PRK
1026
Semester 2

CHEMISTRY II

LABORATORY MANUAL

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

UNIMAS
UNIVERSITI MALAYSIA SARAWAK
PUBLISHER

PRK1026

Chemistry II

Laboratory Manual
Semester 2

PRK1026

Chemistry II

Laboratory Manual

Semester 2

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

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.

Users may reprint this information, on the condition that full attribution is given to the editors and Universiti Malaysia Sarawak. Users may not revise, modify or edit this information without the permission of the editors and Universiti Malaysia Sarawak except for *use by qualified instructors in a chemistry laboratory course at an institution for higher education.*

Published in Malaysia by

UNIMAS Publisher,
Universiti Malaysia Sarawak,
94300 Kota Samarahan,
Sarawak, Malaysia.

Front cover credit : Dhana Jay Raja Gopal
Back cover credit : Wellcome Library, London Chemical Laboratory Room, Experimental Research Labs, Burroughs Wellcome and Co.,Tuckahoe, New York 1935. Wellcome Foundation Archives

Printed in Malaysia by

Malien Press Sendirian Berhad (522811-T)
G/FL Lot 184, Section 49, Jalan Abell,
93100 Kuching, Sarawak, Malaysia.
P.O Box 3068, 93760 Kuching, Sarawak.

Perpustakaan Negara Malaysia Cataloguing-in-Publication Data

PRK1026 Chemistry. II : Laboratory Manual Semester 2 / Edited by:
Chieng Tiong Chin, Sharifah Mona Abdul Aziz Abdullah, Wan Roslina Wan Yusof, Mohd Alhafiizh Zailani
ISBN 978-967-2008-16-3
1. Chemistry--Handbooks, manuals, etc.
I. Chieng, Tiong Chin, 1952-.
II. Sharifah Mona Abdul Aziz Abdullah, 1983-.
III. Wan Roslina Wan Yusof, 1986-.
IV. Mohd. Alhafiizh Zailani, 1986-.
540

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

Contents

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.
3. Keep long hair **tied back** and ensure that long or large necklaces are safely tucked away.
4. Report any spill, accident and dangerous incidents immediately to your instructor.
5. Know the location and operation of safety equipment in the laboratory from the first meeting of the laboratory session.
6. Drinking, eating and smoking are **absolutely forbidden** in the laboratory.
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)

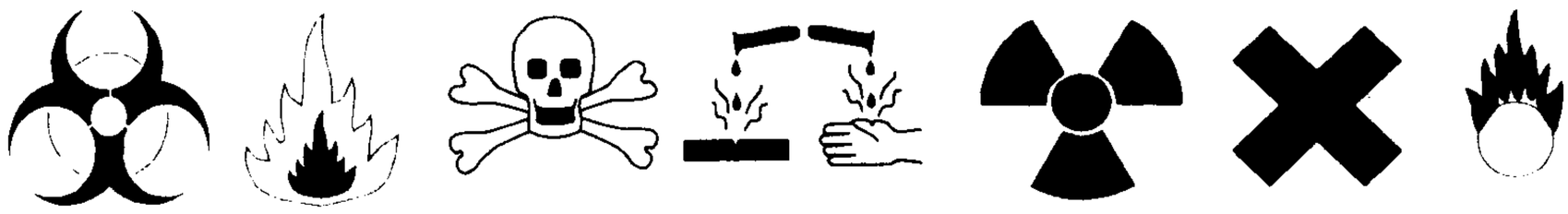
1. Safety glasses should be **worn at all times** while in the laboratory.
2. Contact lenses **should not be worn** in the laboratory because they cannot be removed rapidly if reagents accidentally splash in the eye. Solvent vapours can build up behind the lens.

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.
8. 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.

1. **Always clean** the balance after using it. Use a soft brush or Kimwipe™ to remove any extraneous material from the balance pan.

2. All items and compounds placed on the balance **must be** at room temperature. This can come into play when weighing dried compounds. Cool the dried compounds in a desiccator before weighing them on the balance.

3. If you are making repeated weighing in the same container, it is recommended that 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.

4. If you are instructed to "accurately weigh" something, use a balance with 4 decimal 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.

1. **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 3 times 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.

3. **Never place a solid directly into a volumetric flask** – Dissolve the solid in a 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.

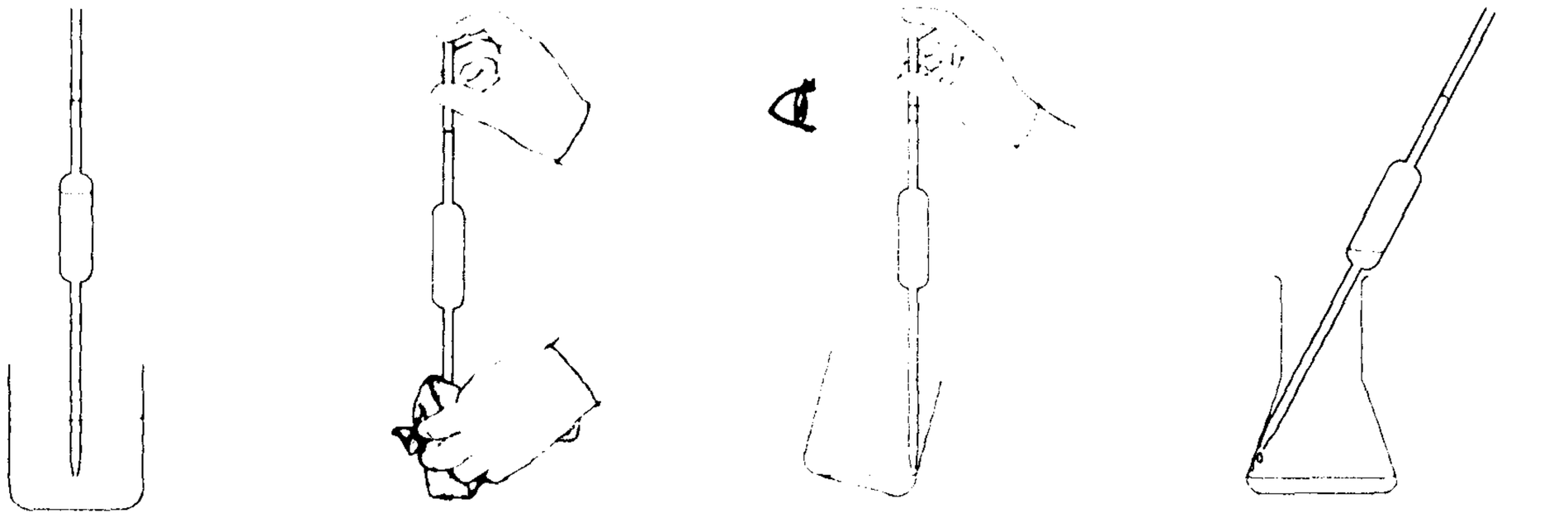
4. **Never shake a volumetric flask** – When mixing a solution in a volumetric flask, gently invert the flask 8 to 10 times.

5. **Quantitative transfers** are vital to accurate analyses. In simple terms, 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.

(c) Run out liquid 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

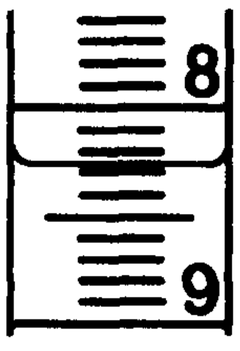
Burettes allow you to accurately deliver volumes of liquid that cannot be measured by volumetric pipettes or micropipettes. The proper use of burettes is essential to accurate titration analyses. In several cases, an analyte can be determined more accurately using a titrimetric method rather than an 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.

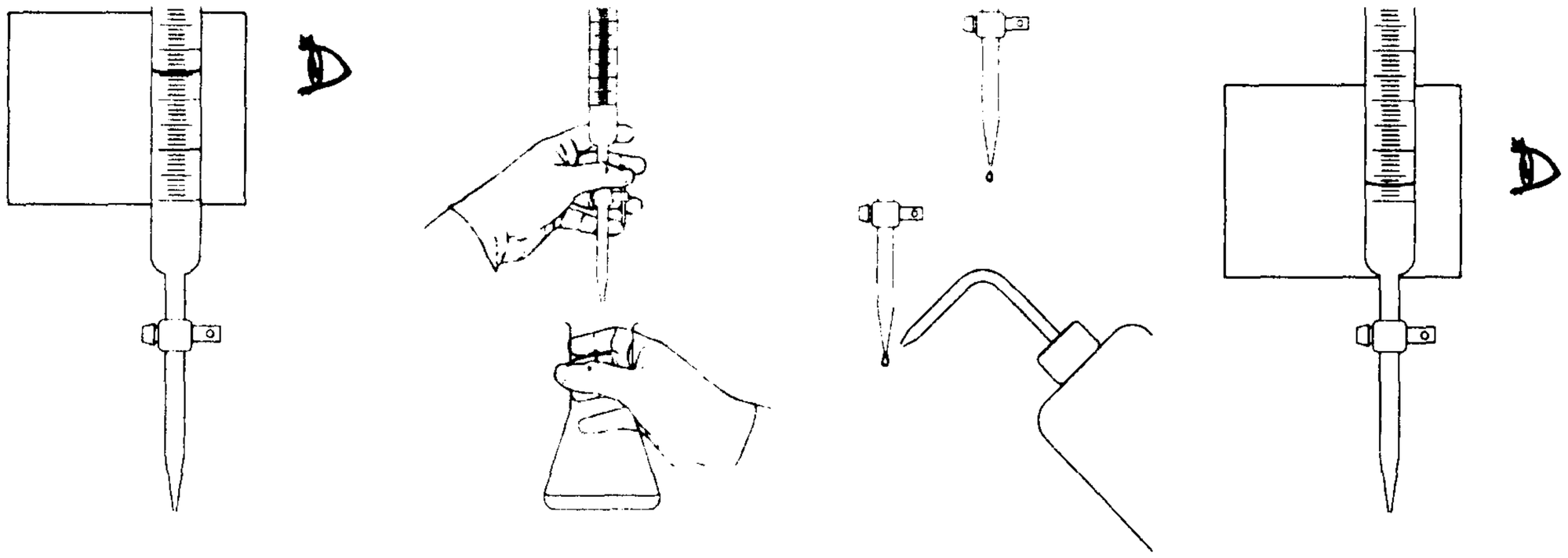
3. **Always record volume levels to the nearest 0.01 mL**. Although the calibration marks are only at every 0.1 mL, you can always estimate the extra decimal place.

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 right-handedness, 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 final reading to two decimal 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,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 http://en.wikipedia.org/wiki/Arsenic

Marking Scheme

Laboratory Report (10%)

No.	Section	Marks (%)
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**

PRK1026

Chemistry II

Experiments

Experiment 2.1: Qualitative Organic Analysis

OBJECTIVE

To identify some homologous series using qualitative analysis.

INTRODUCTION

Organic compounds can be classified into different families or homologous series depending on the presence of the functional groups. Table 2.1 shows a summary of some organic families.

Table 2.1: Homologous series and their functional groups

Families	Functional groups
Alkanes	All C–C single bonds
Alkenes	At least one C=C double bond
Aromatic compounds	Containing benzene ring
Alkyl halides	Halogen bonded to a carbon
Alcohols	At least one –OH group
Aldehydes	–CH=O group
Ketones	C=O
Carboxylic acids	–COOH
Amine	$C–NH_2$
Amide	$–CO–NH_2$
Ester	–COO–
Ether	C–O–C

Performing classification tests allows elucidation of the functional group present in an unknown organic compound. In this experiment, you will carry out various classification tests for organic compounds. This process is known as qualitative analysis. It allows you to apply the principles of organic chemistry.

APPARATUS

Parafilm
Glass rod
Test tubes
Water bath
Litmus paper
Thermometer
Test tube rack
Bunsen burner
Pasteur pipette
Beaker, 250 mL
Beaker, 100 mL
Test tube holder
Measuring cylinder, 10 mL

MATERIALS

Ethanol
Acetone
Propanal
Butan-1-ol
Butan-2-ol
Lucas' reagent
Brady's reagent
Tollens' reagent
Sodium carbonate
tert-Butyl alcohol
Glacial acetic acid
Fehling A solution
Fehling B solution
Sulphuric acid, concentrated
Potassium dichromate(VI), 1%

SAFETY DATA

Butan-1-ol	Harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
Butan-2-ol	Harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
tert-Butyl alcohol	Harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
Ethanol	Harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
Propanal	Corrosive, harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
Acetone	Harmful & highly flammable	Wear suitable gloves & safety goggle. Eliminate ignition sources.
Tollens' reagent	Corrosive & stain on the skin	Wear suitable gloves & safety goggles.
Lucas' reagent	Toxic, irritant	Wear suitable gloves & safety goggles.
Sodium carbonate	Irritant	Wear safety goggles.
Sulphuric acid	Corrosive	Wear goggles & suitable gloves. Wash spillage with water.
Fehling A & B solutions	Corrosive & harmful	Wear suitable gloves & safety goggles.
Brady's reagent	Harmful, explosive & highly flammable	Wear suitable gloves & safety goggles.
Potassium dichromate(VI)	Toxic, corrosive & oxidising	Wear suitable gloves & safety goggles.
Glacial acetic acid	Corrosive & harmful	Wear nitrile gloves & safety goggles.

METHOD

Part A: Aldehydes and Ketones

Propanal and acetone are used in this classification test. Record all the observations in Table 2.1.1.

i. Brady's Reagent

1. Place 1.0 mL of Brady's reagent in two separate test tubes.
2. Add five drops of propanal into one of the test tubes and acetone to the other test tube.
3. Shake the test tubes and record the changes observed.

ii. Fehling's Solution

1. Prepare two test tubes and label them as Test tube 1 and Test tube 2.
2. Mix 2.0 mL of Fehling A solution and 2.0 mL of Fehling B solution into Test tube 1. Prepare the same mixture in Test tube 2.
3. Add five drops of propanal and acetone in Test tube 1 and Test tube 2, respectively, and shake them.
4. Heat the mixture in a boiling water bath.
5. Record the changes observed.

iii. Tollens' Test

1. Place 2.0 mL of Tollens' reagent in two different test tubes. Add five drops of propanal to one of the test tube and acetone to the other.
2. Shake the mixtures and heat in water bath.
3. Record the changes observed.

Part B: Alcohols

Butan-1-ol, butan-2-ol and *tert*-butyl alcohol are used for the classification test (i), (ii) and (iii) below. Ethanol is used for the esterification test. Record all the observations in Table 2.1.2.

i. Litmus Paper

1. Test each alcohol sample with red and blue litmus paper.
2. Record the observations.

ii. Oxidation Test

1. Prepare three test tubes and label them as Test tube 1, Test tube 2 and Test tube 3.
2. Add 1.00 mL of 1% potassium dichromate(VI) solution, followed by two drops of concentrated sulphuric acid into each of the test tube.
3. Add five drops of butan-1-ol, butan-2-ol and *tert*-butyl alcohol into Test tube 1, Test tube 2 and Test tube 3, respectively, and heat them in water bath.
4. Record the observations.

iii. Lucas' Reagent

1. Place 1.00 mL of Lucas' reagent in three different test tubes.
2. Add five drops of the three alcohol samples separately to the three test tubes and cover with parafilm.
3. Shake the mixture and leave for ten minutes.
4. Record your observation and the time required for the changes to occur.

iv. Esterification Test

1. Add 1.00 mL of glacial acetic acid and three drops of concentrated sulphuric acid to 2.00 mL of ethanol.
2. Shake the mixture until it is homogenous and warm it in a water bath.
3. Add 3.00 mL of distilled water and note the smell of the product formed.

Part C: Organic Acid

Glacial acetic acid is provided for the classification tests of organic acid. Record all the observations in Table 2.1.3.

i. Litmus Paper

1. Test the sample with red and blue litmus paper.
2. Record the observations.

ii. Sodium Carbonate

1. Add a small amount of solid sodium carbonate to the test tube containing distilled water and glacial acetic acid.
2. Record the observation.

CALCULATIONS/DATA INTERPRETATIONS

Interpret the results based on your observations.

POST-LAB QUESTIONS

1. Define primary alcohols, secondary alcohols and tertiary alcohols.
2. Compare the chemical properties between aldehydes and ketones.
3. What is Lucas' Reagent?
4. What is Fehling Solutions?
5. What is Tollens' Reagent?

Experiment 2.2: Solvent Extraction and Separation of Plant Pigments by Thin Layer Chromatography

OBJECTIVES

To extract and isolate pigments present in plant leaves using thin layer chromatography (TLC).

To learn terms such as mobile and stationary phase, solvent front, and retardation factor.

INTRODUCTION

When we see a green leaf from a plant what we actually see is light reflected off the surface of the leaf, specifically, the green wavelengths of light. When leaves begin to die, we see different colours from the same leaves. Actually, leaves contain many different pigments – the compounds that absorb and reflect different wavelengths of light.

This lab focuses on the separation of pigments using thin layer chromatography (TLC). TLC is used to separate organic molecules based on their affinity for a mobile and stationary phase. In thin layer chromatography, the stationary phase is composed of a plastic or aluminium film coated with a thin layer of silica gel. The mobile phase or solvent is selected based on the degree to which it interacts with the stationary phase and the solute (or pigments). The mixture to be separated is applied as a spot or line to the solid phase while the mobile phase is allowed to pass through the solute dissolving it along the way. As the solute dissolves in and moves with the solvent along the stationary phase, a chromatogram is formed.

Visible pigments include carotene, pheophytin, chlorophyll a and b, lutein, xanthophylls, and anthocyanins. Due to the polarity of the silica gel plates (stationary phase), the polarity of the developing solvent (mobile phase), and the molecular shape of the analyte (pigment), the pigments separate into distinctive bands of colour.

Table 2.2: Colour of pigments

Pigment	Colour
Carotene	golden
Pheophytin	olive-green
Chlorophyll a	blue-green
Chlorophyll b	yellow-green
Lutein	yellow
Xanthophylls	yellow
Anthocyanins	red