

# Semester 1 LABORATORY MANUAL

# **Edited By: CHIENG TIONG CHIN** SHARIFAH MONA ABDUL AZIZ ABDULLAH WAN ROSLINA WAN YUSOF **MOHD ALHAFIIZH ZAILANI**



Pusat Khidmat Makimat Akademik UNIVERSITI MALAYSIA SARAWAY

P.KHIDMAT MAKLUMAT AKADEMIK



# PRK1016 Chemistry I Laboratory Manual Semester 1

With Compliments from Banggan Peneulitan

PUSHEKALINALAYSIA SAKAWAN

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# PRK1016 Chemistry I Laboratory Manual

Semester 1

Edited by: Chieng Tiong Chin Sharifah Mona Abdul Aziz Abdullah Mardhiah Mohd Shahabuddin Wan Roslina Wan Yusof Mohd Alhafiizh Zailani Dhana Jay Raja Gopal Abdul Al-Hafiz Ismail Dr. Melody Kimi Tee Kim Tian

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#### Disclaimer

The experiments in this laboratory manual are designed to be used by the students of Centre for Pre-University Studies, Universiti Malaysia Sarawak and should not be conducted unless there is an appropriate level of supervision, safety training, personal protective equipment and other safety facilities available for users. Any user of these procedures assumes all responsibility for the safe handling of hazardous chemicals and procedures. The Curators of Universiti Malaysia Sarawak, its officers, employees and agents shall not be liable for any damages in connection with, or arising out of, the performance, or use of these procedures.

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# Preface

This laboratory manual is especially designed for Pre-University students in Universiti Malaysia Sarawak, who are intended to further their education in science programs. The scopes cover basic practical techniques in analytical, physical, inorganic and organic chemistry.

A concerted effort has been made to use environmentally less toxic or non-toxic materials in these experiments. This was not only done to protect students but also to lessen the impact on the environment. Some experiments are completely environmentally safe and in these the products can be disposed of by placing solids in the waste basket and solutions down the sink. Others contain a very limited amount of hazardous waste, which in these cases it must be collected in the proper container for disposal.

The objectives of this laboratory manual are to provide hands-on experience in chemistry experimental techniques, and to reason and analyse the outcomes of the experiments. The sequence of experiments in this Laboratory Manual is designed to follow the lecture curriculum. Hence, these would help the students to have a better understanding on what they have learnt during lectures.

We view this manual as one of continual modification and improvement. We encourage comments and ideas for improvement of this manual or suggestions for new experiments. Finally, we hope that this laboratory manual is helpful in the study of chemistry at pre-university level.

Chemistry Centre for Pre-University Studies UNIMAS

Prisat Khidmat Alakhmat Akadamik

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#### TABLE OF RESULTS

# **Note for Students**

All students are required to attend practical classes of 3 hours each. Students are required to complete seven (7) experiments in one semester. If a student is unable to attend any practical classes, he/she should produce a medical certificate or a letter of exemption.

Students are required to read the laboratory manual before the practical class. The students need to have enough knowledge about the experiment so that they will know

what to do while in the laboratory and what to expect from the experiment. This can be tested by a pre-lab preparation which is a pre-requisite to enter the laboratory.

Safety in the laboratory is the first priority. All students must check the condition of all the apparatus needed before starting the experiments. The students should report any shortage or breakage of apparatus to the instructor, demonstrator or lab assistant immediately. Check all glassware for cracks before usage. Cracks could cause the glassware to fail during use, causing severe injuries.

Raw data obtained during the experiment must be submitted to the instructor demonstrator at the end of each practical class. A handwritten group laboratory report for each experiment must be submitted either within five (5) working days after completion of the experiment or within the duration of time set by the instructor for assessment. Credit is given for attempting an experiment even if the students do not obtain good results. Students will be assessed based on their attendance, involvement in practical work and the laboratory reports submitted.

# Laboratory Safety

Safety in the laboratory is a subject of the utmost importance. All chemicals are harmful to some degree, therefore, it is imperative to learn the safety rules and follow them strictly at all times. You will be expelled from the laboratory for failing to comply with these regulations. These rules are referred to many laboratories as "the usual safety procedures".

#### General

- 1. Wear appropriate shoes at **all times** when you are in the laboratory.
- 2. Wear lab coat at **all times** when you are in the laboratory.
- Keep long hair tied back and ensure that long or large necklaces are safely tucked 3. away.
- 4. Report any spill, accident and dangerous incidents immediately to your instructor.
- Know the location and operation of safety equipment in the laboratory from the first 5. meeting of the laboratory session.
- Drinking, eating and smoking are **absolutely forbidden** in the laboratory. 6.

- 7. Never work alone in the chemical laboratory.
- 8. Dispose the chemicals properly in the container provided, and according to the instructions given by the laboratory instructor. Do not simply pour waste chemicals down the sink.
- 9. Wear gloves only when strictly necessary. You should wear suitable gloves when handling hazardous chemicals.
- 10. Keep your laboratory space tidy and clean.
- 11. Be **aware** of the surroundings and be **alert** for others' mistakes.
- 12. Wash your hands thoroughly when you leave the laboratory.
- 13. Sign in and sign out on the laboratory attendance sheet.

## Safety glasses (goggles)

- Safety glasses should be worn at all times while in the laboratory.
- 2. Contact lenses should not be worn in the laboratory because they cannot be removed rapidly if reagents accidentally splash in the eye. Solvent vapours can build up behind the lens.

#### Chemicals

- 1. Handle all chemicals according to any specific directions on the container, or those given to you by your instructor.
- 2. Avoid contact with skin and clothing.
- 3. Wipe up spills immediately, especially near the balances and reagent shelf.
- 4. When pouring out reagents, hold the stopper in your hand. **Do not** put it on the bench. **Replace** the stopper immediately after use.
- 5. Avoid the inhalation of organic vapours, particularly aromatic and chlorinated solvents.
- 6. Volatile and flammable solvents **should be used** with great care. They **should be used** in a **fume hood** and **away** from possible sources of ignition.
- 7. To avoid contamination, **never** put the pipette into the reagent bottle and **never** return unused chemicals to their respective bottles.
- Take only sufficient amounts of chemicals and use them with care. Share any excess.
   Do not waste chemicals.

Disposals of chemicals

- 1. Dispose of chemicals as directed in each experiment.
- 2. Water soluble substances can be flushed down the drain with large quantity of water.
- 3. Water insoluble solids and liquid, should be placed in the waste container provided.
- 4. Chromium ion in the +6 oxidation state **should be reduced** to the +3 state with a mild reducing agent before disposal.
- 5. Volatile and flammable solvents **must never** be poured down the sink and drain. Use separate waste containers for disposal of halogenated and non-halogenated organic solvents.



# **Apparatus and Techniques**

The following is a summary of the basic analytical laboratory techniques and equipment you will use for this semester. Proper techniques are essential as acceptable error in a quantitative chemical analysis is seldom greater than 0.1%. There are a number of "hard and fast" rules presented that must be followed to minimise any hazards to you, your lab co-workers, and the lab equipment. **Read this section at the beginning of the semester and refer to any of this material as often as necessary.** 

#### I. "Clean" and "Clean and Dry" glassware

You will notice throughout the semester that you are asked to use "Clean" glassware at times and "Clean and Dry" glassware at other times. "Clean" glassware may be wet with your solvent (usually distilled water), and many times it is not worth the effort to dry the piece of glassware.

In general, if you want to maintain the concentration of the solution being transferred, you will want the final container to be "Clean and Dry". However, if you are only concerned about the amount of the compound being transferred, the final container needs only be "Clean".

"Clean" glassware means that all compounds and materials have been washed out. The final washing should be with your solvent. In analytical chemistry, the solvent is defined as the liquid or solution that you would use to dilute the solution in question, usually distilled water.

## **II. Desiccators and Handling Dried Compounds**

When using primary standards (compounds that are presumed to be 100% pure) in analyses, it is essential that there are no crystal waters present so that the mass of the primary standard measured on the balance is equal to the actual mass. Typically, compounds are dried by placing them in an oven at 105 to 120°C. After several hours, all (or most) crystal waters have been driven off. However, hot compounds cannot be accurately weighed (all items weighed on a balance must be at room temperature), so there must be a way to cool a dried compound without re-exposing it

#### to water vapour in the atmosphere.

Desiccators are containers designed to prevent the rehydration of solids. The bottom half of the desiccator is filled with an anhydrous salt, such as calcium chloride. The dried compound and its container sit in the top half, which is separated from the bottom half by a grid or screen. The desiccator lid can be sealed with vacuum grease to prevent water vapour from seeping inside.

Always cool a dried compound in a desiccator before weighing. A dried compound can be kept in the desiccator if that compound has to be available throughout the lab period.

#### **III.Electronic and Analytical Balances**

Electronic balances are quite simple to use. The "tare" button resets the mass reading to zero, and there is usually another button (sometimes labeled "cal" for calibrate) to set the mass units. You will always want the mass units in grams. Because electronic balances are fragile, you need to observe the following guidelines.

- Always clean the balance after using it. Use a soft brush or Kimwipe<sup>TM</sup> to remove 1. any extraneous material from the balance pan.
- All items and compounds placed on the balance **must be** at room temperature. This 2.can come into play when weighing dried compounds. Cool the dried compounds in a desiccator before weighing them on the balance.
- If you are making repeated weighing in the same container, it is recommended that 3. you always tare the empty balance and record the mass of the empty container. Then, record the mass of the container with sample, and calculate the mass of the sample by difference.
- If you are instructed to "accurately weigh" something, use a balance with 4 decimal 4. places. This is referred to as analytical balance. The maximum capacity of an analytical balance is usually small, therefore use only weighing boats or weighing paper as containers on the balance. On the electronic balance with 2 or 3 decimal places, you can often use small beakers or flasks as containers.

#### **IV.** Volumetric Flasks and Quantitative Transfers

Volumetric flasks are calibrated to contain an exact volume of solution when the solution level is exactly at the mark on the neck of the flask (the bottom of the meniscus should be exactly at this mark). Note the following rules in handling volumetric flasks.

- To clean volumetric flasks Each washing should have a volume that is about 10% to 20% of the capacity of the volumetric flask. Typically, you should wash the flask 3 times with dilute acid (e.g., 1 to 6 M HCl), 3 times with distilled water, and 3times with your solvent (if it is not distilled water). You can skip the acid washings if you have no solid residue in the flask.
- 2.Never heat a volumetric flask – Heating causes the glass to expand, changing

the volume it contains.

- Never place a solid directly into a volumetric flask Dissolve the solid in a 3. beaker and quantitatively transfer the solution to the volumetric flask. For example, if you want to make a 100 mL solution of NaCl, dissolve the NaCl in a beaker with 50 mL of water and then transfer this to the volumetric flask.
- **Never shake a volumetric flask** When mixing a solution in a volumetric flask. 4. gently invert the flask 8 to 10 times.

Quantitative transfers are vital to accurate analyses. In simple terms, 5. quantitatively transferring something means washing the original container and all glassware involved in the transfer with solvent and adding those washings to the final container, usually a volumetric flask. Here are some guidelines to transferring solutions and solids to volumetric flask.

Pour the solution through a funnel into the volumetric flask. Wash the original container with a small amount of solvent and pour the washing through the funnel into the volumetric flask. Repeat the washing 2 or 3 times if possible. Dilute the solution in the volumetric flask to the mark.

#### V. Volumetric Pipettes

Volumetric pipettes are calibrated to deliver an exact volume of liquid or solution. Volumetric pipettes have only one calibration mark. You may have seen graduated pipettes that have calibration marks throughout the length of the pipette, but these are far less accurate than volumetric pipettes. To fill a pipette, simply draw in liquid to the mark. Usually it is easiest to initially overshoot the mark and then let the liquid drain from the pipette until the bottom of the meniscus lies exactly on the calibration mark (Figure 1.1).



(a) Fill by suction.

(b) Stopper with finger. Wipe excess liquid with tissue.

Run out liquid (c)smoothly to graduation mark.

(d) Run into receiving vessel. Touch on sides to avoid splashes and give smooth drainage.

Figure 1.1: Handling volumetric pipettes

### Note the following rules in handling pipettes.

- 1. Never pipette with your mouth! Always use a rubber bulb or suitable pipette filler.
- 2. Always clean a pipette before its initial use For each washing, draw liquid into the pipette so that the bulb is quarter to half full (this can be less for large pipettes). Carefully swirl the liquid throughout the inside of the pipette (don't let liquid pour out the top!), and let the liquid drain from the pipette. Wash the pipette 3 times with distilled water and 3 times with the solution you are going to transfer (not just the solvent). If you are using the same pipette for different solutions, you

need to repeat the washing procedure every time you switch solutions.

- 3. Never force liquid out of a pipette Always let the liquid drain of its own accord. The calibration mark takes into account any liquid that is retained in the tip of the pipette. When the liquid has stopped draining, touch the tip of the pipette against the side of the container to release any hanging drops.
- 4. At the end of a laboratory session, always wash used pipettes 3 times with distilled water.

#### VI. Burettes

Burettes allow you to accurately deliver volumes of liquid that cannot be measured by volumetric pipettes or micropipettes. The proper use of burettes is essential to accurate titration analyses. In several cases, an analyte can be determined more accurately using a titrimetric method rather than an instrumental method. It is just that the convenience of the instrumental use is sometimes the deciding factor in choosing an analytical method. Note the following rules and guidelines in using burettes.

#### 1. To clean a burette

Fill the burette with distilled water and drain a large portion of it to see if any water adheres to the inside walls of the burette. If so, clean the burette with a few milliliters of soap solution and a burette brush, and wash the burette with three 5 mL portions of tap water. When washing the inside of a burette, pour about 5 mL of liquid into the burette with the stopcock closed. Carefully swirl the liquid for a few seconds so that it comes in contact with the entire inside surface area of the burette, and pour the liquid out the top. If no water adheres to the inside walls of

the burette, proceed to wash it 3 times with distilled water and 3 times with the solution with which you are going to titrate your sample.

#### 2. Getting rid of air bubbles

When you fill the clean burette with your solution, there will be air bubbles inside the stopcock, and you **must** remove these air bubbles. It is usually easiest to force bubbles out the stopcock, so simply open the stopcock and let the solution drain until the air bubbles are removed. If this is not working, you can try **gently** tapping the base of the burette while the solution is draining. This may force the air bubbles to rise through the solution. 3. Always record volume levels to the nearest 0.01 mL. Although the calibration marks are only at every 0.1 mL, you can always estimate the extra decimal place.

For example:



The reading in this picture is between 8.2 and 8.3 mL. The second decimal place would be estimated to be about 0.07 mL, giving a reading of 8.27 mL.

To obtain readings, it helps to hold a white card behind the burette. Note reading at a particular part of meniscus and always measure at this part.



(a) Note initial
 reading. Read to
 two decimal
 places.

(b) Assuming righthandedness, control flow with left hand whilst swirling flask with right hand. Titrate rapidly for first rough value.

(c) Approach
accurate end
point drop wise
then split drop
wise.

(d) Note finalreading to twodecimal places.

Figure 1.2: Titration by using a burette

- 4. Never record an initial volume level of 0.00 mL since there are no calibration marks above 0.00 mL, you have no upper reference with which to base any possible error in your reading.
- 5. Using your wash bottle, you can add "half drops" to your titration flask (see figure 1.2(c)). Run a drop of solution part-way out of your burette tip. Squirt distilled water on this half drop and into your titration flask. This procedure is perfectly legitimate because you are not worried about the concentration of anything in your titration flask.
- 6. At the end of an experiment, always wash your burette 3 times with distilled water.

# Writing Laboratory Report

After each experiment, you are required to write a laboratory report summarising your observations and explaining your conclusions about what you observed.

Formal laboratory reports contain the following sections: Introduction, Methodology, Results and Discussion, Conclusion and References.

#### Introduction

Introduction includes a brief discussion on the background of the technique(s) used in the experiment. The theory behind any calculation involved should be presented in the introduction as well. The introduction is followed by brief description on the objectives of the outlined experiment, i.e. it tells the reader what you intend to do and why you intend to do it.

#### Methodology

The methodology section should consist of the list of chemicals and apparatus used. All steps performed in the experimental procedure should be listed in the order that they were performed, in exactly the manner in which you performed them. Do not list a step as written in the experimental procedure unless you actually performed that step in an identical fashion. The steps should be written in past tense and using third person and passive voice. Diagrams of the apparatus used in the experiment may be included in this section.

#### **Results/Calculations/Data Interpretations**

The results section should list all data obtained, in raw form, with information provided as to how the data was obtained, as well as the experimental accuracy of all measurements. The data should be compiled into tables, if appropriate. Calculations should be included. The data obtained should be interpreted to fulfill the objectives of the experiment.

#### Discussion

The data should be discussed and evaluated, both positively and negatively in the discussion section. Do not try to manipulate the data to fit the results you think you

should obtain. Evaluate the data fairly, even if the data seem to contradict with theory you may have been expecting the data to follow. A discussion of possible sources of error should be included in this section.

# Conclusion

The conclusion is usually only a paragraph stating the outcome of the experiment and acts as a summary of the results and the discussion.

#### References

All materials that have been used in writing the laboratory report should be listed in the reference section. There are two common types of reference to printed work, journal article and book.

**Journal articles** are referenced by listing the authors (last name first), the year of publication, the title of the article, the title of the journal, the volume number and the page number. For example:

Kuivinenm, J. and Johnsson, H. (1999). Determination of Trihalomethanes and some Chlorinated Solvents in Drinking Water. *Water Research*, 33(5), 1201-1208.

Alves de Lima, R.O., Azevedo, L., Ribeiro, L.R. and Salvadori, D.M.F. (2003). Study on the Mutagenicity and Antimutagenicity of a Natural Food Colour (Annatto) In Mouse Bone Marrow Cells. *Food and Chemical Toxicology*, 41(2), 189-192.

**Books** are referenced by listing the authors (last name first), year of publication, the title of the book, the edition, the city of publication, the publisher and the page number. For example:

John, M.M., Johnston, D.O., Nettervile, J.T., Wood, J.L., Joesten, M.D. (1991). Laboratory Manual to Accompany World of Chemistry. New York: Saunders College Publishing.

#### Tan, Y.T. (1999). Kimia Fizik STPM. Shah Alam: Penerbit Fajar Bakti Sdn. Bhd.

**Internet articles** are referenced by listing the authors (last name first), title of page, date of retrieved and the web address. For example:

Schneider, J., Making Galvanic cells, retrieved on 29 September 2010 from http://www.chalkbored.com/lessons/chemistry-12.htm

Anonymous, Arsenic, retrieved on 22 May 2013 from <u>http://en.wikipedia.org/wiki/Arsenic</u>

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# **Marking Scheme**

# Laboratory Report (10%)

No.	Section	Marks (°o)
1.	Introduction	10

2.	Objectives	5
3.	Materials and Method	10
4.	Results	10
5.	Calculation/Data Interpretation	20
6.	Discussion	20
7.	Conclusion	5
8.	Post-lab Questions	10
9.	References	. 5
10.	Attachment of Raw Data	2
11.	On-Time Report Submission	3
	Total	100

# Laboratory Work (10%)

No.	Section	Marks (°°)
1.	Logbook	3
2.	Ethics	3
3.	Technique	2
4.	Question & Answer	. 2
	Total	10

# **PRK1016**

# Chemistry I

# Experiments

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# **Experiment 1.1:** Simple Acid-Base Titrations - Molarity

#### **OBJECTIVES**

To describe and carry out simple acid-base titrations using an indicator and then calculate the concentration of the solution in g  $L^{-1}$  and mol  $L^{-1}$ .

#### **INTRODUCTION**

It may be necessary to find the concentration of a substance in a solution. For example, if a river has been contaminated with acidic material, environmental authorities will need to analyse a sample from the river to find out the concentration of acid in the water. One way to do this is by conducting a reaction of a certain volume of the river water with an alkali to find out the volume of alkali required to neutralise the sample.



Figure 1.3: Acid and base titration

Finding out the volume ratio in which two solutions react is carried out using a technique known as titration and the usual laboratory process involves the use of a burette, a pipette and a conical flask.

It is important to realise that titrations can be carried out with any two solutions which react together. The only requirement is that there should be a method of determining when one solution has completely reacted with the other.

In the case of a titration between an acid and an alkali, it is usual to fill the burette with the acid, and have the alkali in the conical flask (Figure 1.3). An indicator is used to detect the 'end point' of the reaction.

2 Experiment 1.1: Simple Acid-Base Titration Molarity

#### **APPARATUS**

Glass rod Filter paper Pipette filler Filter funnel Thermometer Pipette, 10 mL Burette, 50 mL Beaker, 250 mL Conical flask, 250 mL

#### MATERIALS

Methyl orange Hydrochloric acid, 0.050 M Calcium hydroxide, saturated

# Retort stand with clamp

#### SAFETY DATA

Hydrochloric acid	Corrosive	Wear suitable gloves & safety goggles. Wash spillage with water.
Calcium hydroxide	Corrosive	Wear suitable gloves & safety goggles. Wash spillage with water.
Methyl orange	Toxic	Wear suitable gloves & safety goggles. Wash spillage with water.

#### METHOD

- 1. Rinse and fill the burette with 0.050 M HCl. Set up the burette in the burette clamp. Make sure that there are no air spaces in your burette. Record the initial burette reading to the nearest 0.01 mL.
- 2. Carefully decant the prepared saturated calcium hydroxide solution into a filter funnel and filter paper over a second conical flask, so as to collect the saturated solution of calcium hydroxide. You need about 100 mL of the solution.
- 3. Measure the temperature of the solution obtained.
- 4. Using a pipette, transfer 10.00 mL portion of this solution into a conical flask.
- 5. Titrate this solution with 0.050 M hydrochloric acid, using five drops of methyl orange as indicator.
- 6. When the titration approaches the end point, add HCl one drop at a time with constant swirling until a single drop of HCl causes a permanent orange colour that does not change on swirling. Record the final burette reading to the nearest 0.01 mL.
- 7. Repeat the titrations until two successive results agree to within 0.20 mL. Record your data in Table 1.1.

## CALCULATIONS/DATA INTERPRETATIONS

- 1. Write a balanced equation for the reaction between calcium hydroxide and hydrochloric acid.
- 2. From the average volume of 0.050 M hydrochloric acid used, calculate the number of moles of hydrochloric acid used in the titration.
- 3. Hence, calculate the number of moles for calcium hydroxide in the conical flask.
- 4. This number of moles of calcium hydroxide must have been in 10.00 mL of the solution. How many moles of calcium hydroxide would have been present in 1 L of

#### the solution?

- 5. Calculate the mass of calcium hydroxide in 1 L of saturated solution.
- 6. Complete the following sentence:

The solubility of calcium hydroxide is therefore

 $\underline{\qquad mol \ L^{-1} \ or \underline{\qquad g \ L^{-1} \ at \_ } e^{C}.$ 

7. The burette measures to  $\pm 0.1$  mL. Calculate the percentage (%) error.

# **POST-LAB QUESTIONS**

- 1. Define 'end point'.
- 2. What would you use to rinse your burette before titration? Explain.
- 3. What is the colour change of the indicator at the end point?
- 4. Based on the experiment above, explain how to ensure the results obtained are precise and accurate.
- 5. State the difference between absolute error and relative error.

4 Experiment 1.1: Simple Acid-Base Titration Molarity

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#### **Experiment 1.2: Determination of Chemical Formula and** the Percentage Yield of Product

**OBJECTIVES** 

To synthesise zinc chloride compound, determine its chemical formula and determine the theoretical yield, actual yield and the percentage yield of product.

#### **INTRODUCTION**

The elements present in a compound can be identified by means of qualitative analysis. Although qualitative analysis is very useful, it would be an added advantage to chemists if they know the quantitative aspect of the compound. The outcome from a quantitative analysis can be used to determine the composition of an unknown compound. Once the composition of the compound is known, its chemical formula can be determined.

This experiment illustrates (i) the law of conservation of mass and (ii) the law of constant composition:

- The total mass of the products of a reaction must equal the total mass of the (1) reactants.
- (ii) Any portion of a compound will have the same ratio of masses as the elements in

#### the compound.

Molecular composition can be expressed in three ways:

- In terms of the number of each type of atom per molecule or per formula unit (1) (the formula).
- (ii) In terms of the mass of each element per mole of compound.
- (iii) In terms of the mass of each element present to the total mass of the compound (mass percent).

The empirical formula of a compound gives the lowest whole-number ratio of the constituent atoms that is consistent with the mass ratios measured by experiment.

In this experiment, you will prepare a simple compound which is composed of  $tw {\rm o}$ 

elements, zinc and chlorine. Once the mass of zinc and the mass of the compound are known, the mass of chlorine can be determined. Using these masses, the percentage composition of the compound can be determined and the chemical formula can be calculated.

The percentage yield of a reaction, on the other hand, is the percentage of the product obtained compared to the theoretical maximum as calculated from the balanced equation. In carrying out a chemical preparation, the aim is to work carefully and recover as much of the desired reaction product as you can, and as pure as is possible

Experiment 1.2: Determination of Chemical Formula and the Percentage Yield of Produc-

and practicable. However, in any chemical process it is almost impossible to get 100% of the product. Therefore, the percentage yield shows the closeness of the measured amount with the expected amount. The calculation is:

Percentage yield = 
$$\frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100\%$$

**APPARATUS** 

#### MATERIALS



Zinc powder

Crucible Hot plate Glass rod Desiccator Weighing dish Porcelain tiles Analytical balance Measuring cylinder, 10 mL

# Hydrochloric acid, 6.0 M

## SAFETY DATA

Zinc powder	Irritant	Do not breathe dust. Wear safety goggles.
Hydrochloric acid	Corrosive	Wear suitable gloves & safety goggles. Wash spillage with water.

## METHOD

- 1. Weigh the crucible and record the exact mass.
- 2. Place approximately 0.25 g zinc powder into a weighing dish. Record the exact mass of the zinc powder used. Transfer all the zinc powder into the crucible.
- 3. Carefully add 10.00 mL of 6.0 M HCl solution into the crucible containing the zinc powder and stir the contents gently with a glass rod.

**Precaution**: A vigorous reaction releasing hydrogen will occur. Carry out this step in fume hood. Do not work near a naked flame.

- 4. Place the crucible on a hot plate in the fume hood and heat the content slowly so that the content will not splatter during the heating process.
- 5. Heat the content until it is completely dry, but make sure that the solid does not melt.
- 6. Allow the crucible and its content to cool to room temperature in a desiccator, and then weigh it.
- 7. Repeat the process of heating, cooling and weighing until the difference in mass is not more than 0.02 g. Record your data in Table 1.2.

6 Experiment 1.2: Determination of Chemical Formula and the Percentage Yield of Product

### CALCULATIONS/DATA INTERPRETATIONS

- 1. Write a balanced equation for the reaction that has taken place.
- 2. Calculate the mass of zinc chloride formed from the final weight of the sample.
- 3. Determine the mass of chlorine in the zinc chloride.
- 4. From the mass of zinc and chlorine obtained, determine the chemical formula of zinc chloride.
- 5. Using your periodic table, calculate the mass in grams of zinc chloride which should

have been prepared. This is the theoretical yield.

6. Calculate the percentage yield of the product.

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# **POST-LAB QUESTIONS**

- 1. Why must the reaction between zinc and hydrochloric acid be carried out in a fume hood and away from naked flame?
- 2. What is the purpose of repeating the process of heating, cooling and weighing until the mass is almost constant?
- 3. What observation did you notice when zinc powder was added to the hydrochloric acid followed by heating?
- 4. Give two reasons why the percentage yield might be below 100%.
- 5. Give two reasons why the percentage yield might be above 100%.

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Experiment 1.2: Determination of Chemical Formula and the Percentage Yield of Product

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