

**COURSE  
GUIDE**

**CHM 292 ORGANIC AND PHYSICAL (PRACTICAL  
MANUAL)**

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**NAME:** \_\_\_\_\_

**PROGRAMME:** \_\_\_\_\_

**MATRIC. NO:** \_\_\_\_\_

**LEVEL:** \_\_\_\_\_

**GROUP:** \_\_\_\_\_

**DATE** \_\_\_\_\_

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## **INTRODUCTION**

This practical manual is designed to guide both student and tutor when carrying practical. It contained following safety measures:

- Wear approved eye protection in the laboratory at all times.
- Never smoke in the laboratory or use open flames in operations that involve low boiling flammable solvents.
- Consider all chemicals to be hazardous and minimize your exposure to them.
- Exercise great care when working with glass and when inserting or removing thermometers and glass tubing
- Wear appropriate clothing in the laboratory
- Dispose all chemicals appropriately
- Never work alone in the laboratory or perform unauthorized experiments

In line with the above, no student should be allowed into the laboratory without possessing the practical manual.

The practical guide is an extract from CHM 392 course material as such it covers practical aspect as contained in Physical Chemistry II (CHM 201), and Organic Chemistry II.

## **EXPECTATIONS FROM THIS MANUAL**

The practical manual is in two section; Organic Chemistry and Physical Chemistry. It contains step by step procedure on how to prepare some organic compounds and further purify or even analyzed them. Students can also learn how to set up a simple paper chromatography which is essential knowledge require understanding the general principle of chromatography. Other experiment includes Qualitative analysis of elements in organic compounds as reference test (group classification test). The physical chemistry aspect of this manual contains procedures such as PH measurement and determination of some colligative properties.

## **THE AIM OF THIS COURSE**

The course is aim at enhancing understanding of practical aspect of both organic and physical chemistry which was earlier detailed in practical chemistry II. It is another opportunity for students to be exposed to general laboratory techniques.

## WORKING THROUGH THIS COURSE

It is expected that students who participate in all the experiments detailed in the practical manual booklet, would have better understanding of practical chemistry II (CHM 292) and by extension organic and physical chemistry. No student should afford to miss out in the practical session as this would expose them on how to carry out some laboratory procedures. Students using this book should endeavor to consult the main practical course guide which would be issued by National Open University of Nigeria (NOUN).

## THE MANUAL

Being an extract, this manual consists of;

1. Introduction to practical
2. Experimental procedures
3. Reference.

Sections of practicals in this manual are:

### Section1 Organic Chemistry

- 1.0 Preparation of Esters
- 2.0 Preparation of Acetone from 2-Propanol
- 3.0 Oxidation of Ethanol to Ethanal using CuO
- 4.0 Vinegar Analysis
- 5.0 Paper Chromatography
- 6.0 Dehydration of Alcohols (Dehydration of Cyclohexanol)
- 7.0 Qualitative analysis for elements (for reference only)
- 8.0 Functional Group Classification Tests

### Section 2 Physical Chemistry

- 1.0 pH Measurement
- 2.0 Temperature measurement
- 3.0 Heat of dissolution
- 4.0 Heat of Neutralisation
- 5.0 Demonstration of Partition Coefficient in two Immiscible Solutions
- 6.0 Ideal gas law: measuring the molar volume of a gas and the universal gas constant.

Section one of this material is a detail experiments in organic chemistry which consist of 8 different experimental procedures. Experiment 1 and 2 cover preparation of esters and aldehyde followed by Oxidation of

Ethanol to Ethanal using CuO. Analysis of vinegar is contained in experiment 4. Others are: Paper Chromatography 5, Dehydration of Alcohols (Dehydration of Cyclohexanol) 6, Qualitative analysis for elements (for reference only) 7, and Functional Group Classification Tests, experiment 8.

The second section deals with pH Measureme experiment 1, Temperature measuremen in 2, while Heat of dissolution and Heat of Neutralisation are contained in experiment 3 and 4 respectfully.

Demonstration of Partition Coefficient in two Immiscible Solutions is explained in experiment 5, whereas the Ideal gas law: measuring the molar volume of a gas and the universal gas constant rounded up in experiment 6.

### **PRESENTATION OF SCHEDULE**

The inscriptional procedures in this manual should be adhered to, the practical session would hold once in a week. Each session will introduce at least one novel procedure depending on the procedural encumbrance as may be assess by the technologist. It is therefore students should endeavor to participate in all the practical sessions.

### **ASSESSMENT**

Practical is one of the assessable aspect of CHM 292, it carries 20 % of the total marks awarded during assessing student's performance on the parent course.

My advice is, you shouldn't miss the practical sessions now that you know you can be assessed.

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## Practical 1      Organic Chemistry

### Contents

- 1.1 Introduction
- 1.2 Safety precautions
- 1.3 Preparation of Esters
  - 1.3.1 Apparatus/Reagents Required
  - 1.3.2 Procedure
- 1.4 Conclusion
- 1.5 Summary
- 1.6 Tutor-marked assignment

### 1.1 Introduction

When an organic acid, R-COOH, is heated with an alcohol, R'-OH, in the presence of a strong mineral acid, the chief organic product is a member of the family of organic compounds known as esters. The general reaction for the esterification of an organic acid with an alcohol is:  $R-COOH + HO-R' \leftrightarrow R-CO-OR' + H_2O$

For further details on esterification, please refer to CHM 292 Course material. However some examples of esters, constituents and their characteristic aroma are provided in the table below. Synthesize at least two of the esters, and note their aromas. Different students might synthesise different esters, as directed by the instructor, and compare the odours of the products.

**Table 1: Common Esters and their Constituents**

Ester	Aroma	Constituents
n-propyl acetate	Pears	n-propyl alcohol/acetic acid
methyl butyrate	Apples	methyl alcohol/ butyric acid
isobutyl	Rum	isobutyl alcohol/propionic
octyl acetate	Oranges	n-octyl alcohol/acetic acid
methyl	Grapes	Methyl alcohol/2-aminobenzoic
isoamyl acetate	Bananas	isoamyl alcohol/acetic acid
ethyl butyrate	Pineapples	ethyl alcohol/butyric acid
benzyl acetate	Peaches	benzyl alcohol/acetic acid

## 1.2 Safety precautions

- Protective eyewear approved by your institution must be worn at all times while you are in the laboratory.
- Most of the organic compounds used or produced in this experiment are highly flammable.

Heating will be done using a hotplate, and no flames will be permitted in the laboratory.

- Sulphuric acid is used as a catalyst for the esterification reactions. Sulphuric acid is dangerous and can burn skin very badly. If it is spilled, wash immediately before the acid has a chance to cause a burn, and inform your instructor.
- The vapours of the esters produced in this experiment may be harmful. When determining the odours of the esters produced in this experiment, do not deeply inhale the vapours. Merely waft a small amount of vapour from the ester toward your nose.
- NaOH solution is highly corrosive to eyes and skin. Wash immediately if spilled.
- Most of the reactants and products in this choice are highly flammable, and no flames are permitted in the lab during this experiment.

## 1.3 Preparation of Esters

### 1.3.1 Apparatus/Reagents Required

Hot plate; 50% sulphuric acid; assorted alcohols and organic acids, as provided by the instructor, for the preparation of fruit and flower aromas; methyl salicylate; 20% NaOH; disposable 4 mL plastic pipette with stem cut to 2.5 cm.

### 1.3.2 Procedure

Set up a water bath in a 250-mL beaker on a hotplate in the exhaust hood. Adjust the heating control to maintain a temperature of around 70°C in the water bath. Some common esters, and the acids/alcohols from which they are synthesized, were indicated in the Table 1 above.

To synthesize the esters, mix 3-4 drops (or approximately 0.1g if the acid is a solid) of the appropriate acid with 3-4 drops of the indicated alcohol on a clean, dry watch glass. Add 1 drop of 50% sulphuric acid to the mixture on the watch glass (caution!). Use the tip of a plastic pipette to stir the mixture on the watch glass, and then suck as much as possible

of the mixture into the pipette. Place the pipette, tip upward, into the warm-water bath, and allow it to heat for approximately 5 minutes.

#### 1.4 Conclusion

Esters can be found as both natural and artificial products. Esters can be prepared in the laboratory using different materials.

#### 1.5 Summary

In this practical, students were able to prepare esters in the laboratory.

<b>RESULT</b>					
<b>Name of organic acid</b>	<b>Name of an alcohol</b>	<b>Volume of acid</b>	<b>Volume of alcohol</b>	<b>Aroma of the product</b>	<b>Name of the esters formed</b>

--	--	--	--	--	--

**1.6 After Lab. activities**

Esters are frequently used as additives in commercial products found at home. From what you prepared today, list two common home use products that have similar aroma as your ester.



## Practical 2 Preparation of Acetone from 2-propanol

### Contents

- 2.1 Materials
- 2.2 Procedure
- 2.3 Summary
- 2.4 After laboratory activities

### Before Laboratory Activities

#### 2.1 Materials

- 20 mL 70% 2-propanol (isopropyl alcohol)
- Distilled water
- 100 mL acidic dichromate ( $K_2Cr_2O_7/H_2SO_4$ ) solution
- Distillation apparatus including thermometer
- Ice/water bath.

#### 2.2 Procedure

Prepare an ice/water bath; this may be conveniently done in a large (i.e., > 500 mL) beaker. Place 20 mL of 70% 2-propanol in a 250-mL beaker, and add 20 mL of distilled  $H_2O$ . Stir to mix, and cool the beaker in an ice bath to about  $10^\circ C$ . With the solution still in the ice bath, add all at once, 100 mL of "acidic dichromate" (set two types of the experiment for each sample of the chromates) solution (**CAUTION**: Corrosive!). In a few seconds the mixture will turn dark, followed by a rather sudden rise in temperature to  $50-60^\circ C$ . Stir the mixture (still in the ice bath!) until its temperature has fallen to below  $50^\circ C$ . **NOTE**: Do not use the thermometer as a stirring rod. Pour the mixture into a 250 mL (or larger) distilling flask using a funnel to prevent spilling any. Assemble a distillation apparatus as demonstrated by your lab instructor. Use a graduated cylinder as the receiver. Heat gently, after 10-15 minutes, the liquid should begin to boil and drops of acetone begin to collect in the receiver. Record the temperature when the first drop appears. Continue the distillation until at least 5 mL of acetone has collected.

Record the temperature again, and then stop the distillation. Measure the volume of acetone obtained.

### 2.3 Summary

At the end of this practical, acetone was prepared from 2-propanol in the laboratory.

#### In-class activities

<b>RESULT</b>					
<b>Chromate type</b>	<b>Initial Temp. of Xtant</b>	<b>Final Temp of Xtant</b>	<b>Initial Temp. Distillate</b>	<b>Final Temp. of Distillate</b>	<b>Volume of Distillate</b>
1.					
2.					
3					

## 2.4 After Laboratory Activity

From the result of the volume obtained, calculate the percentage yield of the acetone (**percent yield =  $\frac{\text{volume of (distillate/volume sample product)}}{\text{sample product}} \times 100$** )

Give the complete reaction for acidification of the chromate

## Practical 3 Preparation of Acetone from 2-propanol

### Contents

- 3.1 Materials
  - 3.1.1 Equipment
- 3.2 Procedure
- 3.3 Conclusion
- 3.4 Summary
- 3.5 Tutor-marked assignment

### Before Laboratory Activities

In this experiment, you will study how to prepare acetaldehyde from ethanol.

#### 3.1 Materials:

- 10 ml Ethanol  $C_2H_5OH$
- Cu wire

#### 3.1.1 Equipment:

- Safety glasses
- Test tube + holder on a stand Tongs
- Bunsen burner

#### 3.2 Procedure

- a. Add the ethanol to the test tube and place the test tube on the stand.
- b. Heat the Cu wire in an open fire until it becomes black (CuO)

#### Confirmation Test

Take 0.5 ml of your product and add to equal volume of Fehling's reagent and record your observation.

#### 3.3 Conclusion

Aldehydes and ketones are polar compounds which you can prepare in your laboratory.

### 3.4 Summary

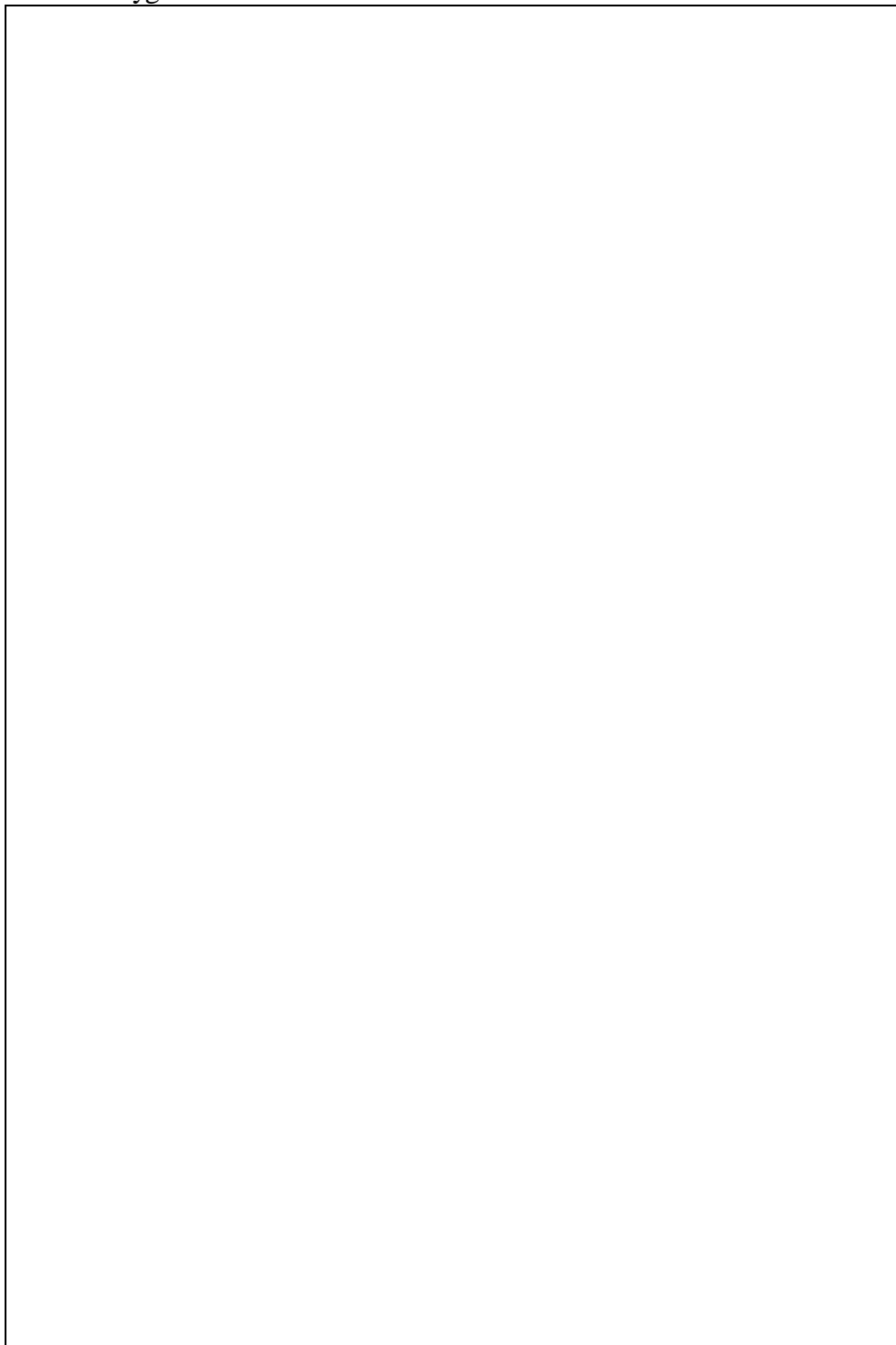
In this practical, attempt has been made to prepare ethanol using copper oxide in the laboratory.

#### In-Laboratory activities

<b>RESULT</b>					
<b>Volume of ethanol (_____ml)</b>	<b>Weight of the Cu wire(_____mg)</b>	<b>Colour of Cu before heating</b>	<b>Colour of Cu after heating</b>	<b>Observation when Cu + ethanol</b>	<b>Observation when Product + Fehling's reagent</b>
Weigh of Cu 1.					
2.					

**3.5 After Lab. activities**

1. Draw the structure of acetone and propanal
2. Write the chemical equation for the reaction between CuO and ethanol.
3. Write the chemical equation for the reaction between copper and oxygen



## Practical 4 Vinegar Analysis

- 4.1 Introduction
- 4.2 Materials
- 4.3 Procedure
- 4.4 Calculations
- 4.5 Conclusion
- 4.6 Summary
- 4.7 Tutor-Marked Assignment

### Content

#### Before Laboratory Activities

##### 4.1 Introduction

Many commercial products contain or are low percent acid solutions. Vinegar is a water solution that is 4 to 5 per cent by weight acetic acid,  $\text{CH}_3\text{COOH}$ . Many manufacturers add flavourings and colour to make the product sell better. Vinegar is commonly used in flavouring and preservation of food. This is because the acidity adds “tang” and inhibits bacterial growth.

##### 4.2 Materials

1. Clean and dry the following material:

500 mL plastic bottle, 100 mL beaker, 25.00 mL volumetric pipet, 250.0 mL volumetric flask, 250 mL Erlenmeyer flask, 50 mL Burette.

##### 4.3 Procedure

1. Measure exactly 25.00 mL of vinegar into a clean 250.0 mL volumetric flask.
2. Dilute the vinegar with 50 mL of de-ionized water to the mark on the volumetric flask.
3. Stopper the flask and mix the solution well. (Invert the solution slowly for at least 10 times to completely mix the contents).
4. Transfer the dilute vinegar solution to a clean and dry 500 mL plastic bottle and label it as A.
6. Pour about 25 mL of the dilute vinegar solution into 100 mL beaker.
7. Add 2 drops of phenolphthalein indicator solution and swirl the flask to thoroughly mix the solution.
8. Rinse your 50 mL burette several times with a few milliliters of your standardized sodium hydroxide solution.

9. Fill the burette with your standardized sodium hydroxide solution and record the volume of the burette to the nearest 0.01 mL
10. Titrate the acid sample to a faint pink end point.
11. Record the final volume of the burette to the nearest 0.01 mL.
12. Repeat the titration procedure described above for at least two more trials.

### In Laboratory activities

RESULTS			
Volume of vinegar ( _____ mL)			
Titre values	Initial Volume of NaOH (A)	Final Volume of NaOH (B)	Volume of NaOH used (A-B)
First titre value			
Second titre value			
Third titre value			

### 4.4 Calculations

Moles of base = (molarity of base) X (liters of base)

*moles of acid (diluted vinegar) = moles of base*

$$\text{molarity of diluted vinegar} = \frac{\text{moles of acid (diluted vinegar)}}{\text{liters of acid (diluted vinegar)}}$$

The molarity of the original vinegar solution can be found by keeping in mind that the vinegar has been diluted ten times (from 25.00mL to 250.0 mL) to obtain the diluted vinegar, whose molarity has been determined by titration with standardised NaOH.

*grams of HCH3O2*

$$\begin{aligned} & \frac{\text{grams of vinegar}}{\text{mol HC2O2}} \\ &= \frac{\text{grams of vinegar}}{\text{liter of vinegar}} \times \frac{\text{g.HC2H3O2}}{\text{mol HC2H3O2}} \times \frac{\text{l. vinegar}}{\text{mol. vinegar}} \times \frac{\text{mol. vinegar}}{\text{g. vinegar}} \\ & \text{Density of vinegar} - 1.0052\text{g/mL} = \frac{\% \text{weigh HC2H3O2}}{\text{weight of vinegar}} \\ &= \frac{\text{g.HC2H3O2}}{\text{grams of vinegar}} \times 100 \end{aligned}$$

### 4.5 Conclusion

Vinegar is essentially a solution of acetic acid, CH<sub>3</sub>CO<sub>2</sub>H, in water.

Vinegar is supposed to have 4 g of acetic acid per 100 mL of vinegar or 4%, does your vinegar meet this requirement?



#### 4.6 Summary

At the end of this exercise, students were able to determine the percent by weight of acetic acid in vinegar and also perform an acid-base titration.

#### 4.7 Tutor-Marked Assignment

From your data, calculate the molarity of the original vinegar solution

## Practical 5      Paper Chromatography

### Contents

- 5.1 Introduction
- 5.2 Materials
- 5.3 Procedure
- 5.4 Conclusion
- 5.5 Summary
- 5.6 Tutor-marked assignment

### Before Laboratory Activities

#### 5.1 Introduction

Chromatography is a separation and analytical technique widely used in chemistry and the biological sciences. Most things that occur in nature are a mixture of substances which can only be separated or analyzed using any of the techniques known. In this unit, you will be involved in practical exercise using paper chromatography. Paper chromatography is one of the most common types of chromatography in which filter paper serves as a support for immobile liquid phase. Removing liquid flows between the fibres of the cellulose but these are not the stationary phase.

For more details on paper chromatography please refer to your CHM 292 Course material

#### 5.2 Materials

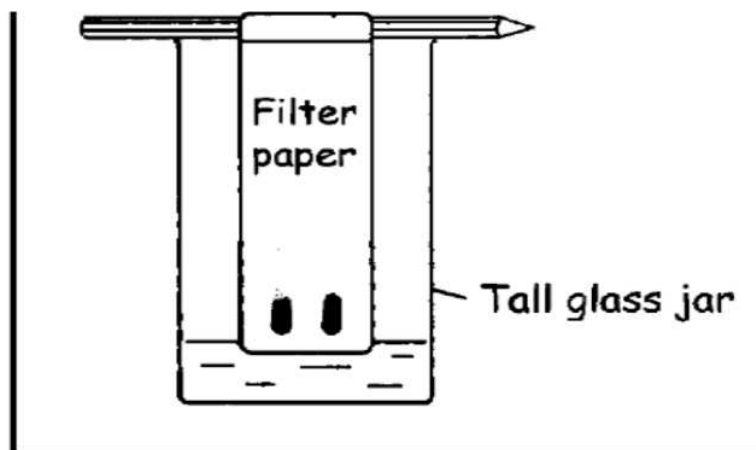
- 1000 ml beaker
- Chromatography Paper
- Small Capillary tubes
- Note book and pencil
- Alanine, Leucine, Lysine and Valine
- Amino Acid Developing Solution

#### 5.3 Procedure

Set up an apparatus as shown in figure below, then Work with a partner; obtain a sheet of 13 x 18.5 cm Whatman NO. 1 chromatography paper. When you handle this paper, hold it only on one of the long (18.5 cm) sides, which will be considered the “top” of the sheet. The amino acids from your fingers will contaminate the paper and lead to erroneous results if it is touched on the “bottom”. Lay the sheet of chromatography paper on a piece of notebook paper, and draw a line in pencil, not pen, 1.5 cm above the bottom. Make small marks along the line using the

dimensions given in by your instructor. Write labels at the top. Use the small capillary tubes provided to make four spots, one of each amino acid (alanine, leucine, lysine and valine), along the pencil line. Follow the labels written at the top of the sheet. Your instructor will assign each of you an “unknown” sample that contains one or more of these four amino acids. Spot this solution on the paper as well. For every spot you make, touch the capillary to the surface of the paper quickly and lightly so that the spot is approximately 2-3 mm in diameter. Allow the spot to dry, and then reapply the solution at the exact same place, again touching the paper quickly and lightly. Allow the spot to dry, and repeat one more time. After all the six solutions have been applied to the paper in this manner; allow the spots to dry for five minutes. Working together, roll the paper into a cylinder with the spots on the outside, and then staple it so that the edges do not overlap or touch as shown in figure below. Pour 50 mL of the amino acid developing solution into a 1000 mL beaker. The developing solvent is comprised of a four-to-one mixture of 1-butanol and glacial acetic acid that has been saturated with water. Position the cylinder inside the beaker with the bottom edge immersed in the solvent. Make sure the paper does not touch the glass. Place a piece of aluminum foil over the mouth of the beaker. Allow the chromatogram to develop undisturbed for 60 to 75 minutes. Do not move the beaker while the chromatogram is developing! When you remove the paper from the beaker, mark the solvent front with a pencil. Set the cylinder on notebook paper, and allow it to dry. When the chromatogram is completely dry, remove the staples, and hang it using a clamp and stand. Wearing gloves evenly coat the paper using the ninhydrin spray. Do not allow the paper to become dripping wet. Place the chromatogram in an oven set at 80° for about 5 minutes. Circle the spots with a pencil. Measure the distance from the origin to the center of each spot and the distance from the origin to the solvent front. Use your result to calculate the  $R_f$  value for each unknown sample.

$$R_f = \frac{\text{Distance the solute moves}}{\text{Distance the solvent front moves}}$$

**In Laboratory activities****Figure1: Paper Chromatography**

Sample	Solvent front	Solute distance	$F_r$ value	Inference
<b>A</b>				
<b>B</b>				
<b>C</b>				
<b>D</b>				
<b>E</b>				
<b>F</b>				

**5.4 Conclusion**

Paper chromatography is a technique employed in separating various organic compounds including amino acids. The reference value obtained for each compound is specific to that compound and can be used to determine the unknown compound.

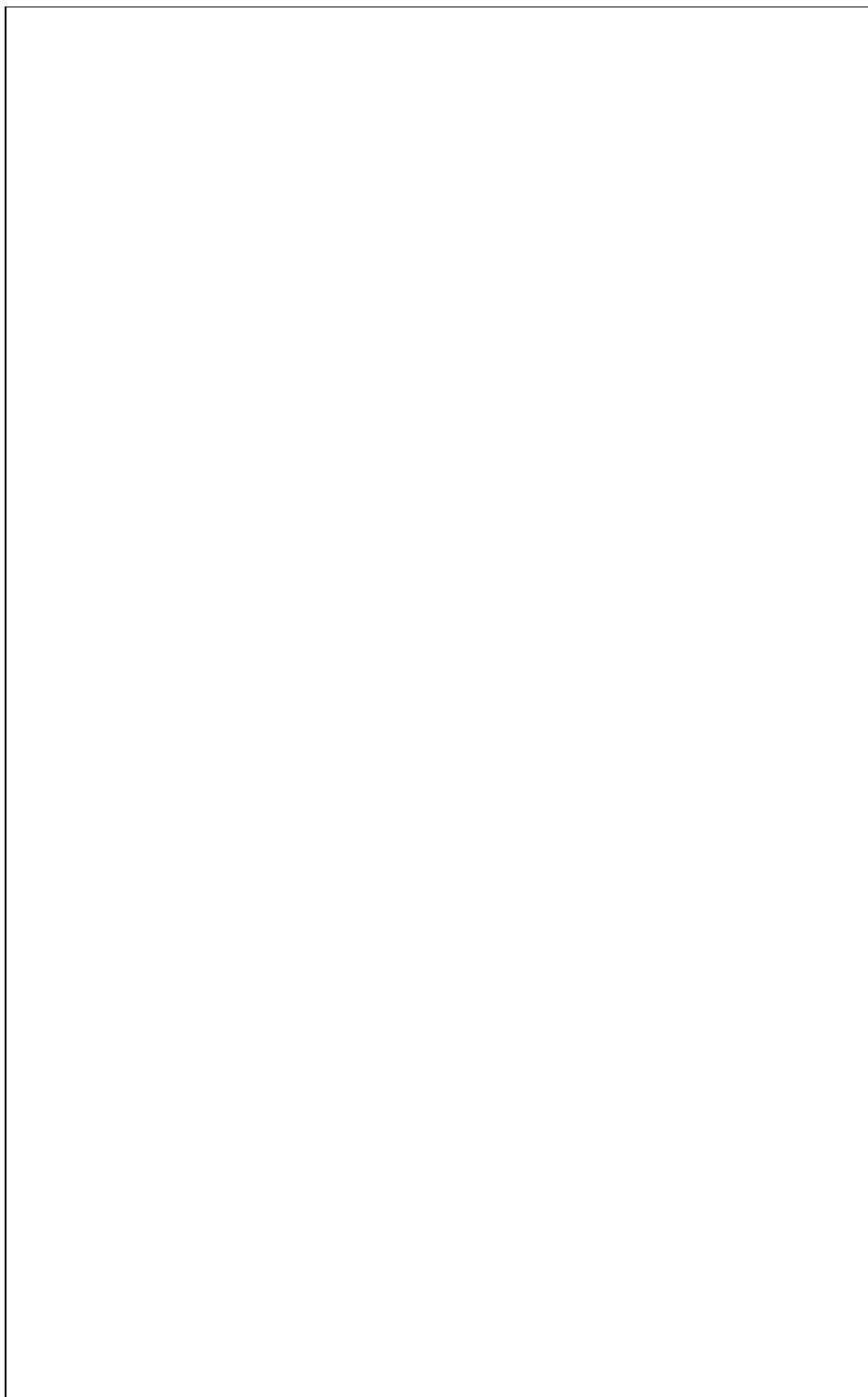
**5.5 Summary**

Students were able to identify each amino acid from the sample of four unknown provided by their instructor.

**5.6 After Laboratory activities**

Give the structural formula of the amino acid that you have identified. What would have been the outcome if pen was used instead of pencil

**List 3 organic samples you know**

A large, empty rectangular box with a thin black border, intended for the student to list three organic samples they know.

## Practical 6 Dehydration of Alcohols (Dehydration of Cyclohexanol)

### Contents

#### Before Laboratory activities

- 6.1 Introduction
- 6.2 Materials
- 6.3 Procedure
  - 6.3.1 The product analysis
- 6.4 Conclusion
- 6.5 Summary
- 6.6 Tutor-marked assignment

#### 6.1 Introduction

The dehydration reaction will be illustrated by the conversion of cyclohexanol to cyclohexene. The choice of cyclohexanol as starting material is based on certain considerations; refer to course materials. Caution: Cyclohexanol is a volatile and flammable liquid and is an irritant. No flames will be allowed in the lab. Wear gloves while handling these chemicals. Concentrated phosphoric acid (or sulphuric acid) is strongly corrosive and toxic -- wear gloves while handling it, and be sure to wash your gloves and your hands immediately after handling it. Sodium sulfate is an irritant; gloves are recommended.

#### In Laboratory activities

#### 6.2 Materials

- cyclohexanol simple distillation set up
- 85% phosphoric acid,  $\text{H}_3\text{PO}_4$  (or conc.  $\text{H}_2\text{SO}_4$ )
- Beakers (150mL, 250mL)
- 10%  $\text{NaHSO}_3$  10-mL
- Graduated cylinder
- Cold 0.50 %  $\text{KMnO}_4$
- Erlenmeyer flask (50 mL)
- $\text{Br}_2/\text{CCl}_4$
- Round bottom flask (25 mL, 50 mL)
- Grease condenser
- Ice thermometer
- $\text{CaCl}_2$  (drying agent)
- Separatory funnel
- Saturated  $\text{NaCl}$  solution
- Rubber tubing (2)

- Glass adaptor (2)
- Thermometer adaptor
- heating mantle

### 6.3 Procedure

Set up a simple distillation as shown in figure 2 below. Add 8.00 ml ( $D = 0.96 \text{ g/ml}$ ) of cyclohexanol and 2 ml of concentrated sulphuric acid (or 5 ml of concentrated phosphoric acid) to a 50 -ml round bottomed flask. Mix the content thoroughly by swirling before connecting the flask to the distillation setup. Add two boiling stones, and heat the flask gently so that the temperature of the distilling vapor does not exceed  $100 \text{ }^\circ\text{C}$ . Continue the distillation until only a few milliliters ( $< 2 \text{ ml}$ ) of high boiling residue remain in the flask. If white fumes appear near the end of the distillation, stop heating at once by lowering the heating mantle. (NOTE - these fumes are oxides of sulfur,  $\text{SO}_2$ , if sulphuric acid is being used).

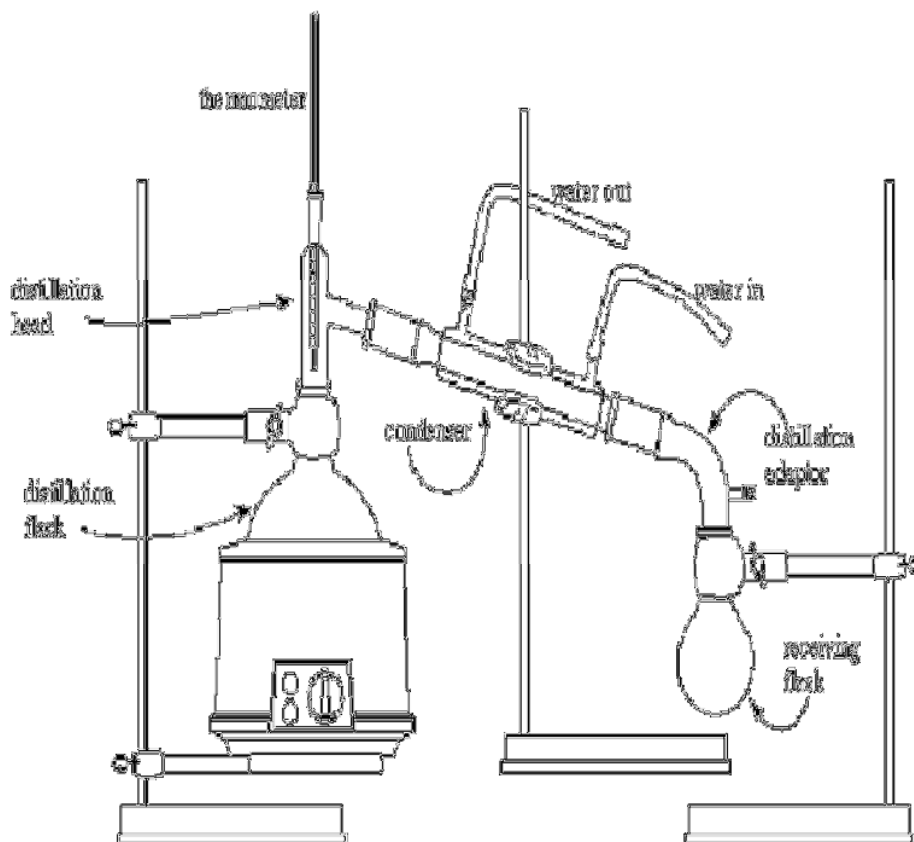


Figure 2: A Simple Distillation Set Up

Note that the distillate in the receiver consists of two layers. Transfer the distillate to a small separator funnel and add 2 ml of saturated sodium chloride solution (to decrease the solubility of cyclohexene in the water layer), then add drop-by-drop 2 ml of 10% sodium bicarbonate solution (to neutralize the traces of any remaining unreacted acid). Swirl or shake the mixture gently. Allow the layers to separate, and then draw off and discard the lower layer (aqueous layer). Pour the upper layer (organic layer - crude cyclohexene) at the top of the separator funnel into a small, dry 50- ml Erlenmeyer flask. Add half a teaspoon of anhydrous calcium chloride (used to dry, remove, traces of water) to the cyclohexene and allow it to stand for 10-15 min, swirling it occasionally. The product should be clear, not cloudy.

### 6.3.1 The product analysis

- I) Baeyer test - To make sure the product is alkene, test your product with potassium permanganate solution (cold  $\text{KMnO}_4$ ), which is a test for the presence of double bond in compound. Potassium permanganate, a purple solution loses colour with alkenes and forms manganese dioxide, a brown precipitate. Place 5-6 drops of your alkene product in a small test tube and add 1-2 drops of  $\text{KMnO}_4$  solution. Swirl the tube to mix the reagents and leave it for observations. Record your observations.
- II) Bromination test - Place 5-10 drops of your alkene product in a small test tube and test with drop- wise bromine (decolouration) for observations. Record your observations.



<b>RESULT</b>	
<b>Reactions</b>	<b>Observation</b>
Filtrate + NaCl (aq)	
Filtrate + CaCl <sub>2</sub> (aq)	
Filtrate + KMnO <sub>4</sub>	
Filtrate + Bromine	

## 6.4 Conclusion

Dehydration of an alcohol is a common method of introducing unsaturation into an organic compound. This type of reaction belongs to the important class of organic reactions called elimination reactions. In the elimination of water from an alcohol, the more highly substituted alkene products are formed.

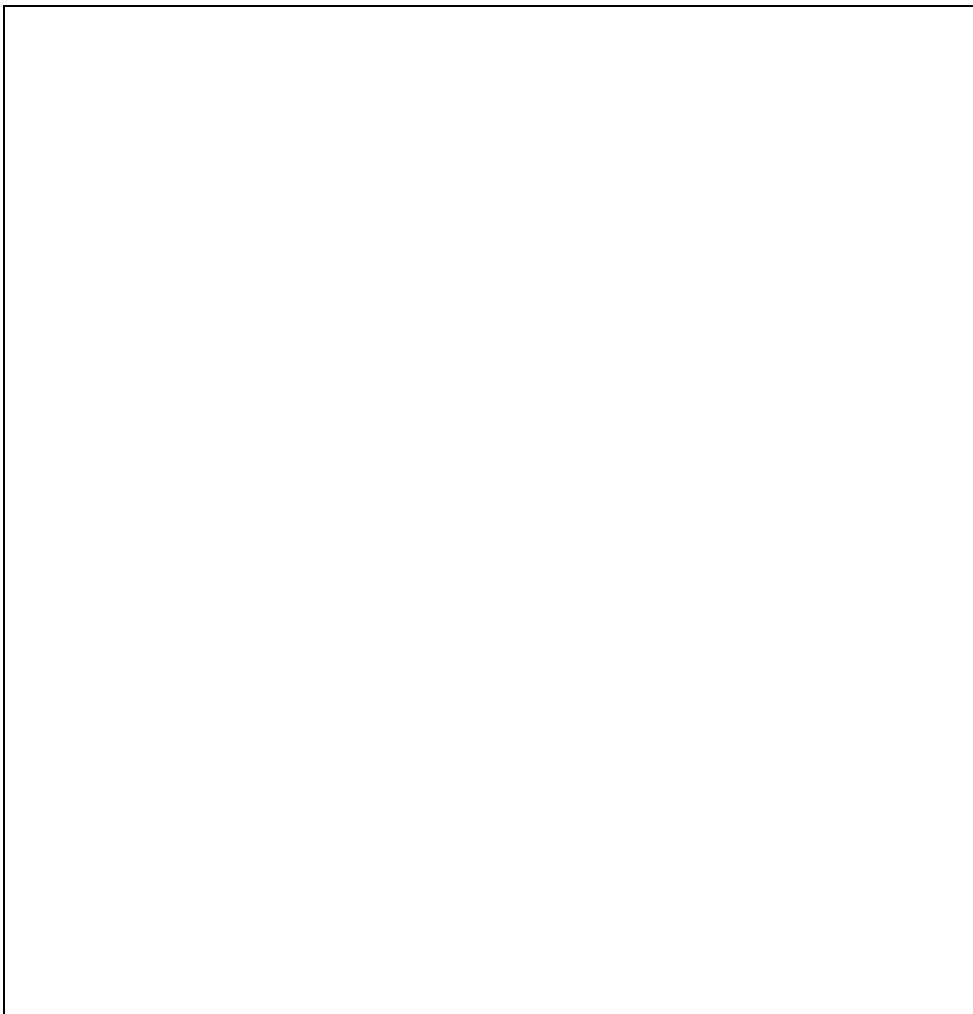
## 6.5 Summary

At the end of these practical, students were able to dehydrate cyclohexanol.

## 6.6 After Laboratory activities

Write the equation for the dehydration of cyclohexanol above.

Draw the structure of a named C-4, C-5 and C-6 alkene



## Practical 7 Qualitative analysis for elements (for reference only)

### Contents

- 7.1 Introduction
- 7.2 Lassaigne's sodium fusion test
- 7.3 Procedure
- 7.4 Group classification tests
  - 7.4.1 Tests for compounds containing nitrogen
  - 7.4.2 Tests for compounds containing C, H and possibly oxygen
- 7.5 Conclusion
- 7.6 Summary
- 7.7 After Laboratory activities

### 7.1 Introduction

In organic compounds the elements commonly occurring along with carbon and hydrogen, are oxygen, nitrogen, sulphur, chlorine, bromine and iodine. The detection of these elements depends upon converting them to water-soluble ionic compounds and the application of specific tests.

Nitrogen, Sulphur, and halogens present in organic compounds are detected by Lassaigne's test. Here, a small piece of Na metal is heated in a fusion tube with the organic compound. The principle is that, in doing so, Na converts all the elements present into ionic form.

- $\text{Na} + \text{C} + \text{N} \rightarrow \text{NaCN}$
- $2\text{Na} + \text{S} \rightarrow \text{Na}_2\text{S}$
- $\text{Na} + \text{X} \rightarrow \text{NaX}$  ( X= Cl, Br, or I)

### 7.2 Lassaigne's sodium fusion test

C, H, O, N, S, X, NaX

NaCN

Na

NaCN<sub>2</sub>S

**In Laboratory activities****Requirement**

Fusion tube (test tube), compound containing any of the above elements, burner, beaker and filter paper

**7.3 Procedure**

<b>Tests</b>	<b>Procedure</b>	<b>Observation</b>	<b>Inference</b>
Preparation	Place 20 mg piece of clean sodium metal, into a fusion tube. Add a little of the compound containing one of the elements above (5 mg or 2 - 3 drops). Heat the tube gently at first, allowing any droplet form to fall back onto the molten sodium. Continue heating until the button of the tube turns dull-red. finally plunge the tube while still hot, into a clean dish containing cold distilled water (6 mL) and cover immediately with clean wire gauze		
<b>Nitrogen</b>	To a portion (2 mL) of the 'fusion' filtrate add 0.2 g of powdered ferrous sulphate crystals. Boil the mixture for a half a minute, cool and acidify by adding dilute sulphuric acid drop wise. Formation of a bluish-green precipitate (Prussian blue) or a blue solution indicates that the original substance contains nitrogen. If no precipitate appears, allow to stand for 15 minutes, filter and inspect filter paper.		

<b>Sulphur (sulphide)</b>	To the cold 'fusion' filtrate (1 mL) add a few drops of cold, freshly prepared, dilute solution of sodium nitroprusside. The latter may be prepared by adding a small crystal of the solid to 2 mL of water. Production of a rich purple colour indicates that the original substance contains sulphur. This test is very sensitive.		
<b>Halogens (halides)</b>	<p>Acidify a portion (1 mL) of the 'fusion' filtrate with 2N nitric acid, and if nitrogen and/or sulphur are present, boil for 1 - 2 minutes. Cool and add aqueous silver nitrate (1 mL), compare with a blank. Formation of a heavy, white or yellow precipitate of silver halide indicates halogen. If a positive result is obtained: acidify the remaining portion of the 'fusion' filtrate with dilute sulphuric acid, boil and cool. Add carbon tetrachloride (1 mL) and a few drops of freshly prepared chlorine water. Shake the mixture.</p> <p>(a) If the carbon tetrachloride layer remains colourless – indicates chlorine.</p> <p>(b) If the carbon tetrachloride layer is brown - indicates bromine.</p> <p>(c) If the carbon tetrachloride layer is violet - indicates iodine. If nitrogen and/or sulphur are also present, the addition of silver nitrate to the acidified 'fusion' solution will precipitate silver cyanide and/or silver sulphide in addition to the silver halides</p>		

## 7.4 Group classification tests

TEST FOR UNSATURATION			
Tests	Procedure	Observation	Inference
Preparation	<ul style="list-style-type: none"> <li>• <b>Alkaline <math>\text{KMnO}_4</math> (1%):</b> Dissolve about 1g of potassium permanganate in 100ml of distilled water. To that solution add 10g of anhydrous sodium carbonate. Shake to dissolve it completely.</li> <li>• <b>Bromine in <math>\text{CCl}_4</math>:</b> Take 5ml of liquid bromine in 100ml of carbon tetrachloride and shake it to dissolve properly.</li> <li>• <b>Bromine Water:</b> Take 5ml of bromine add 100ml of distilled water and shake well. Decant off the clear liquid.</li> </ul>		
Baeyer's test	Add 2 mL of distill water to 0.5 mL of Butene or any unsaturated hydrocarbon. Add few drops of the prepared 1% $\text{KMnO}_4$ dropwise. And record your observation and inference		
Bromine test	Add 1 mL of carbon tetrachloride to 0.5 mL of the test sample in a test tube then add bromine dropwise to the solution of $\text{CCl}_4$ and the test sample. Addition of the bromine should be continued until the brown colour reappeared and persist when shaken. Dip a rod in to ammonium hydroxide and stir the solution. Absence of white fumes confirm unsaturation		

<b>GROUP CLASSIFICATION TEST</b>			
<b>Tests for compounds containing nitrogen</b>			
Amine	<p>Add 1 mL of aniline to 4 mL 80% concentrated Nitric acid in a test tube and cool the mixture in an ice bath for 0-4°C. To the cooled solution, add sodium nitrite solution dropwise and mix well. Test for excess nitrous acid by dipping wet blue litmus paper. Add excess of nitrous acid if the litmus did not turn red.</p> <p>Note:</p> <ul style="list-style-type: none"> <li>• Primary aliphatic amines react with nitrous acid to give nitrogen gas, which is seen as bubbles.</li> <li>• Secondary amines react with nitrous acid to form a yellow oily nitrosamine.</li> <li>• Tertiary amines react with nitrous acid to form soluble nitrite salts.</li> </ul>		
<b>Tests for compounds containing C, H and possibly oxygen</b>			
Carboxylic acids	To 1 mL each of ethanoic, propanol, propanoic, ethanol and acetic acids, add a pinch of sodium bicarbonate and note the effervescence observed.		
Phenols	Prepare 10 % of gallic acid in a beaker. To 2 Ml of distill water, add few drops of the gallic acid solution the shake to dissolved thoroughly. Add few drops of 1% FeCl <sub>3</sub> to the solution above. Do same to solution containing acetyl salicylic acid. Note the colour change in the two solution and report		
Aldehyde and ketone	Place 5 Ml of 2,4dinitrophenylhydrazine in three separate test tubes and label as 1,2,3. To the test tube 1, add 10 drops of acetone, test tube 2 add 10 drops of glyceraldehyde and to the test tube 3 add 10 drops of ethanol and tap the test tubes with the tip of your finger and observe any formation of precipitate. If no precipitation, gently heat in water bath at 60°C for 4-5 minutes and cool in a beaker containing ice until crystals are formed. Collect the crystals by vacuum distillation using Hirsch		

	funnel and to dry on the funnel. Measure the melting range of the crystal using melting point apparatus.		
Lucas test for primary, secondary and tertiary alcohols	<p><b>Preparation:</b> Preparation of Lucas Reagent – Take equimolar quantities of zinc chloride and concentrated HCl and make a solution. Take 0.5 mL of ethanol, 2-pentanol and 2-methyl butanol in three different test tubes. To each of the test tubes add 2 mL of the Lucas reagent previously prepared and observed the rate at which the solution turns turbid.</p> <p>The solution turns turbid and forms an oily layer immediately then the sample is a tertiary alcohol. The solution turns turbid and forms an oily layer in three to five minutes then the sample is a secondary alcohol. The solution remains colourless unless it is subjected to heat. The solution forms an oily layer when heated, then it is a primary alcohol</p>		

## 7.5 Conclusion

The qualitative analysis is a general name for the methods used in the determination of the identity rather than the amount of chemical species (quantitative analysis). The qualitative process usually utilises the reaction(s) characteristic for the given chemical species and interprets the obtained results using a deductive thought process

## 7.6 Summary

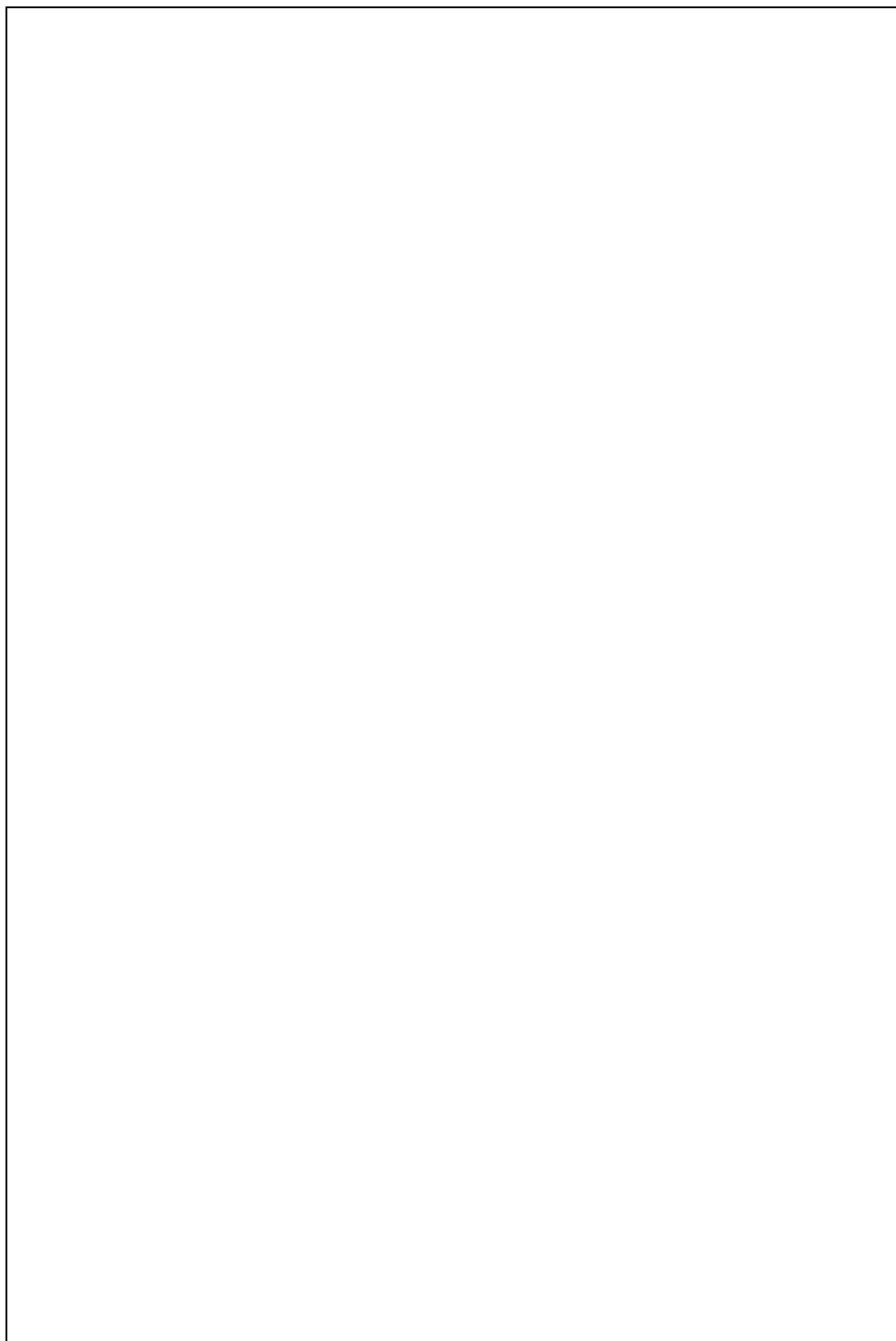
In this practical session, we have:

- Identified some functional groups and give examples of members of functional group families.
- learnt how to predict the results of solubility tests of known compounds, and to use solubility test data to classify unknown compounds.



**7.7 After Laboratory activities**

1. Give the structure of ethene
2. Write the equation for bromination of a named unsaturated hydrocarbon
3. What are amines?



## SECTION II      PHYSICAL CHEMISTRY

### Contents

- Practical 1    pH Measurements
- Practical 2    Determination of Relative Molar Mass from Colligative Properties
- Practical 3    Temperature Measurements and Heat of Dissolution
- Practical 4    Heat of Neutralization
- Practical 5    Demonstration of Partition Coefficient in two Immiscible Solutions
- Practical 6    Ideal Gas Law: Measuring the Molar Volume of a Gas and the Universal Gas Constant

### Practical 1    Ph Measurements

#### Contents

- 1.1    Introduction
- 1.2    Materials
- 1.3    Conclusion
- 1.4    Summary
- 1.5    Tutor-marked assignment
- 1.6    After Laboratory activities

#### Before Laboratory activities

##### 1.1    Introduction

For most science experiments, you will need a pH indicator, such as wide-range litmus, pH paper, or pH meter. These pH indicators contain a chemical that changes colour when it comes in contact with acids or bases. For example, litmus and pH paper turn red in strong acids and blue in strong bases. Because only a few pH indicators measure pH over a wide range of pH values, you will need to find out the pH range of the indicator you use. Typically, the colour chart provided with each pH indicator kit will show the pH range of that indicator. Colour pH indicators provide only an approximate measure of the pH, or the strength of the acid or base. They are not as accurate as the expensive instruments' scientists use to measure pH, but they are adequate for the experiments you need to do at this level.

When measuring pH with pH paper, dip the end of a strip of pH paper into each mixture you want to test. After about two seconds, remove the paper, and immediately compare the colour at the wet end of the paper with the colour chart provided with that pH indicator. When measuring pH with pH meter, dip the end of electrode of pH meter into each mixture you want to test. Write the pH value and colour. Always use a clean, unused strip of pH paper for each mixture that you test.

**Table II: List of Indicators and their pH Range**

Indicators	Acid (colour change)	Base (colourchange)	pH range
Methyl orange	Red	Yellow	3.1-4.4
Methyl Red	Red	Yellow	4.2-6.3
BromothymolBlue	Yellow	Blue	6.0-7.8
Phenol Red	Yellow	Red	6.4-8.0
Phenolphthalein	Colourless	Pink	8.0-9.8
Thymol Blue	Red	Yellow	1.2-2.8
Alizarin Yellow	Yellow	Red	10.1-12.0

## In Laboratory activities

### 1.2 Materials

pH paper and colour chart (pH range 3 to 12) or pH meter, distilled water, white vinegar, household ammonia (or baking soda) spot plate test or 3 small test tubes, stirring rod solutions/fruits juice (lemon, lime, orange, or melon), beverages (cola, carbonated non-cola, milk).

### 1.3 Procedure

1. Dip an unused strip of pH paper into solution. Leave until wet (about 2 seconds). Immediately compare with the color chart. Write down the approximate pH value of the solution. If you're using pH meter write down the approximate pH value of the solution.
2. Repeat the same process for the other solutions and record your observation in a table.

Measurement of pH									
	H <sub>2</sub> O	White vinegar	Household ammonia	Lemon	Lime	Melon	Lipton tea	Fanta	Liquid Milk
Red Litmus									
Blue Litmus									
pH Meter									

#### 1.4 Conclusion

A solution with a low pH value is called an "acid," while one with a high pH is called a "caustic." The common pH scale extends from 0 (strong acid) to 14 (strong caustic), with 7 in the middle representing pure water (neutral).

#### 1.5 Summary

In this unit, you have been exposed to the use of litmus paper to measure pH as well as determining the pH of common substances using litmus paper.

#### 1.6 After Lab activities

1. What is the pH and pOH of a solution that contains  $3.50 \times 10^{-5}$  M hydronium ions?
2. Give reason why the litmus paper must be wet when measuring pH of a substance

1.

2.

## **Practical 2            Determination of Relative Molar Mass from Colligative Properties**

### **Contents**

- 2.1 Introduction
- 2.2 Procedure
  - 2.2.1 Determining the freezing point of pure paradichlorobenzene
  - 2.2.2 Determination of the freezing point depression constant
  - 2.2.3 Determination of the molar mass of an unknown solute
- 2.3 Conclusion
- 2.4 Summary
- 2.5 After Laboratory activities

### **Before Laboratory activities**

#### **2.1 Introduction**

The vapour pressure of a pure liquid at a given temperature is a characteristic property of that liquid. However, when a nonvolatile solute is dissolved in the liquid, the vapour pressure of the liquid is reduced. This lowering of the vapour pressure causes a change in the melting point, boiling point, and osmotic pressure of the liquid. The magnitude of the change in these properties depends upon the number of solute particles dissolved in a given amount of the solvent, but not upon the nature of the particles (their identity). Such properties are called colligative properties. The addition of ethylene glycol to the water in a car radiator in order to raise its boiling point, or the use of salt to lower the melting point of ice on a sidewalk is some everyday applications of colligative properties.

### **In Laboratory activities**

#### **2.2 Procedure**

##### **2.2.1 Determining the freezing point of pure para-dichlorobenzene.**

Set up a hot water bath using a tripod, burner, and an 800 or 1000 mL beaker. Obtain the mass of an empty large test tube. Fill the test tube approximately one-fourth to one-third full of solid Para=dichlorobenzene and again determine the mass of the tube. Subtract the two masses to obtain the mass of Para-dichlorobenzene. Clamp the test tube in place in the hot water bath. Once all the Para-dichlorobenzene has melted, remove the tube from the water bath and insert a rubber stopper containing a thermometer and wire stirrer.

Stirring the sample constantly, record the temperature of the sample every 30 seconds. Graph your results and record the melting point of pure Para-dichlorobenzene.

<b>Determining the freezing point of pure para-dichlorobenzene</b>			
Mass of test tube (M <sub>1</sub> )	Mass of test tube + P-dichlorobenzene (M <sub>2</sub> )	Mass of P-dichlorobenzene (M <sub>2</sub> -M <sub>1</sub> )	Temperature of P-dichlorobenzene At varying time T
			30 sec. _____
			60 sec _____
			90 sec _____
			120sec _____
			150sec _____
			180sec _____

### 2.2.2 Determination of the freezing point depression constant.

Weight out approximately 0.50 grams of naphthalene, C<sub>10</sub>H<sub>8</sub>, on a small piece of weighing paper. Return the test tube to the hot water bath and once the sample has completely melted, add the naphthalene. Remove the test tube from the hot water bath and repeat the above procedure. Calculate the freezing point depression of the solution, the molality of the solution, and from this data the freezing point depression constant

$$\Delta T_f = T_f(\text{solvent}) - T_f(\text{solution}) = K_f \times m$$

where  $\Delta T_f$  is the freezing point depression,  $T_f(\text{solution})$  is the freezing point of the solution,  $T_f(\text{solvent})$  is the freezing point of the solvent,  $K_f$  is the freezing point depression constant, and  $m$  is the molality.

for paradichlorobenzene. Return the test tube to the hot water bath and heat until the sample has melted. Pour the melted solution into the "PDB WASTE" container. If any solid remains in the test tube, wash it with a small portion of acetone. Any acetone waste should also go into the waste container. You may wish to repeat this procedure a second time.

### 2.2.3 Determination of the molar mass of an unknown solute.

Refill the test tube with fresh paradichlorobenzene and determine the mass of the test tube and contents. Weight out approximately 0.50 grams of unknown solute on a small piece of weighing paper. Add this to the test tube. Heat the test tube in the hot water bath until the mixture is completely melted. Remove the test tube from the water bath, insert the stopper containing the thermometer and wire stirrer, and record the temperature of the solution as in the previous trials. Determine the freezing point of the solution, the freezing point depression, and from this data calculate the molar mass of the unknown solute. Reheat the test tube and pour the molten solution into the "PDB WASTE" container. If the tube does not come completely clean, wash with a small portion of acetone. You may wish to repeat this procedure a second time.

From your result:

1. Make three graphs - a cooling curve pure paradichlorobenzene versus time, a cooling curve of for the paradichlorobenzene naphthalene mixture versus time, and a cooling curve of the paradichlorobenzene - unknown solute mixture versus time.
2. Calculate the freezing point depression constant for paradichlorobenzene.
3. Calculate the molar mass of the unknown solute.

NOTE: Please refer to your CHM 292 for any formula

## 2.3 Conclusion

Most substances can exist as solid, liquid, or gas, depending upon the temperature and pressure. Whether a particular substance exists as a solid, liquid, or gas under conditions of standard temperature and pressure is dependent upon the nature of the substance and includes such properties as the molecular weight of the substance or the intermolecular forces of attraction between molecules.

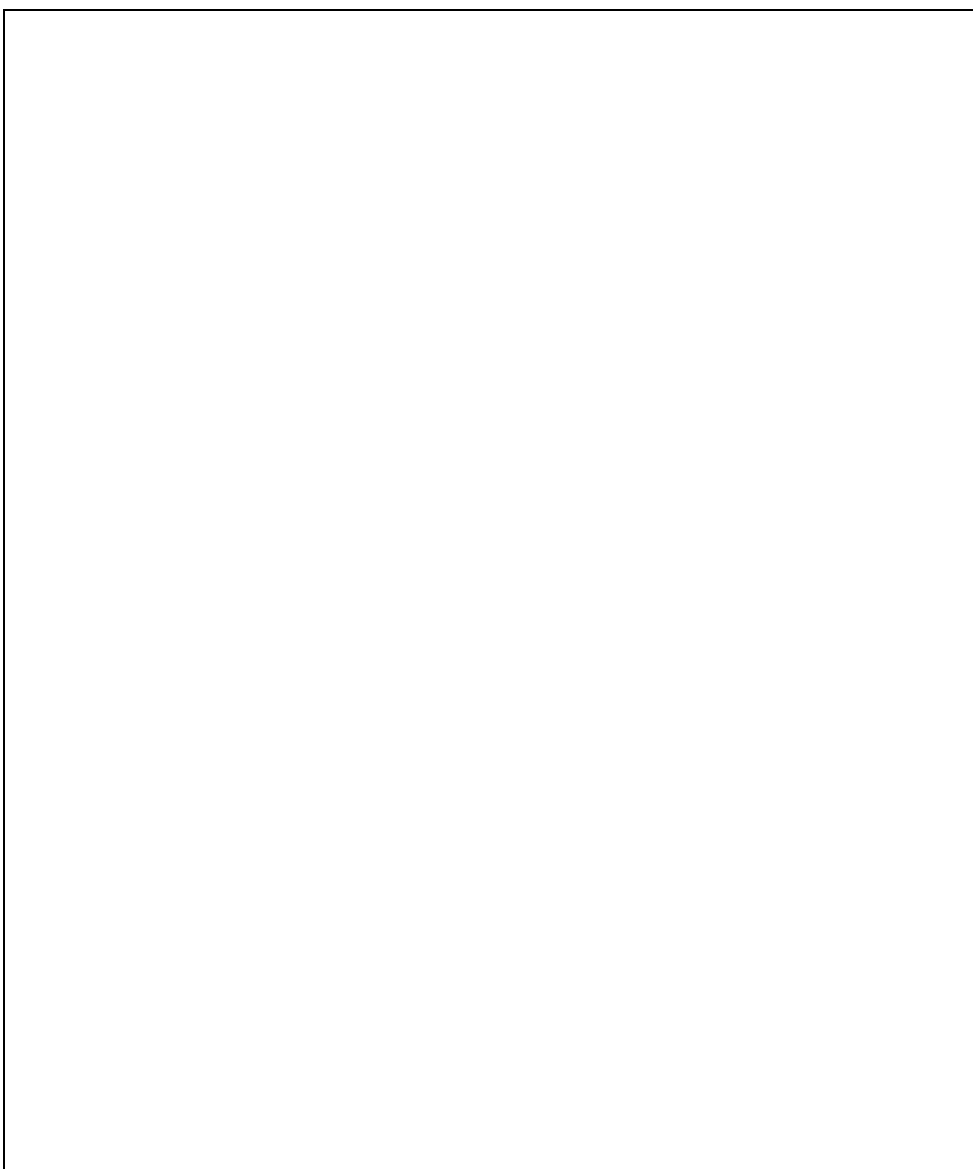
## 2.4 Summary

In this unit, you learnt how to investigate the phenomenon of freezing-point depression as a colligative property and also determine the molar mass of an unknown solute using freezing-point depression.

## 2.5 After Laboratory activities

What are colligative properties?

2.00 g of some unknown compound reduces the freezing point of 75.00 g of benzene from 5.53 to 4.90<sup>0</sup>C. What is the molar mass of the compound?





## Practical 3 Temperature Measurements and Heat of Dissolution

### Before Laboratory activities

- 3.1 Introduction
- 3.2 Material
- 3.3 Procedure
  - 3.3.1 Temperature measurement
  - 3.3.2 Heat of dissolution
- 3.4 Report Sheet
- 3.5 Conclusion
- 3.6 Summary
- 3.7 After Laboratory activities

### 3.1 Introduction

Temperature is a measure of how hot or cold an object is. Whenever there is a temperature difference, there will be a spontaneous heat flow from the object at higher temperature to the object at lower temperature. Thermometer is the instrument used to determine temperatures. Celsius and Kelvin scales is used for metric and SI units respectively while Fahrenheit scale is the choice for British units. These three scales are related by the following relationships:

$$K = ^\circ\text{C} + 273 \quad ^\circ\text{F} = 9/5 (^\circ\text{C}) + 32 \quad ^\circ\text{C} = 5/9 (^\circ\text{F} - 32)$$

Every change, physical or chemical, is associated with a change in energy, usually in the form of heat. The energy change of a reaction that occurs under a constant pressure is defined as the heat of the reaction or the enthalpy change. If heat is evolved during the change, the process is exothermic, and if heat is absorbed during the change, the process is considered to be endothermic. By convention, enthalpy change for an exothermic process has a negative value while that of an endothermic process has a positive value. We are familiar with different forms of energy. Heat energy, light energy, electrical energy, nuclear energy, chemical energy of the bonds in a molecule, are just a few examples of different forms of energy. From the law of conservation of energy, during any physical or chemical change:

Energy lost = Energy gained.

In this experiment, you will become familiar with temperature measurements and record the temperature changes that occur when ammonium chloride and calcium chloride are dissolved in water. From this data, you will be able to calculate the heat energy given off or absorbed during this dissolution process (heat of dissolution).

Heat absorbed/ evolved = (mass) (specific heat) (temperature change)  
The SI unit for heat is joule (J) while a non-SI unit calorie (cal) is widely used in scientific measurements. The relationship between these two units is:

$$1 \text{ cal} = 4.184 \text{ J}$$

Specific Heat is the amount of heat required to raise the temperature of one gram of a substance by one degree Celsius. It can be expressed in cal / g.<sup>0</sup>C or Joules / kg Kelvin.

Water has a relatively high specific heat of 1 cal /g.<sup>0</sup>C while metals usually have low specific heat. To calculate the heat of dissolution in water, specific heat of the aqueous solution will be considered to be that of pure water, 1 cal / g.<sup>0</sup>C.

Calorimeter is an instrument used to measure heat flow in and out of a system. In this experiment, the calorimeter will consist of two Styrofoam cups, one nesting in the other.

### 3.2 Materials

Beaker (100 mL), Thermometer, Hot Plate, Graduated Cylinder (50 mL), Ring Stand, Thermometer Clamp, Stirring Rod, Balance, Spatula, Styrofoam Cups (2), Cardboard Square, Sodium Chloride (NaCl), Calcium Chloride (CaCl<sub>2</sub>), Ammonium Chloride (NH<sub>4</sub>Cl), Ice.

### In Laboratory activities

### 3.3 Procedure

#### 3.3.1 Temperature measurement

1. Using thermometer, measure the temperature of 50 mL of water in a 100 mL beaker. Be sure that the bulb is steady during the measurement and not touching the glassware. The bulb needs to be fully immersed in the liquid.
2. Place a 100 mL beaker with 50 mL water on a hot plate. Place a thermometer in the water with the help of a stand and clamp. Bring the water to a boil indicated by steady stream of bubble formation from within the liquid. Once water starts to boil temperature is going to be steady until all of the water boils off. Measure the boiling point of water.
3. Make about 30 mL of an ice-water mixture in a 100 mL beaker. Stir the ice slush and measure the temperature.

4. Add three tea spoons full of table salt, sodium chloride, to the slush and stir. Measure the temperature of the mixture.

<b>Temperature measurement</b>			
<b>Temperature of 50 mL water before heating</b>	<b>Temperature of 50 mL after heating</b>	<b>Temperature of ice</b>	<b>Temperature of mixture of ice + salt</b>

### 3.3.2 Heat of dissolution

1. Work in pairs for this section.
2. Weigh out about 10 grams of  $\text{CaCl}_2$ . Be sure to record the exact mass. Construct a calorimeter by nesting two Styrofoam cups, one inside the other. Add 50 mL of water to the calorimeter.

Allow the water to stand for five minutes to reach a stable temperature. Place a small piece of card board to cover the cup. Make a small hole at the center of the card board and insert the thermometer through the hole. Make sure the thermometer bulb is under water. Measure the temperature of water.

This is the initial temperature ( $T_i$ ).

3. Holding the calorimeter steady, add all of the  $\text{CaCl}_2$  to water, place the cover, and stir rapidly with a thermometer. Be careful with the bulb of the thermometer while stirring.
4. After mixing, time - temperature data should be recorded. One partner should record the temperature while both read the time and keep the record.
5. For five minutes, right from the start of mixing, take temperature at intervals of every 30 seconds. The highest temperature reached is the final temperature ( $T_f$ ) of water.
6. Print the temperature versus time plot using the graph paper provided in the lab book.
7. After recording your data, wash contents of the cup down the sink with lots of water.
8. Repeat steps 1 - 7 using approximately 10 grams of ammonium chloride. The minimum temperature reached in this case is the final temperature ( $T_f$ ).

### 3.4 Report Sheet

Mass of water	Mass of CaCl <sub>2</sub>	Mass of water + CaCl <sub>2</sub>	Temperature at time (T <sub>i</sub> )	Temperature at time (T intervals) up to 5 minus	Temperature of Ammonia at time (T) intervals

Calculate the heat of dissolution per gram of CaCl<sub>2</sub> in cal/g (show calculation)

- Indicate if the dissolution of CaCl<sub>2</sub> is exothermic or endothermic?
- Calculate the heat lost by water during the dissolution of NH<sub>4</sub>Cl:
- Calculate the heat of dissolution per gram of NH<sub>4</sub>Cl in cal/g.
- Is dissolution of NH<sub>4</sub>Cl an exothermic or endothermic reaction?

### 3.5 Conclusion

Temperature and heat of dissolution are quantities that can be measured in the laboratory.

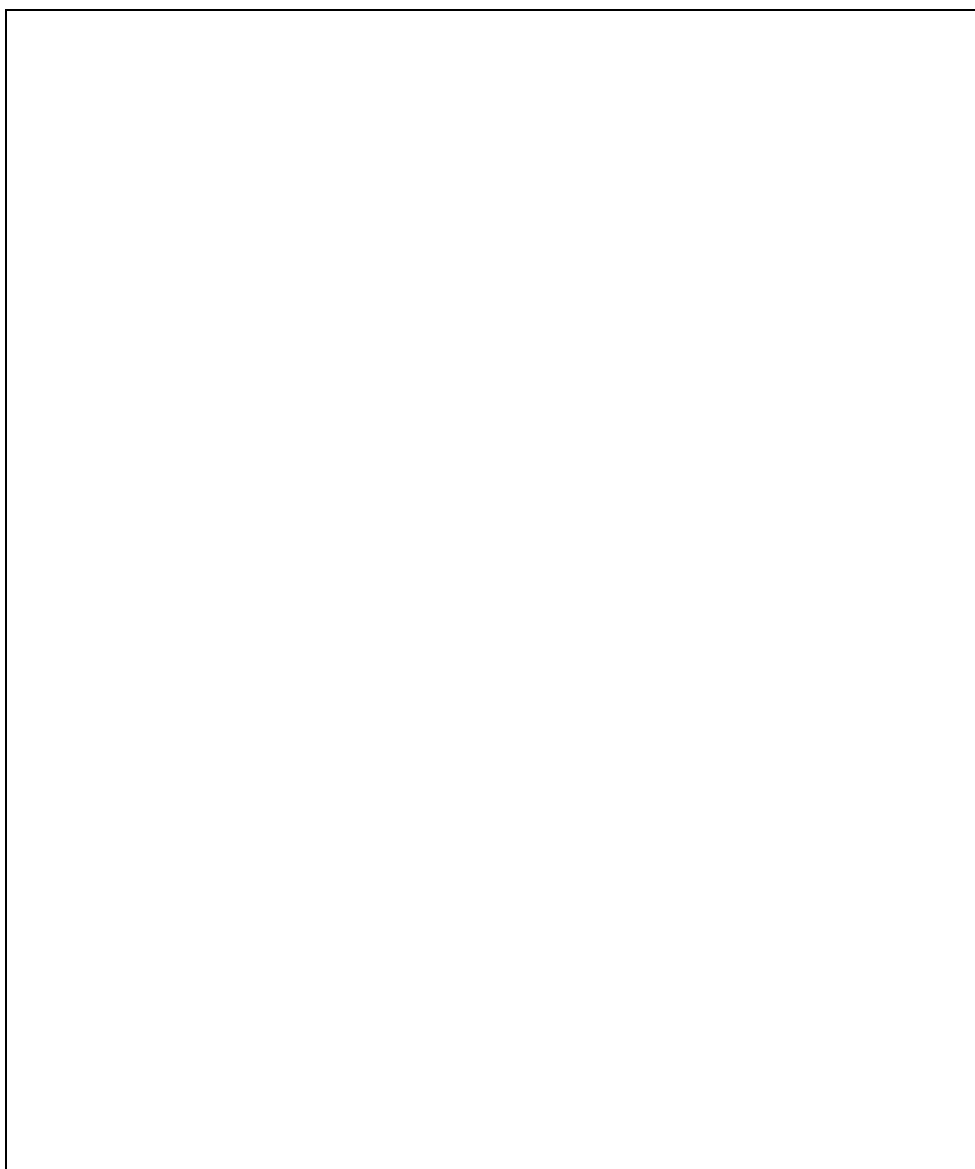
### 3.6 Summary

In this session, you have been able to:

- demonstrate the enthalpy/heat flow as different solutes are dissolved in aqueous solution
- determine the heat of solution.

### 3.7 After Laboratory activities

1. Aluminum metal melts at  $660.37^{\circ}\text{C}$ . What is the temperature in Kelvin?
2. Define heat of solution.



## Practical 4 Heat of Neutralization

### Contents

- 4.1 Introduction
- 4.2 Equipment
- 4.3 Procedure

### Before Laboratory activities

#### 4.1 Introduction

Energy changes always accompany chemical reactions. If energy, in the form of heat, is liberated the reaction is exothermic and if energy is absorbed the reaction is endothermic. Thermochemistry is concerned with the measurement of the amount of heat evolved or absorbed. The heat (or enthalpy) of neutralisation ( $\Delta H$ ) is the heat evolved when an acid and a base react to form a salt plus water. We had started a discussion in the last unit on heat. You performed an experiment on heat of dissolution. You also studied molar enthalpy (heat flow) as different solutes were dissolved in different solutions. In this unit we shall focus on another type of heat flow known as heat of neutralisation.

#### 4.2 Equipment

To determine the heat of neutralization of hydrochloric acid and sodium hydroxide you shall need the following equipment:

- Calorimeter
- Beckmann thermometer
- Graduated cylinder
- Beaker (250 ml)
- Distilled water
- Burette
- Pipette
- Conical flask
- Funnel
- Spatula
- Phenolphthalein -pH indicator.

## In Laboratory activities

### 4.3 Procedure

1. Prepare 500 ml of 5N HCl and 500 ml of NaOH, thermostat the two solutions at room temperature T1.
2. Titrate 50 ml NaOH (in a flask) against 5N HCl by using phenolphthalein as pH indicator, determine V1 (HCl). Weigh the empty calorimeter W1.
4. Place 50 ml of 5N NaOH in the calorimeter and record the temperature at steady state T2.
5. Introduce quickly V1 (HCl) and justly record the temperature T3.
6. Weigh the calorimeter with its contents W2.
7. The weight of NaCl solution,  $W3 = W2 - W1$ .

**Table III: Data recording**

Cc	CNaCl	V1	W1	W2	W3	T1	T2	T3

#### Calculations:

1. Calculate  $\Delta T$ ;  $T3 - \frac{T1+T2}{2}$ ; T1 may be = T2.
2. Calculate the heat absorbed by 5N NaCl solution where,  
 $\Delta H1 = C_{NaCl} \times W3 \times \Delta T$

Consider ( $C_{NaCl} = 3.895 \text{ cal}$ )

3. The heat absorbed by the calorimeter;  
 $\Delta HZ = Cc \times \Delta T$

**Note:** The heat capacity of the calorimeter is given as:

$$Cc = Q / \Delta T$$

Where Q the amount of heat absorbed by the calorimeter: the amount of heat lost by water:

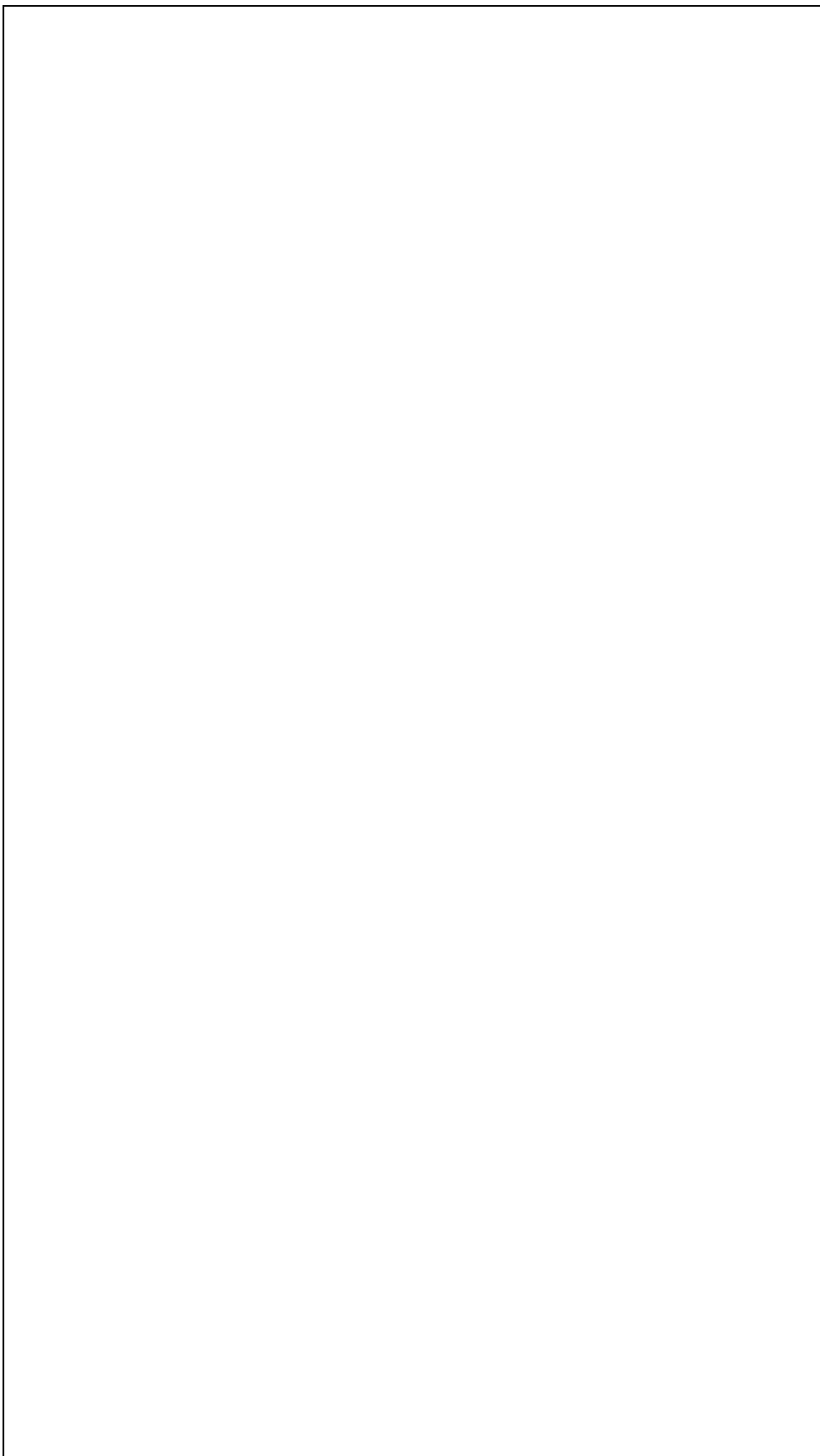
4. Calculate heat of neutralisation where:

$$\Delta H = \Delta H1 + \Delta H2$$

5. Calculate the number of water produced from neutralization reaction (undissociated water) where:  $n = (5 \times 36.5 \times V1) / 1000$



6. Calculate the molar heat of neutralisation;  
 $\Delta H^\circ - \Delta H/n$



## Practical 5      **Ideal Gas Law: Measuring the Molar Volume of a Gas and the Universal Gas Constant**

### Contents

- 5.1 Introduction
- 5.2 Materials/Apparatus
  - 5.2.1 Reagents and materials
- 5.3 Procedure
  - 5.3.1 Data and calculations
- 5.4 Conclusion
- 5.5 Summary
- 5.6 Tutor-marked assignment

### Before Laboratory activities

#### 5.1 Introduction

In this experiment, you will determine the volume that is occupied by one mole of gas. You will use the reaction of magnesium with hydrochloric acid to generate hydrogen according to the equation:



Knowing the volume (V) of one mole ( $n = 1$ ) at a temperature (T) and pressure (P) allows the calculation of the universal gas constant (R) in the ideal gas equation of state, commonly known as the ideal gas law:  
 $PV = nRT$

The value of R will differ depending on the units used for pressure and volume. When P is in atmospheres and V is in liters, the value of R is 0.08206 (L atm)/(mol K).

This equation is useful because it allows one to calculate the pressure, volume, temperature or number of moles of a gas simply by knowing the other three variables and doing a little algebra.

#### 5.2 Materials/Apparatus

- Vernier caliper
- 400 mL beaker
- 10.0 mL graduated cylinder
- 1-hole stopper to fit the cylinder
- 10 - 15 cm of 22-gauge copper wire
- Wash-bottle (with water)

### 5.2.1 Reagents and materials

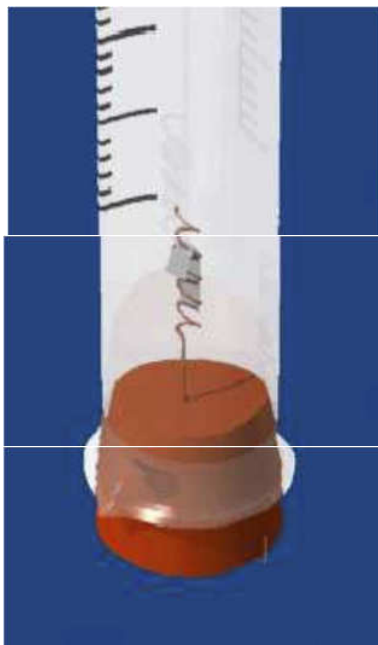
- Magnesium ribbon (0.9 - 1.0 cm) cut carefully so that the ends are "square"
- 3 M hydrochloric acid
- Sodium hydrogen carbonate (to neutralize the hydrochloric acid)

### In Laboratory activities

### 5.3 Procedure

You will be working with small quantities of quite concentrated hydrochloric acid. **Be careful** not to get any on your skin, eyes or clothing. Wipe up small spills immediately - consult your demonstrator in the case of a serious spill. It is strongly recommended you wear latex gloves, particularly at steps (6) and (8). Repeat the experiment three times.

1. Almost fill a 400 mL beaker with tap water. The water should be close to room temperature, if possible, by the time you begin the reactions.
2. Obtain a short (0.9 - 1.0 cm) length of magnesium ribbon. Measure the length of the ribbon as precisely as possible with a ruler. (Ideally this should be done with a vernier caliper.) *The ends should be cut as squarely as possible - the length measurement will be used to calculate the mass of the magnesium and needs to be as accurate as possible.*
3. Wind one end of the copper wire around the magnesium ribbon and then bend them together so that the ribbon cannot slip out. (Hydrochloric acid does not react with copper, so copper wire can be used to hold the magnesium in place). Pass the free end of the wire through the rubber stopper so that the magnesium is positioned 2 to 3 cm from the narrow end of the stopper and bend it over the wide end to hold it loosely in place (Figure 5).



4. Wash the 10 mL cylinder with detergent so that it is not greasy and drains clearly. Then pour into it about 3 mL of 3 M hydrochloric acid.
5. Using a plastic wash-bottle, very carefully add water slowly down the side of the cylinder so that the denser acid in the bottom does not mix significantly with the water as you add it - you are trying to float the water on top! Fill the cylinder to the brim. Carefully insert the stopper with the magnesium ribbon. Some water should come through the hole in the stopper. Make sure that the hole is full of water when the stopper is firmly in place. [If you see any sign that the ribbon is reacting at this point, the acid was stirred up too much as you added the water – you should get to the next step as quickly as possible!]
6. Use your index finger to close the hole in the stopper, invert the cylinder and lower it into the beaker of water. As soon as the stopper is below the water surface you can remove your finger. Clamp the inverted cylinder in a vertical position. The denser hydrochloric acid should mix with the water in the cylinder and begin to react with the magnesium. You should see bubbles of hydrogen rising to fill the base end of the cylinder. Wait for all the magnesium to dissolve and then wait 2 or 3 minutes more.

Tap the sides of the cylinder to dislodge bubbles from time to time.

7. Adjust the height of the cylinder so that the water levels, and the pressures, inside and outside are the same (i.e. atmospheric pressure). Your demonstrator will tell you what this pressure is; it will have been measured on a barometer elsewhere in the

department. Record the volume of hydrogen evolved by reading the water level on the scale on the cylinder as accurately as possible ( $\pm 0.02$  mL).



8. Clean-up: Remove the cylinder and turn it the right way up. Pour the water out of the beaker into the sink and then empty the cylinder into the beaker. Add sodium hydrogen carbonate to the acidic solution until the evolution of carbon dioxide stops. Then flush the mixture down the sink with plenty of water. Rinse the pieces of apparatus and repeat the experiment twice more.

### 5.3.1 Data and calculations

Complete the data table below. The following notes refer to the entries in the Data column:

- i. Your demonstrator will probably give you the value of atmospheric pressure read from the barometer in the lab in mm Hg (torr). Convert it to atmospheres using  $1 \text{ atm} = 760 \text{ mm Hg}$ .
- ii. Refer to the small table below to determine the vapour pressure of water at the temperature you are reporting. Convert it to atmospheres.

#### Vapour pressure of water at various temperatures

Temperature ( $^{\circ}\text{C}$ )	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Pressure (mm Hg)	12.8	13.6	14.5	15.5	16.5	17.5	18.6	19.8	21.1	22.4	23.8	25.2	26.7	28.3	30.	31.8.

iii. The actual hydrogen pressure will be the current atmospheric pressure (i) minus the vapour pressure of water from (ii).

Data	Run 1	Run 2	Run 3
Water temperature (oC)			
Water temperature (K)			
Atmospheric pressure (atm)			
Vapour pressure of water (atm)			
Corrected pressure of hydrogen (atm)			
Volume of gas collected (L)			
Length of magnesium ribbon used (mm)			
Mass of magnesium used (g)			
Moles of magnesium used			
Theoretical moles of hydrogen produced			
Volume hydrogen/moles hydrogen (L/mol)			
Gas constant R. Average value of the gas constant R (with units!)			

## 5.4 Conclusion

Using the ideal gas law/equation, the volume of a gas can be determined in the laboratory.

## 5.5 Summary

In this unit, we were able to demonstrate how to determine the volume of a given mole of a gas.

### After Laboratory activities

Calculate the volume of exactly 1 mole of an ideal gas at STP (Standard Temperature and Pressure, 0°C and 1 atm). Note:  $P = 1 \text{ atm}$   $n = 1 \text{ mol}$   $V = ?$   $T = 0 \text{ }^\circ\text{C} = 273.15 \text{ K}$   
 $R = 0.08206 \text{ (L atm)/(mol K)}$ .

2 A 9.0 L volume of chlorine gas is heated from 300 K to 400 K at constant pressure. What is the final volume?

**Reference**

**CHM 292 Course Material, National Open University Of Nigeria**