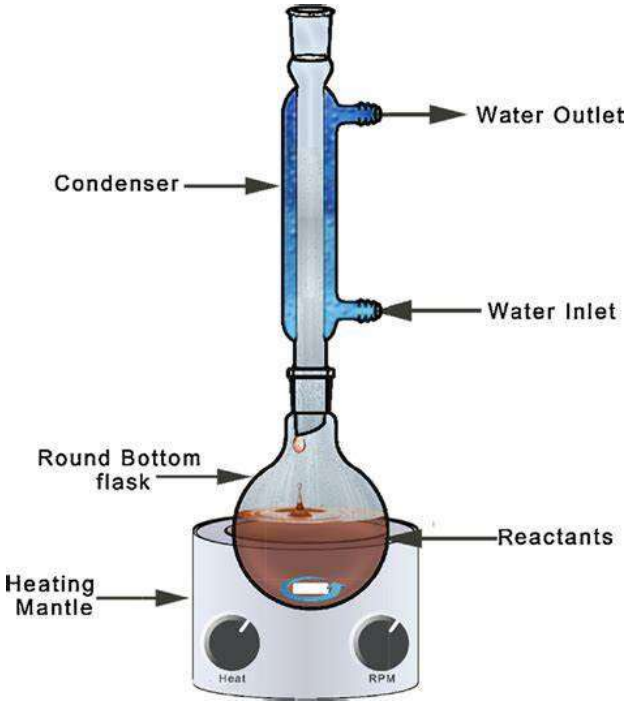


Figure 5.1: Set up for saponification process using Liebeig condenser.

PRE-LAB STUDY QUESTION EXPERIMENT

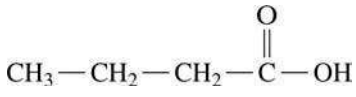
CARBOXYLIC ACID AND ITS DERIVATIVES

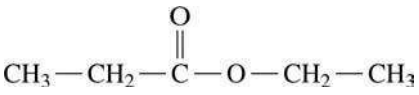
NAME :_____ DATE EXPERIMENT :_____

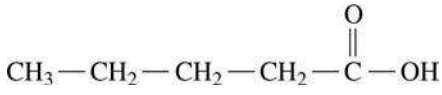
STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

1. Give the IUPAC name for each of the following:

a)  _____

b)  _____

c)  _____

2. Write the condensed structural formula of the organic product for propanoic acid reacting with each of the following:

   a) NaOH

   b) CH_3OH

   c) H_2O

3. Write the condensed structural formula of the organic products for ethyl ethanoate when it reacts with each of the following:

   a) NaOH

   b) HO_2 and HCl

DATASHEET EXPERIMENT 5

CARBOXYLIC ACID AND ITS DERIVATIVES

NAME :_____ DATE EXPERIMENT :_____

STUDENT ID :_____ GROUP :_____

GROUP MEMBERS :_____

A. Solubility of carboxylic acid

	Acetic acid	Benzoic acid	Stearic acid
Condensed structural formula			
Solubility in ether			
Solubility in water			
Solubility in NaOH			

Q1 Write the balanced equation using condensed structural formulas for the reaction of acetic acid with NaOH.

Q2 Write the balanced equation using condensed structural formulas for the reaction of benzoic acid with NaOH

B. Hydrolysis of acid chloride

	H_2O	$AgNO_3$
Temperature (°C)		
pH		
Observation		

C. Saponification of ester

1. Condensed structural formula of ethly benzoate
2. Appearance and odor
3. Changes in appearance and odor
4. Equation for saponification
5. Observations
6. Condensed structural formula of the compound formed by adding HCl

Lecturer's signature,

_____