SIC1011

Basic Laboratory Techniques

Name:

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Preface

This laboratory class caters **elemental practical skills applied in all sections of chemistry**. The experiments are embedded into specific contexts and located at different laboratories, which are operated by the organisational sections of the Chemistry Department, *i.e.* Inorganic Chemistry, Physical Chemistry and Organic Chemistry. This separation, however, is solely based on organisational reasons and differing infrastructure for laboratories, but does not reflect varying skill requirements for different directions of chemical laboratory work.

The emphasis of this course is **NOT the theory** behind the experiments**, but the practical skill for laboratory operations**. The focus on laboratory skills is reflected in a final **practical examination**, where students will have to demonstrate effective use of laboratory techniques learned in the experiments. These examinations differ from the previous experiment, since the time allocation for exams does not reach those for the respective experiments. Only selected laboratory operations will be examined on individual samples. The experimental output will be marked based on objective criteria. These can cover the quality and amount for sample preparations (e.g. purifications like crystallisation or distillation) or the accuracy of analytical determinations (e.g. titration). In addition, observations during the operation of the exam tasks, reflecting the experimental setup and conduction, may be considered as well.

Besides the indicated motoric lab skills, lab safety is another focus of this course. While the theory is addressed in lectures, its practical execution will be evaluated during the experiments. However, working in a chemical laboratory requires not only experimental skills and consideration of safety, but also record taking of operations and observations as well as writing of comprehensive reports. Lab-notes can be crucial documents in practically every job involving a lab-component, with potential impacts on the entire organisation/company. Therefore, the recording of lab-notes is an important lab skill as well. Records have to be taken immediately **during the operation of the lab** in a book. This requirement reflects the potential use of lab notes for documentation purposes, e.g. in case of a patent conflict in a company. Numbered pages are encouraged, while continuous and dated records are essential. A lab jotter does not require particular neat writing, but it must enable another person to follow the entire experiment – preferably without need of additional documents, like a lab manual or a reference procedure. Therefore, your lab jotter alone should enable you to write the experimental section of a report without further reference to the lab manual. In view of the documentation function, **lab-records must not be erasable**. Mistakes shall be documented by crossing out, without permanently blocking the wrong record. This also means that the removal of pages from a lab jotter is not-permitted. Proper lab records need to be learned. Typical challenges for students are:

1. parallel recording of notes during the execution of the experiments
2. recording of observations during the experiment.

Experimental science requires a careful observation of experiments. However, it requires training to record observations. Particularly important are observations, which you cannot explain at the moment. In case of an unexpected result of the experiment, such observations provide valuable clues. While during your studies unexpected observations may help to explain a failure of an experiment, in later years such observations can lead to new discoveries. Unfortunately, it is not possible to predict, which observation has high impact at the beginning. Therefore, it is important to practice the recording of observations from the beginning on.

Course Learning Outcomes

1. Follow guidelines of safe and clean working inside a chemical laboratory.
2. Complete volumetric analysis in a chemical laboratory.
3. Perform basic separations of chemical compounds inside a chemical laboratory.

Chemistry Laboratory Safety Agreement

In the interest of safety and accident-prevention, there are regulations to be followed by all students in designated Chemistry Laboratories at the Department of Chemistry, Faculty of Science, University of Malaya. Faculty and staff members are authorised to deny the use of any laboratory to students who do not adhere to the regulations mentioned below or in instances when the safety of any of the student, staff or faculty member in the laboratory might be jeopardised.

The regulations for all Chemistry Laboratories are as follows:

1. Proper attire must be worn at all times in all laboratories. This includes shoes that completely cover the foot (no high-heeled shoes), and a shirt that covers the entire upper torso, including the stomach and the back. Lab coats must be worn in the laboratory at all time. Long hair must be tied back. No loose or baggy clothes and dangling jewellery is allowed.
2. Safety eyewear must be worn at all times during laboratory sessions.
3. Food, drinks, chewing gum, tobacco products, and applying cosmetics are prohibited in the laboratories. Hands, pencils, pens, etc. must be kept away from the eyes, nose, and mouth in order to avoid contamination.
4. Fume hood sashes are not to be opened beyond the 18'' mark when in use. (Never put your head into the hood.)
5. Be organised. Maintain a clean, open work area free of anything except materials directly required for the exercise. Keep laboratory material/equipment away from edges of work surfaces and electrical cords from hanging below the surface of tables.
6. Equipment and/or chemicals should never be taken out of the lab unless authorised by the instructor or laboratory staff.
7. Many of the lab activities have students moving around the lab or involve moving objects. Be alert and aware of what's going on around you.
8. Be familiar with the location and the use of the following in your laboratory: e.g. broken glass receptacle, first-aid kit, emergency gas shut-off valves, closest fire alarm, fire extinguisher, eye wash, safety shower, and emergency exits and routes.
9. It is of utmost importance to know the rooms that are off-limits to the students. The students should not enter those prohibited areas.
10. Be prepared. Study the assigned experiment before you come to lab. Being familiar with the lab exercise to prevent confusion and accidents. The preparation for an experiment also includes the study of safety data sheet for the chemicals used. These can be found on the internet. No unauthorised experiments are to be performed. Students must follow the procedural instructions in the lab handout/manual unless modifications to the procedures have been announced by the laboratory supervisor, in which case the student must follow the supervisor's procedural instructions.
11. NEVER TOUCH ANY FORM OF BROKEN GLASS. Broken glass should be disposed of only by laboratory staff.
12. Unused reagents should not be returned to the reagent stock bottle. One should make sure to take only what is actually needed out of the regent bottle. Reagents must not be contaminated.
13. CONTACT LENSES should not be worn in the lab, as chemicals can get between the eye and the lens.
14. Lab experiments have been designed to minimize unnecessary exposure to any hazardous substances; however, it is not advisable for pregnant women or those with certain medical conditions to be exposed to any chemicals. We cannot ensure that a pregnant student will not be exposed to chemicals that might be unhealthy for her or her fetus. In addition, we cannot know the level of exposure, the length of exposure or the

number of encounters that might occur with any chemical during a semester. By maintaining the safety rules, we expect that all students, including a pregnant student, should be able to carry out lab procedures safely. However, it is the Department's professional advice that pregnant students should be advised NOT to take a lab course unless she is willing to understand and assume the risks. She should certainly be seeking and following proper medical advice from her physician.

15. If you are pregnant, or you suspect, should become, or plan to become pregnant during the semester, or have any medical condition or concern, including but not limited to the following, immunocompromised system, seizures, epilepsy, severe allergies, it is your, the student's, responsibility to consult with your medical care provider regarding any medical issue associated with taking this lab. Students are encouraged to provide their physician with a list of the chemicals that they might be exposed to while in lab. They should also check the MSDS sheets to be aware of the hazards of the chemicals.

SAFETY INFORMATION ACKNOWLEDGEMENT
INFORMED CONSENT

(Sign and keep for your record)

I acknowledge receipt and that I have read and understand the lab safety regulations and that I received a briefing on these regulations from my laboratory Instructor/Lecturer. I also acknowledge that I was given the opportunity to ask any relevant questions during the safety briefing. I understand that there may be inherent risks and possible hazardous exposure with laboratory experiments depending on one's medical condition. If pregnant, or you suspect, should become, or plan to become pregnant during the semester, or have a medical condition that may be affected by my participation in this laboratory session, I understand that it is my responsibility to discuss any and all issues with my medical care provider.

Further, I accept any and all risk associated with the use of the Chemistry laboratory(s) and the equipment contained therein. I also understand that I am responsible for my personal property at all times. By signing this agreement, I fully understand and consider it my responsibility to comply with the safety regulations outlined above. I hereby agree for myself, my family, successors, and assigns to hold harmless the University of Malaya (UM), Department of Chemistry of the University of Malaya, Faculty of Science of the University of Malaya, Lecturers, Laboratory Staff and assigns from any and all claims, causes of action, suits, liabilities, damages, losses, demands, costs, expenses or judgments for damages or injuries to myself or others arising from my participation in the lab, whether or not I consulted a medical provider as delineated above.

Signature of the student: _____ Course: _____

Name: _____ Lecturer: _____

Matric number: _____ Session: _____

IC number: _____ Semester: _____

Date: _____

Provide the name and telephone number of two "Emergency Contacts" that can be reached during lab class times. Please note that your medical or physical condition may be released to the contact person at the time of the emergency call.
Indicate the relationship to the person and also the telephone location (office, home or cellular). Please print clearly.

_____	_____	_____
Emergency Contact (Name)	Relationship	Phone
_____	_____	_____
Emergency Contact (Name)	Relationship	Phone

Student's copy

SAFETY INFORMATION ACKNOWLEDGEMENT
INFORMED CONSENT

(Return this signed page to your lecturer)

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Signature of the student: _____ Course: _____

Name: _____ Lecturer: _____

Matric number: _____ Session: _____

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Provide the name and telephone number of two "Emergency Contacts" that can be reached during lab class times. Please note that your medical or physical condition may be released to the contact person at the time of the emergency call.
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Emergency Contact (Name)	Relationship	Phone
Emergency Contact (Name)	Relationship	Phone

Department's copy

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SAFETY IN THE FIRST YEAR LABORATORY

Further information in the details of the safety and health practice in The University of Malaya can be found at:

Office of Safety and Health, University of Malaya	University of Malaya Safety Handbook	Manual Keselamatan dan Kesihatan Pekerjaan, Universiti Malaya

The University has a statutory obligation to comply with the safety requirements and you, as a student, have a duty to abide by the regulations. The following notes are to guide you in good laboratory practice and to familiarize yourself with the safety aspects of your laboratory work.

Emergency Telephone Numbers:

National Emergency Number	999 (Mobile phone, dial 112)
University Security Office	03 7967 7070 / 3582
University Malaya Medical Centre (UMMC)	03 7949 2898 / 2190
Students' Health Clinic	03 7949 2837 / 3737
Office of Safety and Health (OSH)	03 7967 7925
Department of Chemistry office	03 7967 4024 / 2128
Pantai Fire Station (Jalan Pantai Baru)	03 2282 4444
Pantai Police Station (Jalan Pantai Baru)	03 2282 4222 / 2207

Responsible staffs

Lecturer in charge

Inorganic Chemistry Laboratory

Ms. Zailawati Mohamad Zakaria	
Ms. Nurul Hafizah Hamid	**(Inorganic Chemistry I)**
Mr. Muhamad Hafizie Muhamad Sofi	(Inorganic Chemistry II)
Mr. Sugakumar A/L Varuthan	(Inorganic Chemistry III)
Ms. Maemawati Mohamad	(Inorganic Chemistry III)

Physical Laboratory

Mr. Mohd Shukri A. Aziz	
Mr. Hashim Mohammad Salleh	**(Physical Chemistry I)**
Ms. Najiyah Ghazali	**(Physical Chemistry I)**
Mr. Md. Hakim Zakaria	(Physical Chemistry II)
Ms. Noor Azlin Che Din	(Physical Chemistry III)

Organic Laboratories

Ms. Dara Fiona Mohamad	
Ms. Nurul Ain Hassani	**(Organic Chemistry I)**
Ms. Siti Nur Faridatul Asyikin Said	(Organic Chemistry II)
Ms. Juria Seko	(Organic Chemistry III)
Mr. Zulkiflee Hussin	(Organic Chemistry III)

Safety is the primary concern in any chemical laboratory. Chemicals are almost all potentially hazardous. Fortunately, with sensible and correct precautions, the risks can be minimized if basic safety practices are followed. The responsibility for laboratory safety lies with everyone working in the laboratory. Sensible laboratory conduct does not mean memorizing a list of rules! The true test is the actual conduct in the laboratory and safety rules apply to all laboratory activities. Individual safety is affected by the action of fellow workers in the laboratory. Therefore, it is in everyone's best interest to follow safety work practices.

General Safety Rules for the Undergraduate Laboratories

The guidelines below are recommended for working safely in the laboratory.

- No work is to be carried out unless a member of staff is present.
- Plan your work. Follow instructions. If you do not know how to do the experiment safely, ask the lecturer or demonstrator.
- Know the location of all exits for the laboratory and the building. There are two exits in every teaching lab.
- Know the location of the alarm and fire extinguishers and how to operate them. There are two fire extinguishers located at the two sides in the lab.
- Know the location and use of safety showers, eye-washers and safety aid boxes. The safety shower and eye washer are located right next to the exit the lab.

Fire extinguisher, eye wash and safety shower in First Year Laboratories.

- Know the location of the nearest telephone that can be used during an emergency.
- All persons in laboratories (whether or not they are actually doing practical work) **must wear safety glasses** or goggles and laboratory coats. Be aware, that spectacles are NO REPLACEMENT for safety glasses, as they lack side protection. If you need to ware spectacles, ensure that your safety glass can cover the glasses properly. You might find safety glasses a nuisance to wear, but your eyes are very sensitive and chemical spills inside the eye bear high chance for permanent damage. You are not allowed to wear contact lenses in the laboratory.
- Hair should be secured so that it does not hang below the neck. Other clothing that may become entangled should also be secured. It is important to wear suitable clothing. In view of fire hazards, natural fibres, like cotton, are encouraged. Your footwear must incorporate flat heels, slip-resistant soles and the uppers must fully enclose the foot.
- No food, drink (including drinking water!), cigarettes and cosmetics are allowed to be taken into the laboratory or storage place for chemicals.
- Do not smell or taste chemicals.

- Know the potential hazards of the materials and equipment with which you will work. The preparation for an experiment involves the study of the respective material safety data sheets for all chemicals used in that experiment. Refer to the chemicals' Material Safety Data Sheet (MSDS) before usage.
- Do not make skin contact with any substances. Use gloves where necessary, always remembering that they are semi-permeable. Gloves typically only provide a short time protection; when you notice the glove to get wet, remove it asap and replace with a new one. This particularly applies for the common single-use protective gloves.
- Experiments must be conducted on clean working surfaces; any spillage should be cleaned immediately. A high standard of tidiness should be maintained at all times. Contaminated surfaces and equipment must be cleaned as soon as it is practicable after use. The equipment should then be put away. Do not clutter bench space with unused equipment and bottles of chemicals.
- Waste should be disposed of in appropriate containers. Special waste includes:
    - Broken glassware and other sharps
    - Contaminated solid waste (e.g. used silica gel)
    - Mercury waste (e.g. broken thermometer)
    - Aqueous waste containing heavy metals (e.g. nickel or manganese solutions)
    - Organic waste: Organic waste is segregated into two (2) groups and stored separately, *i.e.* halogenated waste (examples are chloroform, dichloromethane) and non-halogenated waste (examples are acetone, alcohol, toluene, xylene).
- Bags and other personal items should be placed in the lockers provided outside the laboratory and not left along corridors or on benches.
- All accidents and dangerous occurrence must be reported immediately to the lecturer in charge or the demonstrator or the laboratory assistant. The first aid box is located inside the preparation room of the laboratory. The accident book is also kept in the preparation room; the laboratory assistant must file out a report for all incidents.
- It is important to ensure that hands are washed, and all protective clothing removed before leaving the laboratory.
- Do not wear laboratory coats, gloves or other personal protective clothing outside of the laboratory and in non-laboratory areas. These clothing may have become contaminated.

Additional Guidelines

Remember that in a laboratory you have fellow students around you. They do not know what you are doing, but they hope and expect that what you are doing is sensible and safe. Always think carefully about what you are about to do.

- Know the lecturer in charge, the demonstrator and the laboratory assistants of the laboratory.
- Undergraduates are not allowed to work or even be in any of the teaching laboratories at any time outside of the specified laboratory hours, unless they have explicit permission from the lecturer in charge. This includes times before and after class, and the lunch break.
- Students should come to the laboratory on time and be prepared by studying the experiment. Therefore, plan your activities before you come to the laboratory.
- Write everything you do, and observations in your notebook so that you can trace your action and make corrections if necessary. Please designate one notebook for this purpose and use it for the whole session / cycle.
- Do not use cracked or broken glassware. Check glassware before using it.
- Never use open flames, unless instructed by the lecturer in charge. If flames are permitted, plan your experiments so that you never leave your flame unattended. There are other sources of heat such as steam baths and hot plates.

- Handle all chemicals with care and read labels before attempting to get them.
- Use a spatula to get solid chemicals. Never using your fingers.
- Be careful not to contaminate reagents with your spatulas or droppers. Do not take more than needed. If you take too much of a chemical or reagent, give it to a fellow student – but do not return it to the bottle.
- Do not wander off with the only bottle of reagent that everyone needs; keep it in its assigned location.
- Do not pipette by mouth. Use only mechanical pipetting devices.
- Never look directly into the mouth of a flask containing a reaction mixture.
- Never point a test tube or reaction flask towards yourself or your neighbour.
- When using a separating funnel, vent frequently and remove the stopper immediately upon setting it upright for separation.
- Never use a thermometer as a stirrer! If a mercury thermometer breaks, immediately contact the lecturer in charge, the demonstrator or laboratory assistant.
- Turn off water, burners or electrical equipment when not in use.
- Wash your glassware at the end of the laboratory session. You will have clean and dry glassware ready to be used for the next laboratory class.
- Make sure glassware or equipment is put away in the correct locker – your personal locker or the common locker.
- Clean your work area and equipment used before leaving the laboratory.

Experiments' Planning

The laboratory component is an essential part of SIC1011. Attendance at all laboratory classes is compulsory. Students are expected to be prepared. Students may be prohibited from doing an experiment, if we believe that they are unprepared.

LAB SCHEDULE

In view of the organization of this course in sections, which are operated at different laboratories, it is important to have your own individual lab schedule. Therefore, please prepare the following personal schedule. Although lab-classes are typically scheduled from week 2 to 13, public holidays may require an adjustment of the schedule.

Lab	Sem.-Week	Date	Laboratory	Experiment
1				Experiment 1
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				Exam:
12				Exam:

PREPARATION FOR THE LAB

A proper preparation for a laboratory class involves:

- Studying of the lab-manual for the respective experiment
- Reviewing of the hazards for the experiment
  This particularly covers the study of safety data sheet for **all chemicals used** in the experiment.

Comprehension of the Experiment

A proper understanding of an experiment is essential for its safe and successful execution. All actions described in the experimental procedure shall be critically red and questioned on their importance for a successful operation of the experiment. If the relevance of a certain step cannot be determined, the respective section shall be discussed with the lecturer in charge during the briefing section of the lab. In general, every step in an experimental procedure should have a rational, otherwise the respective step shall be omitted.

The study of the laboratory manual might not be enough to grasp the scope of an experiment. In this case, additional references (text books, internet resources etc) shall be looked into. This time-consuming preparation is reflected in the learning time assignment for laboratory classes, which includes a significant time allocation for the preparation before the actual class.

Based on the study of the laboratory manual you shall be able to:

- Identify the objectives for the experiment
- Relate the conduction of the experiment with the learning objectives
- Construct a roadmap for the experiment, to guide you during the actual lab class

Hazard Analysis

Safety data sheet for most chemicals can be easily found at the internet. Although the information is specific for every product, the safety data sheets for different manufacturers rarely differ much. For a chemical hazard analysis of an experiment, it is, therefore, not important to know the specific brand and product code of the chemical.

The study of safety data sheet for chemicals exceeds a simple reading of the document. Safety data sheets are prepared to summarise all known hazards associated with the chemical. However, the relevance of the hazards depends on the conditions, at which the chemicals are operated. A simple example might help to illustrate this.

> A solid chemical with severe risk for lung damage upon inhalation requires considerable measures, if fine powders are operated in possibly open machinery places, e.g. milling of the chemical into dust. On the other hand, the lung hazard for the same chemical can be considered irrelevant, if the chemical is operated in large chunks to be dissolved in a solvent, since the inhalation risk of either a solution or large particles is near to zero.

The analysis of hazards for a chemical, therefore, requires a cross-check of potential hazard scenarios within a safety data sheet with the experimental conditions and the specific specification of the compound (e.g. fine powder, granules or blocks). This affects not only the identification of hazards but also has impact on personal protective equipment needed for the operation. While dust or even respirator mask are highly recommended for the milling operation, no particular protection is required for the other application above.

If significant hazards of a chemical for an experiment are detected during the preparation of an experiment, the matter shall be discussed with the lecturer in charge of the experiment during the briefing session of the experiment.

Experiment Roadmap

Experimental procedures are rarely written to follow gradually word-by-word. Typically, a procedure shall provide all information required for the successful operation of the experiment. However, a proper following of the procedure frequently requires the reading of **the entire process** prior to operation at the lab. Again, a simple example can help to illustrate this:

Add solution B to solution A **at a rate to maintain moderate reflux of the reaction mixture**. In case of a word-by-word instruction, the constraining statement for the experiment will be read at a time, when the addition of solution B to solution A is already complete, hence too late for any for any adjustments. The second part of the sentence in the procedure cautions on the operation of the previous instruction – in this case to slowly add solution B to solution A in order to avoid overheating the reaction mixture due to a strong exothermic reaction. In order to simplify the execution of the experiment, it is recommended to transform the experimental procedure into a diagram, scheme or tabular instruction that can be followed stage by stage. This experimental roadmap can subsequently be used as detailed instruction for the execution of the experiment.

Creation of experiment roadmaps

1. Separate the experimental procedure into a set of simple operation instructions
2. Combine the individual instructions in a hierarchical order, or road map
3. Specify important conditions that must be met prior to any step. Place a caution reminder note **BEFORE** the respective step and highlight it to avoid overreading.
4. Include a statement for waste treatments and cleaning-up instructions, if relevant for the experiment.

Images can be faster captured than text. Therefore, an experimental roadmap in form of a diagram or scheme or flow chart (with limitations of words) is usually more effective than text-based instructions.

LABORATORY NOTEBOOK

A lab notebook is used to record all the work carried out in the laboratory and the experimental data. In industry or in academic research, it is an important legal document that can be used to provide evidence regarding the discoverer and date of discovery of new chemicals or processes.

In the undergraduate laboratory course, it is important to develop the skill of recording a good lab notebook. The records will be needed to generate lab reports at some point in the course, and the keeping of the lab notebook will be assessed regularly by your lecturer.

Marks will be awarded for continued good use and practices of the notebook throughout the laboratory classes.

All relevant aspects of an experiment should be recorded, together with the order in which steps were carried out. All observations should be noted, in principle even those that at first sight appear unimportant.

General Guidelines

1. Use ballpoint pen and press hard if you are using duplicate pages.
2. Write on one side only.
3. Do not erase or use whiteout. If you make a mistake, draw a single line through the error (strikethrough) and write the correct entry on the top or side of it.
4. Do not remove an original page. If the entire page is incorrect, draw a single diagonal line through the page and state the reason for this line.
5. Record all data and results (with units) directly into your notebook.
   DO NOT record data on scrap paper, your hand etc. **to be transferred later**. A laboratory notebook does not need to look nice, but must be logically ordered and reasonable readable.
6. Start a new page for each new experiment.
7. Write the title of the experiment, date and your name at the top of each page.
8. NEVER skip a space for later additions.
9. Be neat and thorough! Someone should be able to pick up your notebook twenty years from now and be able to repeat your experiments.
10. NEVER mix-up instructions for the lab with records of experimental procedures. A lab notebook should only **reflect solely the procedure you HAVE followed**, not the one you intended to follow. Instruction notes of schemes for the operation of an experiment – typically prepared before the lab starts – are useful and encouraged. However, this should NOT be placed into the laboratory notebook.

At the beginning of each experiment, record:

- The date
- Structural formula (abbreviated, if necessary) and all reagents in order of addition
- Molecular formula (preferably structural formula) and molecular weights
- Literature references for the procedure (or for analogous preparations)
- List of apparatus (with sketches in unusual cases)
- Simplified procedures from your reading of lab manual; it can be a flow chart or schematic diagram, to your preference, which transforms the lengthy and wordy procedures into simple yet informative procedures at a glance. If this scheme is placed inside the laboratory notes, it is essential that the section is clearly labelled as ‘instructions or ‘procedure’ and **NOT MIXED UP WITH ANY EXPERIEMNTAL RECORDS** during the experiment. Also, instruction notes do NOT REPLACE any MISSING INFORMATION within the experiment records. The lab notes recorded during the experiment must be complete on its own.

Good Lab Notebook Organization.

The first page of the lab notebook should be used as a cover page and should include name, course and email address (in case of loss). The second page should be left blank to be used as a content's page. This page should be completed as the lab course progresses. Begin to write experimental data into the lab notebook from the third page onwards. A ball point pen is better than a fountain pen as it is less likely to smudge if water is splashed on it.
Lab notebooks need to be looked after carefully. Do not soil them with chemicals as they may transfer hazardous substances out of the laboratory. Do NOT place TLCs into your lab notebook. The chemicals can contaminate your lab notes and even damage your lab notes over time. If you want to keep record of TLC, sketch it instead.

Practical Lab-Note Design

Date

Title / chemical reaction for synthesis
(add molecular weights below formula)

include information on **team members**, if applicable

Reference (authors may be omitted and an abbreviation used)

(replace on subsequent pages with reference for previous page)

Time	Action	Observation
Example		
9:25	1.2 g compound A (optional calc. amount in mol)	
9:30	30 mL solvent B	
	dissolve A in B	yellow solution of A in B, exotherm
9:32	cool with water bath	
9:45		temperature T
9:45	record spectrum 1 (conditions)	
9:50	2 mL reagent C	measurement 1 (reference
9:55	1 mL reagent C	measurement 2
	…	

if experiment records continue, reference to page number

page #

The tabular format for lab notes above is typically suitable for almost any kind of chemical experiment, including syntheses, analyses and instrumental measurements. The time record is not always required, but enables later on a detailed analysis of the experiment. Calculated secondary data, like amounts in mol, are not important inside the lab notes, because they, unlike any primary data or observation, can be determined later. A all operations and observations shall be recorded instantly, since otherwise there is a chance for either wrong or missing records. This practically disfavours the inclusion of secondary data, unless the experiment provides a lot of time between actions/observations. Important physical data for chemicals applied, like concentration or density, shall be recorded to avoid time consuming data search after the experiment.

WRITING A REPORT

A good clear report is easy to produce, if one has a comprehensive description of the work including all relevant data on the starting materials and products as well as all the experimental details in one's laboratory notebook.

A lab report is typically structured into different sections. A typical structure includes the following:

1. Title of the experiment
2. Objectives
3. Experimental procedure
4. Results
5. Data analyses
6. Discussion of results
7. Conclusions
8. References

(4–6: these sections may be combined to 'Results and Discussion')

For certain experiments an additional section on the theory of the experiment might be necessary as well, while in other cases it is enough to include theoretical aspects inside the discussion of experimental results or the data analysis section.

Title

The title of an experiment shall be short and concise. It should reflect the scope of the entire experiment. In most cases the title of an experiment inside a laboratory manual will serve the purpose.

Objectives

The objectives of the experiment shall cover all aspects of the experiment. Ideally, every experimental operation (preparation, purification, measurement, spectra, analysis *etc*.) shall be reflected in one of the objectives. While many experiments have a dominant major objective, which is closely related to the title of the experiment, it frequently requires additional objectives to cover the entire experimental scope.

Experimental Procedure / Methodology

The experimental procedure should be described concisely with neat formula and relevant references. Do **not copy the lab manual**, but write the procedure in YOUR OWN WORDS based on your laboratory notes only. While the laboratory manual may indicate amounts as orientation value (about …), the procedure in a lab report reflects your specific experiment with well-defined measurements.

The description of the experiments should be sufficiently detailed to permit the repetition of the reaction without further consultation of the literature. This particularly requires the specification of the following:

- amounts of all compounds
  Amounts shall be provided in typical units for measurements. Accordingly, solids are quantified in g, while the amount of liquids is typically provided in mL (exceptions may apply). Moreover, in synthetic experiments the amount of reacting chemicals and catalysts are also provided in mol to enable a fast analysis of the ratio or reagents.
- concentration of solutions
  Various units may be used to specify the concentration of solutions. Typical are either molarity (mol/L or M) and normality (mol/L or N for reactive species). Besides, some

concentrations are provided as compound purity, reflecting the percentage of compound in either mass or volume, %(m/m), %(m/v), %(v/v), where the first letter specifies the measurement of the compound and the second letter the measurement of the solvent.
- time intervals
- significant observations during the experiment
- temperature and pressure, where relevant
- specification of the apparatus for the experiment
  In some cases it is recommended to provide of the experimental set up. However, a sketch for a common apparatus should be avoided, except if specific aspects are indicated as learning outcome (e.g. experimental setup of a distillation device).

In scientific reports, the use of 'I' and 'we' is discouraged. Instead, experimental procedures are typically written in passive voice. Since the procedure reflects an operation that has been conducted in the past, past tense is used. Common and detailed safety instructions are not part of an experimental procedure. However, particularly important safety aspects should be indicated.

Results and Data Analysis

This section includes your personal results. The results in your report MUST match those in your laboratory notebook. Mismatching of experiment reports and laboratory notes reflects a serious offense in science. An 'alteration' of experimental results is typically answered by complete disqualification. A scientist having found guilty of 'fabrication of experimental results' practically needs to look for another job, since the scientific community will not look at his/her results anymore. It is a one-way-exit. The severity of such an offence practically justifies a FAILUE OF A STUDENT WITH FABRICATED LAB RESULTS FOR THE ENTIRE SEMESTER, a disciplinary action upon which a University court will have to decide.

Observations, measurements and data (with units, where relevant) must match the records in your laboratory notes. Do not copy data from someone, but use your own data. If some data do not match the expectations, rather try to explain your differing result than modify your data. The **results of a lab report is a record of exactly what was observed and measured, not what is predicted to happen or be observed.** However, inside the discussion of the results, deviations of your experiment from manual instructions or common observations of class mates can be indicated and a possible explanation as well as suggestions to avoid the potentially wrong results may be provided. If data are shared with a partner or group, clearly flag the observations and data that originated from someone else.

Data should be recorded in a table, where possible. The table should be written in vertical columns using headers and units at the top of each column. Individual cells in the table should only contain a number; units only appear in headers. Please review experimental resolution constraints and provide your data only in significant figures. Besides the data, an explanation should be provided, if applicable (e.g. assignment of a spectral absorption).

Besides the presentation of primary experimental data, reports typically also require data certain analyses and the calculation of secondary data (data determined by calculation based on experimental data). Please ensure that secondary data do not exceed the precision of the experimental data they originate from. Example: If you determine the yield of a product in two (2) significant figures on a balance (e.g. 0.43 g), the calculated percentage MUST NOT exceed two (2) significant figures either. Hence, decimals for the percentage yield will be mathematical nonsense. Please be aware that calculators and computers in instruments typically do not consider matters of resolution. It is the job of the scientist to constrain the data precision to experimental limits. While final results shall be constrained to experimental limitations, the rounding of intermediary results can cause significant mistakes. Therefore, non-rounded data shall be used during the calculations.

Discussion

In the discussion section the obtained results are related to theories or hypotheses. Potential experimental mistakes can be highlighted to explain deviations of experimental results from previously reported results or theoretic predictions. In this section experimental constrains, affecting the accuracy or precision of results, should be indicated as well. Besides, suggestions for experimental improvement can be made.

An important aspect is a critical reflection of the obtained results. Typically, this involves a comparison of the obtained results with reported data and a statement on the quality of the experimental result. If data at the result section have been left without interpretation or explanation, the discussion section provides a forum to add these. Any particular difficulties, you came across during the experiment, should be reflected within the discussion as well. Moreover, the discussion provides a possibility to rationalise observations during the experiment.

The depth of theory that is included inside the discussion section depends on the experiment. While mechanistic details for chemical reactions are common for synthetic experiments, experiments focussing on measurements may require a development of a certain formula. In general, the objective of the discussion section is to demonstrate your understanding of the experiment and its theoretical background. Because of this objective, the depth of discussion must exceed the one at the laboratory manual. Particularly a copy of laboratory manual contents does not provide any information regarding your understanding. This includes not only the lab manual, but other references as well. It is crucial to write the discussion in YOUR OWN WORDS. Besides the offense of claiming other's work by copying text into a lab report, it also gives a bad impression on your understanding, if the copy is detected. (Note: There are computer tools available to detect copied texts.) A general rule indicates: Proper understanding requires the ability to explain it. In other words, if you cannot explain in your own words, you probably did not really understand it. Unfortunately, it is rather common to hide missing understanding behind sophisticated phrases. Good understanding, typically, is achieved if you are able to explain WITHOUT the use of complicated vocabulary.

Conclusion

The conclusion section relates the previously defined objectives of the experiment with your experimental results. Besides a final assessment of major results of the experiment should be performed. The section should be concise and brief. While the discussion should address all experimental results, the conclusion only refers to key aspects of the experiment.

References

The explanations and background of the experiment typically reflects work of others. To give them credit and relate your discussion to the result of others, references are provided.

References in a laboratory report should reflect different aspects of the experiment, like the experimental procedure, theoretical background or reference data that are used to evaluate the quality of the obtained results. It is good to provide more than one reference for the different aspects. However, the number of references should be balanced with the experiment. Therefore, more than three references for each aspect appears too much. In principle, you should only provide references you have reviewed, but not cite others.

While information can be obtained from a wide variety of sources, not all qualify for scientific references. Typically, references require a scientific checking. This is achieved for printed books and journals, as they are evaluated by experts in the field. Typically, internet resources from academic sites also are qualified for educational reports. However, it does not apply for other internet resources. A particularly problematic reference is Wikipedia. This reflects the concept of

the online encyclopaedia, which emphasises on participation of practically everyone. Although most contents in Wikipedia have nowadays passed a reviewing process, it is still not accepted as scientific reference. Therefore, you are encouraged to obtain information from Wikipedia, but need to refer to other sources in your lab reports.

There are various styles for references. Unless the lecturer in charge requests any specific format, any one will do. However, the formatting of references should be consistent throughout the report.

Frequent mistakes for references include missing information of the respective author. While this rarely applies for text books, the authors for internet sources are more difficult to determine and, hence frequently omitted. You can identify the author typically by looking at the page hierarchy. Look at the overview to identify the author. If this is not possible, you may refer the authorship to the institution that publishes the website (typically an Institute at a University). Owing to the fast-changing contents on the internet, online references have to labelled with the date of access.

References for journal articles typically exceed the level of a laboratory report. More practical are, typically, data collections or hand books and text books. However, journal papers may be cited as well, but not as internet links only. The minimum requirements are the author names and the journal with publication year and page number. Besides, the title of the paper should be provided to enable a quick check on the potential relevance of the paper for the experiment. An internet link can be provided as so-called DOI-reference. However, the DOI cannot replace the journal information indicated above. Only for very recent papers, which have not yet been published in print, or solely internet-based publications, the page number may be replaced by the DOI.

Plagiarism Warning!

Some of these experiments are carried out in groups of usually a pair of students. Therefore expectedly, each member of a group followed an identical procedure in the laboratory and has the same set of raw data. Members of a group are allowed to discuss the analysis of data with one another. However, preparation of the report including data analysis, interpretation and discussion must be prepared by the individual student submitting the report.
The Department does not tolerate plagiarized report!

Experiment 1 Generic

VOLUME MEASUREMENTS

1. INTRODUCTION

Volume measurements inside a chemical laboratory can apply different types of calibrated glassware. Two different calibration types, aiming for different applications, are distinguished: Volumetric flask, and measuring cylinders are calibrated on 'in'. That means that the calibration emphasises on the amount that has been placed inside the glassware. However, if the amount is supposed to be transferred subsequently into another vessel, an unknown amount of the liquid will remain inside the glassware, thereby reducing the measured amount. Pipettes and burettes, on the other hand, are calibrated on 'ex'. Here the calibration emphasises on the amount that is transferred into another container, while the amount that is removed from the original container exceeds this amount due to remaining liquid inside the calibrated glassware after the transfer.

This experiment compares two common volume measurements for chemical operations, *i.e.* pipette and measuring cylinder for different volumes. Although pipettes are more accurate in dispensing defined volumes, measuring cylinders are still frequently applied for volume measurements. The experiment aims to provide the experimenter with information regarding potential mistakes in volume measurements using a measuring cylinder in order to enable a justified decisions on when a measuring cylinder may be used to simplify the volume measurements.

2. LEARNING OUTCOME

1. Recognition of different types of volume calibrations (in vs. out) and the impact it has on measurements
2. Safe and clean operation in the chemical laboratory

3. METHODOLOGY

3.1. Materials

Glassware
Volumetric flask (25 mL)
Graduated pipette (10 mL)
Measuring cylinder (10 mL)
Beakers (400 mL & 50 mL)
Pasteur pipettes
Conical flask with stopper (100 mL)

Chemicals
Non-specified calibration liquid (100 mL)
(moderately basic aqueous solution)

Instruments
Analytical balance
Pipetting aids

3.2. Operation of calibrated glassware and the calibration fluid

- Calibrated glassware must not be dried high temperature to avoid temperature induced alteration of the volume
- Drying of calibrated glassware is best achieved by rinsing with a low boiling solvent. The solvent must be miscible with the measuring fluid applied. For practical purpose, the glassware should be rinsed with water and subsequently with acetone or ethanol.

- For proper operation of calibrated glassware, the meniscus of the measured liquid must be observed at eye level. This applies for volumetric flasks, pipettes and measuring cylinders.
- Pipettes are operated in vertical orientation only. Unintended flow-out is prevented by firm closing of the upper opening and avoiding fast movements.
- Pipettes must be operated without pressure. The liquid is left to flow out at vertical position. For liquids with low viscosity, it is typically sufficient to wait for about 5 seconds once the main volume has flown off. It is recommended to touch the vessel into which the liquid should be transferred with the tip of the pipette to avoid incomplete emptying of the calibrated amount.
- The calibration fluid is moderately basic. It is not particularly hazardous, but prolonged exposure of the skin may cause chemical burns. Unfortunately, skin sensors are detecting basic reagents much less efficiently than acids. To avoid harm, ensure that your hands remain dry and wash with water in case of contact. There is no reason to worry about health effects in case of a short time skin exposure, as long as the compound is timely washed away.
- While the impact of the calibrating fluid on skin is minor, exposure of the eye requires immediate action to avoid potential permanent damage.
- **The calibration fluid should not be disposed, but reused for the entire experiment**.

3.3. Experimental Procedure

3.3.1. Density of the calibration fluid

1. Measure and record the accurate mass of the volumetric flask
2. Carefully fill the volumetric flask with calibration fluid. Use a Pasteur pipette for the transfer. Avoid touching the glass wall above the calibration mark, because remaining water droplets increase the overall volume above the calibration.
3. Measure the mass of the filled volumetric flask on the analytical balance.
4. Determine the mass of the liquid and calculate the density.
5. Repeat the measurements once with new filling of the volumetric flask.
   Notes: It is not necessary to remove the entire volume before adding more calibration fluid. Instead, it is sufficient to remove 1-2 Pasteur pipettes to ensure the liquid level is below the calibration mark.
6. If the data for the two measurements show significant deviation, repeat the measurement a third time.
7. Calculate the average density and its standard deviation.

3.3.2. Evaluation of measurement accuracy for a pipette

1. Measure and record the accurate mass of a small beaker
2. Pipette 1 mL of the calibration fluid into the beaker
3. Measure the mass of the beaker with the calibration fluid.
4. Calculate the volume of the transferred calibration fluid based on the density from 3.3.1.
5. Repeat the measurement by adding another 1 mL into the beaker. There is no need to dry the beaker between measurements, if the mass of the previous measurements is taken as basis m_0.
6. If the results for the measurements differ significantly, perform a third measurement.

7. Repeat the process with 2 mL, 5 mL and 10 mL measurements

3.3.3. Evaluation of measurement accuracy for a measuring cylinder

1. Measure and record the mass of a measuring cylinder
2. Measure 1 mL calibration fluid in the measuring cylinder
3. Measure the mass of the filled measuring cylinder
4. Calculate the volume based on the density from 3.3.1.
5. Transfer the content of the measuring cylinder into a bleaker
6. Measure the mass of the re-emptied measuring cylinder
7. Calculate the transferred volume based on the density from 3.3.1
8. Repeat the measurements. If the results for the measurements differ significantly, perform a third measurement.

9. Repeat the process with 2 mL, 5 mL and 10 mL measurements

3.3.4. Data analysis and problem solving

1. Compare the volume measurements for pipette and measuring cylinder on both measuring modes (in and ex)
2. Evaluate constraints for volumetric measurements using a measuring cylinder by comparing relative errors as a function of the volume

Problem:

1. Propose a procedure for a reasonable accurate measurement of 1 mL using a measuring cylinder and a Pasteur pipette
2. Evaluate the accuracy of your proposal

3.3.5. Visualisation of calibration fluid

1. Fill about 30-50 mL of the calibration fluid into a conical flask
2. Stopper the flask and shake intensely.
3. Record your observations
4. Repeat the process.

Experiment A1 Inorganic Chemistry

CALIBRATION OF A 25-ML PIPETTE

1. INTRODUCTION

The graduation mark on a pipette being usually made at 20 °C (whereas room temperature is much higher than this), the volume of the pipette must be calibrated before any volumetric analysis is carried out. Otherwise, the error in the graduation mark may exceed the error allowed in a measurement. A pipette is designed to deliver only one fixed volume of a liquid and it is calibrated for this volume only. Accuracy to two decimal places in mL is generally possible.

The pipette is calibrated by weighing distilled water in it at room temperature, and then calculating the volume from the weight of water in air. A correction for the buoyancy of air is included. The formula allows for the determination of the weight in vacuum, W after correction, where W_a is the weight in air. The volume is calculated from the weight and the density.

$$W = Wa + 1.06\ Wa/1000$$

Table 1: Density of water at various temperatures

Temperature / (°C)	**Density of water / (g mL^{-1})**
25	0.99705
26	0.99681
27	0.99654
28	0.99626
29	0.99597
30	0.99567
31	0.99537
32	0.99503
33	0.99473
34	0.99440

2. LEARNING OUTCOME

1. Able to handling a pipette, including -
   selecting the appropriate pipette for a given task, and using the selected pipette in the proper manner
2. Able to understand the accuracy of pipettes in the laboratory by relating pipetted volumes to mass measurements

How to use a pipette

1. Rinse a 25-mL pipette with two or three small volumes of distilled water, and then with a complete volume of distilled water.
2. Do not immerse the tip of the pipette too deep into the water. The tip should not be above the water level so as to avoid any mishap during the suction of the water into the pipette via the pipette filler or suction bulb.
3. Draw the solution into the pipette until it reaches a level above the graduated mark.
4. Take the pipette away and wipe its tip with a piece of tissue paper.
5. Hold the pipette upright, and slowly release the pressure of the suction bulb or finger untilthe meniscus level is on the graduated mark. Touch the tip of the pipette on the dry side of the container until the drop at the end has been drained off.
6. Drain the contents to another container. Hold the pipette upright until the solution has been completely drained. Allow the liquid to flow out and then wait for 15 seconds after the last drop has emerged from the tip.
7. During the delivery, ensure that the pipette tip is always above the level of the solution inthe receiver.
8. Finally, touch the tip of the pipette on the side of the container until the meniscus level in the pipette tip does not fall any farther.
9. **Do not blow or force out whatever remains in the tip.**

3. METHODOLOGY

1. Take the weight of the weighing bottle and its cover. (The volume of the weighing bottle must exceed 30 mL).
2. Transfer the distilled water in a 25-mL pipette into the weighing bottle.
3. Weigh the bottle, and repeat the weighing until two consecutive readings do not differ by more than 0.005 g.
4. Record the room temperature.
5. Calculate the volume of the pipette.

4. QUESTIONS

1. Does the pipette empty from full volume to zero or from zero to full volume?
2. Is the pipette designed to be emptied by gravity with the tip in contact with the vessel or to be expelled by blowing out with pipette filler? Explain.

Experiment A2 Inorganic Chemistry

VOLUMETRIC ANALYSIS

1. INTRODUCTION

Volumetric analysis is a quantitative analytical process whereby volumes are measured. A volume of reagent known as the standard solution of known concentration is chemically reacted witha solution of unknown concentration in order to determine the concentration of the unknown.

The solution that is used as a standard is called the **titrant**, which is delivered from a burette and is added until the reaction is complete. Titrations are based on acid-base neutralization, oxidation-reduction, and complex formation or precipitation reactions. The end-point of the titration,i.e., when the **analyte** has been completely reacted, is noted by observing the change of color of an**indicator**. The titrant is standardized against a **primary standard**, which is a pure reagent. In a **direct titration**, the titrant is added to analyte until the end point is observed whereas in a **back titration**, a known excess of a standard reagent is added to the analyte. A second standard reagentis then used to titrate the excess of the first reagent. Back titrations are used when the end point of the back titration is clearer than the end point of the direct titration or when excess of the first reagent is required. The experiment starts with preparation of potassium permanganate solution as primarystandard. The equation for the standardization experiment is given in the ionic form as

$$5\ C_2O_4^{2-} + 2\ MnO_4^- + 16\ H^+ \longrightarrow 10\ CO_2 + 2\ Mn^{2+} + 8\ H_2O$$

The potassium permanganate solution now becomes the “standard solution” after it has been standardized against sodium oxalate, i.e., the molarity is known exactly.

This solution is then used to determine the quantity of nitrite ions in a back titration in the first part. A known volume of potassium permanganate solution is added to the nitrite solution under acid conditions. Because there is an excess, the excess is titrated against iron(II) ammonium sulfate solution. The back titration is explained by the equations

$$5\ NO_2^- + 2\ MnO_4^- + 6\ H^+ \longrightarrow 5\ NO_3^- + 2\ Mn^{2+} + 3\ H_2O$$

$$2\ MnO_4^- + 10\ Fe^{2+} + 16\ H^+ \longrightarrow 10\ Fe^{3+} + 2\ Mn^{2+} + 8\ H_2O$$

The second set of titration experiments involve the uses of sodium thiosulfate pentahydrate (formula weight of $Na_2S_2O_3 \cdot 5H_2O$ is 248.21), which is similarly standardized against the iodine in potassium iodate. Potassium iodate reacts with potassium iodide to release iodine. The reactionsare represented by the ionic equations

$$IO_3^- + 5\,I^- + 6\,H^+ \longrightarrow 3\,I_2 + 3\,H_2O$$

$$2\,S_2O_3^{2-} + I_2 \longrightarrow S_2O_6^{2-} + 2\,I^-$$

The standardized sodium thiosulfate solution is used to determine the amount of chlorine in *Chlorox*:

$$ClO^- + 2\,I^- + 2\,H^+ \longrightarrow I_2 + Cl^- + 2\,H_2O$$

2. LEARNING OUTCOME

1. Able to understand the concept of volumetric analysis
2. Capable of using the standardized solution to determine the unknown solution concentration via titration

3. METHODOLOGY

Part A

1. Weigh about 0.3 g sodium oxalate (Formula weight = 133.99 g/mol) and transfer it into a 100 mL volumetric flask. Add 20 mL distilled water followed by 50 mL dilute sulfuric acid (2.5 M), and then top up to the calibration mark with distilled water.
2. Weigh between 0.8 to 1.0 g of potassium permanganate and transfer it in a 250-mL volumetric flask. Add 50 mL distilled water to the flask and shake it to dissolve the solid, and then top up to the calibration mark with distilled water. Place some of the solution in the burette.
3. Pipette 25 mL of the sodium oxalate solution into a 250-mL conical flask. Heat the solution to 80 °C, and titrate the hot solution with the potassium permanganate solution. Repeat two more times.
4. Calculate the molarity of the potassium permanganate solution.
5. Transfer 10 mL of the potassium permanganate solution into a 400-mL beaker and add dilute sulfuric acid (1:5) to the 100-mL mark.
6. Warm the beaker to 40 °C, and then add 8 mL of the nitrite solution from a burette. The tip of the burette must be below the surface of the solution. Rinse the tip of burette with distilled water. Cool the solution to room temperature.
7. Place the standard iron(II) ammonium sulfate solution in a burette and titrate it against your nitrite solution. Repeat two more times.
8. Calculate the molarity of the nitrite solution.

Part B

1. Fill up a burette with sodium thiosulfate solution.
2. Weigh about 0.2 g potassium iodate and dissolve it with distilled water in a 100 mL volumetric flask.
3. Pipette 25 mL of the potassium iodate solution into a 250 mL conical flask. Add 1 g of potassium iodide followed by 5 mL of sulfuric acid (1 M).
4. Titrate the liberated iodine with the thiosulphate solution immediately. When the color of the mixture is pale yellow, add about 100 mL distilled water followed by 1 to 2 mL of freshly prepared starch solution.
5. Continue the titration until the solution is colorless.
6. Repeat the titration one more time and calculate the average molarity of the thiosulphate solution.
7. Pipette 10 mL of the chlorox solution into a conical flask. Add 40 mL of distilled water, 1 g of potassium iodide and 10 mL of sulfuric acid (1 M).
8. Titrate the liberated iodine with standard sodium thiosulphate solution.
9. Repeat the titration, and calculate the average concentration of hypochlorite in the Chlorox (expressed in % Cl_2).

4. QUESTIONS

1. What is an acid base indicator and how it is been used for?
2. How to determine the equivalence point of a titration?

Experiment A3 Inorganic Chemistry

GRAVIMETRIC DETERMINATION AND IR-CHARACTERIZATION OF NICKEL COMPLEX

1. INTRODUCTION

The quantity of nickel ions in a salt is determined by precipitating the ions with dimethylglyoxime in an ammoniacal solution. The product is bis(dimethylglyoximato)nickel(II), chemical formula $Ni(C_4H_7O_2N_2)_2$. It is an insoluble red precipitate. The precipitate is collected and dried, and is then weighed. Dimethylglyoxime behaves as a monobasic acid in the reaction:

$$Ni^{2+} + 2\ C_4H_8O_2N_2 \longrightarrow Ni(C_4H_7O_2N_2)_{2.} + 2\ H^+$$

Precipitation is effected by the addition of dilute ammonium hydroxide to a hot solution of the nickel salt and dimethylglyoxime.

The prepared compound will be characterized by Fourier Transform Infrared Spectroscopy, also known as FTIR Analysis or FTIR Spectroscopy, which is an analytical technique used to characterize organic, polymeric, and in some cases, inorganic materials. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties. It is used by chemists to determine functional groups in molecules. FTIR Spectroscopy measures the vibrations of atoms, and based on this it is possible to determine the functional groups. Generally, stronger bonds and light atoms will vibrate at a high stretching frequency (wavenumber).

2. LEARNING OUTCOME

1. Able to understand the concept of gravimetric analysis
2. Able to derive the mass of nickel calculated from the mass of precipitate

3. METHOLOLOGY

Part A

1. Pipette 25 mL of a nickel(II) sulfate solution into each of the two 400-mL beakers.
2. Add 5 mL of dilute hydrochloric acid (2 M) and dilute the solution to 200 mL with distilled water. Heat the solution to 60 – 80 °C.
3. Add about 20 mL of dimethylglyoxime solution (1%) to the hot solution, followed by dropwise addition, with stirring, of ammonium hydroxide until precipitation occurs and the solution is slightly basic (about 2 mL of excess ammonium hydroxide). Test for completeness of precipitation by adding more dimethylglyoxime solution (1%).
4. Warm the precipitate for 30 minutes. Cool to room temperature and filter through a weighed No. 4 sintered glass crucible previously dried at 110°C.
5. Wash the precipitate with cold distilled water until it is free from any chloride (test with silver nitrate solution) and dry overnight.
6. Cool the crucible in a desiccator and weigh it to constant weight.
7. From the weight of the precipitate and a knowledge of the theoretical quantity of nickel in bis(dimethylglyoximato)nickel(II), calculate the concentration (in g L^{-1}) of nickel(II) ions in the solution.

Notes:

1. Carry out the analysis twice.
2. After the experiment, clean the crucibles by washing with hydrochloric acid followed by distilled water.
3. Keep the precipitate for IR analysis.

Part B: Solid FTIR sample

Preparation Method 1: Nujol mull

Simplest method to obtain IR spectra of solids are Nujol (mineral oil) mulls between KBr plates.

Good results are obtained by this method only if the average particle size of the solid is somewhat less than the wavelength of light the particles are to transmit. Samples should therefore be grounded in a mortar to reduce the average particle size to 1 to 2 microns.

1. About 5 to 10 mg of finely ground sample are then placed onto the face of a KBr plate, a small drop of mineral oil is added and the second window is placed on top.
2. With a gentle circular and back-and-forth rubbing motion of the two windows, evenly distribute the mixture between the plates. The mixture should appear slightly translucent, with no bubbles, when properly prepared.
3. Place the sandwiched plates in the spectrometer and obtain a spectrum. Ideally, the strongest band should have a transmission of 0 to 10% and should not be totally absorbing for more than 20 cm^{-1}.

Attention:

If the bands are distorted (show fronting or tailing) the particle size is too large and some radiation incident on the mull has been scattered out of the sample beam. If a better spectrumis required, reduce the particle size further.

The KBr plates must be thoroughly cleaned after this procedure to prevent contamination of future samples. Wipe the windows with a tissue, then wash several times with methylene chloride, then ethanol. Use the polishing kit in the lab to polish the window surface. Wear glovesto prevent fogging. The cleaned surface should be clear and free from scratches.

Notes:

Remember that Nujol by itself shows a characteristic spectrum!

Preparation Method 2: KBr pellet/disk

Sample/KBr ratio

The concentration of the sample in KBr should be in the range of 0.2% to 1%. The pellet is much thicker than a liquid film, hence a lower concentration in the sample is required (Beer's Law). Too high a concentration usually causes difficulties in obtaining clear pellets. The IR beam is absorbed completely, or scattered from the sample which results in very noisy spectrum.

Although a homogeneous mixture will give the best results, excessive grinding of the potassium bromide is not required. The finely powdered potassium bromide will absorb more humidity (it is hygroscopic) from the air and therefore lead to an increased background in certain ranges. Make sure to work fast. Transfer some KBr out of the oven. Add about 1 to 2%of your sample, mix and grind to a fine powder in mortal with pestle. For very hard samples, add the sample first, grind, add KBr and then grind again. The sample must be very finely ground as in the Nujol mulling technique to reduce scattering losses and absorption band distortions.

Place just enough sample (fine powder) to cover the bottom of the pellet die. Place in press and press at 5000-10000 psi. Check pellet press brochure for details. Carefully remove the pressed sample from die and place in the FTIR sample holder. The pressed disc should be nearly clear if properly made. If it is translucent, regrind and repress. Insert into the IR sampleholder and attach with scotch tape. Run the spectrum.

After use, the mortar and pistil should be cleaned with acetone and double distilled water, andbe put back on top of the oven for drying.

4. QUESTIONS

1. Draw the structural formula of bis(dimethylglyoximato)nickel(II) and describe the type of hybridization involved.
2. Explain why the temperature of the solution must be maintained at 60 – 80 °C during the precipitation process.

Experiment B1 Physical Chemistry

ACID BASE TITRATION

1. INTRODUCTION

The process of adding acid to a base (or vice versa) to produce a salt and water is called **neutralization**. In the neutralization of hydrochloric acid with sodium hydroxide, the reaction that occurs is:

$$HCl + NaOH \rightarrow H_2O + NaCl$$

When an acid and a base are present in stoichiometric amount, for example one mole of hydrochloric acid is added to one mole of sodium hydroxide as in the above neutralization process, this means that the equivalent point has been reached in the acid-base system. Suitable indicators (refer to table 1) can be used to monitor the end-point (the point at which an indicator changes colour) of the titration.

Table 1: Common indicators and their colours

Indicator	**Colour in acid**	**End-point colour**	**Colour in alkali**	**pK_{ind}**	**For titration of**
Litmus	RED	PURPLE	BLUE	6.5	strong acid and strong base
Methyl orange	RED	ORANGE	YELLOW	3.1-4.4	strong acid and strong base or strong acid and weak base
Phenolphthalein	COLOURLESS	PALE PINK	PINK	9.3	weak acid and strong base
Bromotymol blue	YELLOW	GREEN	BLUE	6.2-7.6	strong acid and strong base

In the above example, pH of the hydrochloric acid will change with increasing amount of sodium hydroxide added. At the end-point the solution will become neutral with pH of 7. This is only true for strong acid-strong base titration. For strong acid-weak base, weak acid-strong base and weak acid-weak base titration, the salt that is formed may undergo hydrolysis and this may cause the pH not to be equal to 7 at the end-point. Graph of pH versus volume of base that is added to the acid of constant volume or otherwise is called the **pH titration curve**.

2. OBJECTIVE

To determine pH curve for titration of strong acid-strong base and weak acid-strong base

3. METHOLOLOGY

3.1. Materials

Chemicals: Sodium carbonate, hydrochloric acid, sodium hydroxide and acetic acid.
Indicators: Methyl orange, phenolphthalein and bromothymol blue.

3.2 Experimental Procedure

3.2.1. Standardization of acidic and basic solution

3.2.1.1. Preparation of Standard Sodium Carbonate Solution

1. Weigh accurately about 1.3 g of the primary standard, anhydrous sodium carbonate in a weighing bottle.
2. Transfer the materials quantitatively into a 250 ml volumetric flask; Then add about 80 mL distilled water and shake the flask until all the sodium carbonate dissolve. Add distilled water to the volumetric mark and then shake the flask well.
3. Calculate the concentration of the standard sodium carbonate solution in unit of mol L^{-1}. (Report the concentration to four significant figures from the decimal point).

3.2.1.2. Standardization of hydrochloric acid

1. Pipette 25 mL of standard sodium carbonate solution into a 250 mL conical flask, add 2 drops of methyl orange.
2. Titrate with the given hydrochloric acid until the solution starts becoming red.
3. Repeat the titration, this time immediately add the acid until it is short of the titer value obtained in (ii) by 0.5 mL. Then titrate slowly until the end-point is reached.
4. Repeat the titration until the titer value does not differ by more than 0.05 mL from the previous titration.
5. Calculate the concentration of the hydrochloric acid given. (Report your result to four significant figures from the decimal point).

3.2.1.3. Method of calculating the concentration of hydrochloric acid solution

Consider the concentration of the standard sodium carbonate solution is *c* mol dm^{-3}, and *v* mL of hydrochloric acid is required to neutralize 25 mL of this standard sodium carbonate solution. The reaction that occurs is:

$$2\ HCl + Na_2CO_3 \rightarrow 2\ NaCl + H_2O + CO_2$$

From the above reaction it is clear that the neutralization of 1 mole of sodium carbonate requires 2 moles of hydrochloric acid.

25 mL of the carbonate solution used contains $\frac{25c}{1000}$ moles of sodium carbonate. Therefore the neutralization requires $2 \times \frac{25c}{1000}$ moles hydrochloric acid. Hence, the concentration of the hydrochloric acid solution is

$$\frac{2 \times 25c}{1000} \times \frac{1000}{v} = 2 \times \frac{25c}{v} \text{ mol dm}^{-3} \text{ (or M)}$$

3.2.1.4. Standardization of sodium hydroxide solution

1. Pipette 10 mL of hydrochloric acid that has been standardized into a 250 mL conical flask.
2. Add 2 drops of methyl orange.
3. Titrate with the given sodium hydroxide solution.
4. Repeat the titration; this time simply add the sodium hydroxide until it 0.5 mL less than the titer value obtained in step (ii). Then titrate slowly until the end-point is reached.
5. Repeat the titration until the titer value does not differ by more than 0.05 mL.
6. Repeat steps 1 to 4 using phenolphthalein as the indicator.
7. Calculate the concentration of the sodium hydroxide solution obtained from the titrations using methyl orange and phenolphthalein as indicators.

3.2.1.5. Standardization of acetic acid solution

1. Pipette 10 mL of acetic acid into a 250 mL conical flask and add 2 drops of phenolphthalein indicator.
2. Titrate with sodium hydroxide solution that has been previously standardized.
3. Repeat the titration; this time the sodium hydroxide is quickly added until it is 0.5 mL short of the titer value obtained in step (ii), then titrate slowly until the end-point is reached.
4. Repeat the titration until the titer value obtained does not differ by more than 0.05 mL.
5. Repeat steps 1 to 4 but using bromothymol blue as the indicator.
6. Calculate the concentration of acetic acid from the titration using phenolphthalein and bromothymol blue as the indicators.

3.2.2. pH curve for strong acid-strong base titration

1. Prepare a solution of sodium hydroxide that is diluted 10 times by pipetting 25 mL of sodium hydroxide into a 250 mL volumetric flask and adding distilled water to the mark.
2. Prepare two burettes, one is filled with the standard sodium hydroxide solution and the other is filled with sodium hydroxide that has been diluted 10 times.

   (You are required to share your burette with your neighbour in this part)

   In order to fill the burette with diluted sodium hydroxide, empty the standard sodium hydroxide from the burette and rinse the burette with distilled water several times. Finally, rinse the burette with 3 parts (each part 10 mL) of dilute sodium hydroxide, then the burette is filled with the diluted sodium hydroxide solution.

3. Pipette 10 mL of standard hydrochloric acid into a 50 mL beaker.
4. Titrate this acid with standard sodium hydroxide until the pH is 2.8 or 2.9 (you may use a calibrated pH meter to determine the pH of the solution). Record the volume of the required standard sodium hydroxide solution.
5. Continue the titration with dilute sodium hydroxide in order to get pH 4, 5, 6, 7, 8, 9 and 10.
6. Continue the titration with standard sodium hydroxide in order to get pH 11 and 12.
7. Plot a graph of pH of solution against the volume of standard sodium hydroxide used.

   (Attention: 10 mL of dilute sodium hydroxide equals 1 mL of standard sodium hydroxide).

3.2.3. pH curve for weak acid-strong base titration

1. Pipette 10 mL of standard acetic acid into a 100 mL conical flask.
2. Titrate the acetic acid with standard sodium hydroxide in order to get pH 3, 4, 5 and 6.
3. Continue the titration with dilute sodium hydroxide in order to get pH 7, 8, 9 and 10.
4. Continue the titration with standard sodium hydroxide in order to get pH 11 and 12.
5. Plot a graph of pH of solution against the volume of standard sodium hydroxide used.

4. QUESTIONS

1. Calculate the theoretical pH of the solution at the following instances when 0.00, 5.00, 9.00, 9.50, 9.80, 9.92, 10.00, 10.02, 10.20 and 15.00 mL of standard sodium hydroxide solution are added to 10 mL hydrochloric acid. Plot the pH curve obtained by theoretical calculations together with the pH curve obtained in **Part 3.2.2.** of this experiment. Compare the two pH curves and give your comments on the differences that are observed.

2. Calculate the theoretical pH of the solution at the following instances when 0.00, 5.00, 9.00, 9.50, 9.80, 9.92, 10.00, 10.02, 10.20 and 15.00 mL of standard sodium hydroxide solution are added to 10 mL acetic acid. Assume K_a (acetic acid) to be 1×10^{-5}. Plot the pH curve obtained by theoretical calculation together with the pH curve obtained in **Part 3.2.3.** of this experiment. Compare the two pH curves and give your comments on the differences that are observed.

3. The colour changes corresponding to the pH intervals for the three indicators are given in the following table:-

Indicators	Colour change	pH Interval
Methyl orange	Red to orange	3 - 5
Bromothymol blue	Yellow to blue	6 - 8
Phenolphthalein	Colourless to pink	8 - 10

Comment on the end-point reached by using different indicators in **Part 3.2.1.4.** and **Part 3.2.1.5**. Confirm the suitability of each indicator by referring to the pH curves determined in this experiment.

5. REFERENCES

1. Mahan, B.H. (1987). University Chemistry, 4th edn. Addison-Wesley.
2. http://www.ausetute.com.au/indicata.html
3. Vogel, Athur T. (1994). Vogel's Textbook of Quantitative Chemical Analysis. Longman.

nb: Please quote experimental error estimates for all your data presented.

Experiment B2 Physical Chemistry

pH MEASUREMENT

1. INTRODUCTION

Water can undergo self-ionization or auto-ionization in which two molecules of water react with each other to form ions in the following manner:

$$H_2O + H_2O \rightleftharpoons H_3O^+ + OH^-$$

This is a very important equilibrium because it is present not only in pure water but also in all aqueous solution. The degree of ionization of water can be characterized by an **equilibrium constant,** K, given by

$$K = \frac{[H_3O^+][OH]}{[H_2O][H_2O]}$$

The molar concentration of water, which appears in the denominator of this expression, is very nearly constant (≈ 55.6 M) in both pure water and dilute aqueous solutions. Therefore, $[H_2O]^2$ can be included with the equilibrium constant K to produce a new combined constant, K_w

$$K_w = [H_3O^+]\ [OH^-]$$

where K_w is called the **ion product constant** for water, or the **ionization/dissociation constant** of water. This means that the product of the concentration of H_3O^+ and the OH^- ions in any aqueous solutions is a constant. In fact, the value of K_w depends only on temperature. At 25 °C, $K_w = 1.0 \times 10^{-14}$.

A solution is said to be:-

neutral	if $[H_3O^+] = [OH^-]$	$= 10^{-7}$
acidic	if $[H_3O^+]$	$> 10^{-7}$
basic	if $[H_3O^+]$	$< 10^{-7}$

The pH-concept

H_3O^+ and OH^- ions enter into many equilibra in addition to the dissociation of water, so it is frequently necessary to specify their concentrations in aqueous solutions. These concentrations may range from relatively high values to very small ones (for example, from 10 M to 10^{-14} M), and a logarithmic notation has been devised to simplify the expression of these quantities. In general, for some quantity X, the quantity pX is defined as

$$pX = \log_{10} \frac{1}{X} = - \log_{10} X$$

Thus, the value of the concentration of H_3O^+ ion in a solution can be expressed in terms of its **pH**, which is defined as

$$pH = \log_{10} \frac{1}{[H_3O^+]} = - \log_{10} [H_3O^+]$$

Hence, for a solution with $[H_3O^+] = 10^{-4}$ M, the pH of the solution is said to be 4. Following the same approach for the hydroxide ion concentration, we can define the pOH of a solution as

$$pOH = - \log_{10} [OH^-]$$

Ionization Constant of Weak Acids

The following is the **ionization** which occurs in an aqueous solution containing a weak acid HA:

$$HA + H_2O \rightleftharpoons H_3O^+ + A^-$$

The equilibrium constant or the **ionization constant**, K_a , for the weak acid HA is

$$K_a = \frac{[H_3O^+][A^-]}{[HA]}$$

In this expression we have again neglected the molar concentration of water which is assumed to be constant. Because the values of K_a cover a wide range, it is more appropriate to substitute K_a with **pK_a** which is defined as

$$pK_a = - \log_{10} K_a$$

Ionization Constant of Weak Bases

The ionization which occurs in an aqueous solution of a weak base B is

$$B + H_2O \rightleftharpoons BH^+ + OH^-$$

and the ionization constant, K_b given by

$$K_b = \frac{[BH^+][OH^-]}{[B]}$$

and

$$pK_b = - \log_{10} K_b$$

It can be shown that for any acid-base conjugate pair,

$$K_a K_b = K_w$$

pH Meter

A pH meter consists of a pair of electrodes; one sensitive to the H_3O^+ concentration, usually a glass electrode, and the other a reference electrode such as the calomel electrode. The manual for the pH meter gives a full description on its usage and also contains other useful information. Users are required to read this manual carefully before proceeding to use this instrument. The pH-meter MUST be **calibrated** using standard buffer solutions before it can be used to measure the pH of any solutions.

2. OBJECTIVE

To determine the *ionization constant* of various acids

3. METHOLOLOGY

3.1. Materials

Two standard buffer solutions, phenolphthalein indicator, 0.1 M acetic acid solution, propanoic acid, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, and sodium hydroxide.

3.2. Experimental Procedure

Prepare 50% neutralized acid solutions for each of the given acids in the following manner:

1. Titrate 25 ml of the given acid with 0.1 M sodium hydroxide solution using phenolphthalein as indicator.
2. Pipette another 25 ml of the same acid into the solution which has been neutralized in step 1.

(Steps 1 and 2 must be carried out carefully to ensure the success of this experiment.)

3. Calibrate the pH meter using the two standard buffer solutions with pH values of 4.0 and 7.0 (The calibration only needs to be carried out only once.)
4. Measure the pH of the 50% neutralized acid solution prepared in step 1.
5. Record the temperature of the solution.
6. Repeat step 1 to step 4 (except step 2) for all the acid solutions to be studied in this experiment.

4. REFERENCES

1. Mattock, George, (1961). pH measurement and titration. Heywood.
2. McMillan, Gregory K., (1994). pH measurement and control. Instrument Society of America.

nb: Please quote experimental error estimates for all your data presented.

Experiment B3 Physical Chemistry

MOLAR CONDUCTIVITIES OF AQUEOUS ELECTROLYTES

1. INTRODUCTION

1.1. Electrical conductivity in solutions

An electric current in solution is the result of the net movement of free ions in a specific direction. The current may be determined by measuring the resistance R between two similar inert electrodes immersed in the solution, as in the figure below where the oval region represents the solution; A represents the electrode area and l is the normal distance between the electrode planes. In actual practice an A.C. current with a low frequency of the order of approximately 1000 Hertz is used (to prevent electrolysis) in the measurement, and the components representing the resistance R in the complex impedance Z for the circuit is determined. We will always refer to this component (the real portion of the complex impedance) for what follows. The resistance is also dependent on the frequency (Debye-Falkenhagen effect). The theory and measurement here focus on low frequency measurements, where the Onsager equation is meaningful. The fully automated measuring apparatus has been configured for low frequency measurement in accordance with the theory of electrolytes.

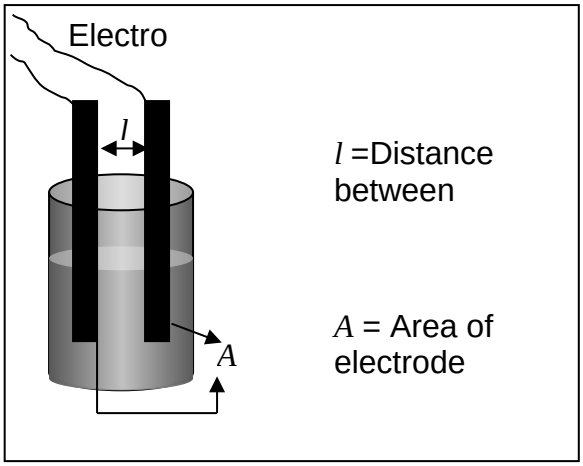

Figure of conductivity circuit

According to Ohm's law, the resistance R (unit Ohm, symbol Ω) for the above circuit is given by

$$R = \rho \frac{l}{A} \qquad (1)$$

where ρ is the resistivity of the solution. The cell constant refers to $\frac{l}{A}$ but there is no need to refer to this quantity here. The electrical conductivity of the solution κ is defined as $\kappa = \frac{1}{\rho}$, where the current density, j, is given by $j = \kappa E$. The conductance of the solution, L, is defined as $L = \frac{1}{R}$ and has units Siemen, where $\frac{1}{\Omega}$ = 1 S (1 Siemen). On the other hand, from (1), κ has the units (S.I) [S M^{-1}]. This is the quantity that the conductivity apparatus measures. Normally, (such as the ones

currently used in the laboratory), the units of $\kappa = \frac{1}{\rho}$ is $mS\ cm^{-1} \equiv 10^{-1}\ S\ cm^{-1} \equiv 10^{-1}\ S\ m^{-1}$. Because the conductivity is dependent on the concentration of the electrolytes, Kohlrausch defined the molar conductivity, Λ_m, so that comparisons can be made at any concentration as follows

$$\Lambda_m = \frac{\kappa}{c} = \frac{\Lambda}{c}; \kappa = \frac{l}{RA} \qquad (2)$$

where c is the concentration of the electrolyte concerned. Clearly, Λ_m has S.I. units $S\ m^2\ mol^{-1}$. Normally Λ_m drops in value as the concentration of the electrolyte increases. This observation can be explained by the Debye-Huckel theory where an ionic atmosphere of opposite charge to the ion is formed which retards the motion of the ion by inducing a force in the opposite direction to the motion of the ion. This dynamic asymmetry effect (also a relaxation effect) reduces the value of the molar conductivity compared to that when $c \rightarrow 0 (\Lambda_0)$ by a factor of form $BA\Lambda_o\sqrt{c}$; there is another effect called the "electrophoretic effect" which refers to the retardation due to the movement of solvent molecules dragged by the ion and the ionic atmosphere and this effect has the form $A\sqrt{c}$ for univalent ions. Thus, the total effect due to the two separate effects has the form

$$\Lambda = \Lambda_o + B\Lambda_o\sqrt{c} + A\sqrt{c} = \Lambda_o - \beta c^{1/2} \qquad (3)$$

for any one solvent at any one temperature for a univalent system. If the conductivity of the solution is $\Lambda\ (c.g.s) = x\ mS\ cm^{-1}$ as given in most readouts, and the concentration is c mol dm^{-3} (as is the case is most machine readouts), then the molar conductivity in S.I. units will be $\Lambda = \frac{x}{c} x10^{-4}\ S\ m^2\ mol^{-1}$. We recommend that you plot the values using c.g.s units. Plotting Λ vs $\sqrt{c}$ linearizes the curve and determines Λ_o as the intercept and $-\beta$ as the gradient for "strong" electrolytes that are essentially fully dissociated at all concentrations. This plot does not work well for "weak" electrolytes because the steep gradient in the vicinity of low concentrations does not yield accurate values of the intercept. This is what will be discovered in the course of the experimentation according to the procedure below. The steep slopes are due to the constant dissociation of the electrolyte. For such cases, the following law is required.

1.2. Kohlrausch's Law

This law states that the molar conductivity at infinite dilution Λ_o for an ionic salt is the sum of the molar conductivities at infinite dilution of its separate ions λ_o^+, λ_o^-, the signs referring to charge, i.e.,

$$\Lambda_o = \lambda_o^+ + \lambda_o^- \ . \qquad (4)$$

Note that at infinite dilution, even weak electrolytes are fully dissociated and cannot be distinguished from strong electrolytes.

1.3. Dilution Effects of Weak Electrolytes

For any one temperature and concentration, the degree of dissociation α implies the following for a weak electrolyte such as acetic acid HAc, where the weak acid has the following equilibrium relative to its total concentration, c.

Reaction:	*HAc(aq)*	+	*H_2O*	$\rightleftharpoons$	*H^+ (aq)*	+	*Ac (aq)*	(5)
Conc.:	*(1-α)c*				*αc*		*αc*	

From the above equilibrium (5), ignoring activity coefficients, the acid dissociation constant K_a is given by

$$K_a = \frac{[H^+][Ac^-]}{[HAc]} = \frac{\alpha^2 c}{1-a} \qquad (6)$$

where, according to Arrhenius, α is given as

$$\alpha = \frac{\Lambda_m}{\Lambda_o} \qquad (7)$$

for all concentrations (and $\alpha \to 1$, when $\Lambda_m \to \Lambda_o$). The normal procedure is to measure Λ_m and do the above extrapolation to determine Λ_o for some related strong salt of the above acid and then to use the Kohlrausch law to determine Λ_o for this acid, rather than to do a direct extrapolation, which is used only for strong electrolytes. In this experiment, you will determine Λ_o for the related salts CH_3COONa, HCl and NaCl and then use some linear subtractions to yield Λ_o for CH_3COOH.

2. OBJECTIVE

To determine the conductivity of various acids and the dissociation constant, K_a, for acetic acid

3. METHOLOLOGY

3.1. Variation of molar conductivities with different electrolytes

An already calibrated machine is available, which consists of a rod-like electrode connected by an electrical lead to a box housing the read-out display and the electronics. Please ensure that you NEVER use the electrode as a stirrer. Instead, please agitate or shake the beaker in which the test solution is placed; the electrode must remain vertical and stationary at all times, clamped to its stand. Electrodes are very expensive and damage to them must be minimized. The reading for the conductivity is the one for the test solutions K_s less the conductivity of distilled H_2O κ_{H_2O} used for the preparation of the solutions. (Ask the technician for samples of distilled water used for the preparation), so that $\Lambda_M = (\kappa_s - \kappa_{H_2O})/c_M$. Normally, κ_{H_2O} is not significant and may be neglected, but check to be sure and decide for yourself. You will be briefed on the usage of the device. The range of measurement is approximately 0-19.99 mS.

1. Prepare the following solutions with the indicated concentrations by using the 100 mL volumetric flasks and the 5, 10, 25 and 50 mL pipettes (either bulb or graduated pipettes). In each case (a)-(d) below, the first value in the series is the stock solution, and the rest are prepared from it by successive dilution. Normally 100 mL is sufficient for each solution volume, but your situation may differ.

   (a) 0.1, 0.05, 0.025, 0.01 mol L^{-1} for NaCl,
   (b) 0.1, 0.05, 0.025, 0.01, 0.005 mol L^{-1} for CH_3COONa,
   (c) 0.02, 0.01, 0.005, 0.0025, 0.001 mol L^{-1} for HCl, and
   (d) 0.1, 0.05, 0.025, 0.01, 0.005, 0.0025 mol L^{-1} for CH_3COOH.

2. Measure the conductivity of each of the solutions at least three (3) times, and take the average value and determine the mean **temperature** of your solutions for which the conductivities apply. Tabulate the solution concentration c, $c^{1/2}$, the conductivity and the molar conductivity for each of the preparations using the **table 1(a) -1(d)** on the handout sheets provided.
3. Draw a graph Λ_m *vs.* $c^{1/2}$ for each of the electrolytes using your experimental data and determine Λ_o (using (3)) by extrapolating to $\sqrt{c} = 0$ for each of the electrolytes and compare the values of Λ_o derived from experiment to the ones from literature (**table 2**). The method fails for one of the electrolytes. Identify the electrode and why?

3.2 Determination of the degree of dissociation index α of acetic acid

1. From the determinations in (iii) above, use Kohlrausch's law to determine the degree of dissociation index, α , for acetic acid.
2. For each of the concentrations given in (i) (4), calculate the required constant; α may be determined from (7) with the experimental values of Λ_m and Λ_o for each electrolyte. K_a is determined from α which was derived previously and (6), where the concentration of the acid is known (Ask technician on duty). Since these measurements are at constant temperature, the K_a values should not vary much and the average gives an indication of the absolute K_a value. Compare the literature value of K_a=1.8 x 10^{-5} (mol dm^{-3}) at 298.15K with your own. Record your calculation into **table 3**.

4. QUESTIONS

1. Relative to your error bounds, are the values for Λ_o and K_a derived from these experiments close to the literature values?
2. The degree of dissociation is given by $\alpha = \Lambda_m/\Lambda_o$. Why is this expression not very accurate? How can it be corrected so that it becomes exact?
   [Hint: refer to the Onsager equation]

4. REFERENCES

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2. Moore, W. J. (1972). Physical Chemistry, 5th edn. Longmans.
3. Levine, I.N. (2002). Physical Chemistry, 5th edn. McGraw-Hill.

Experiment C1 Organic Chemistry

RECRYSTALLIZATION

1. INTRODUCTION

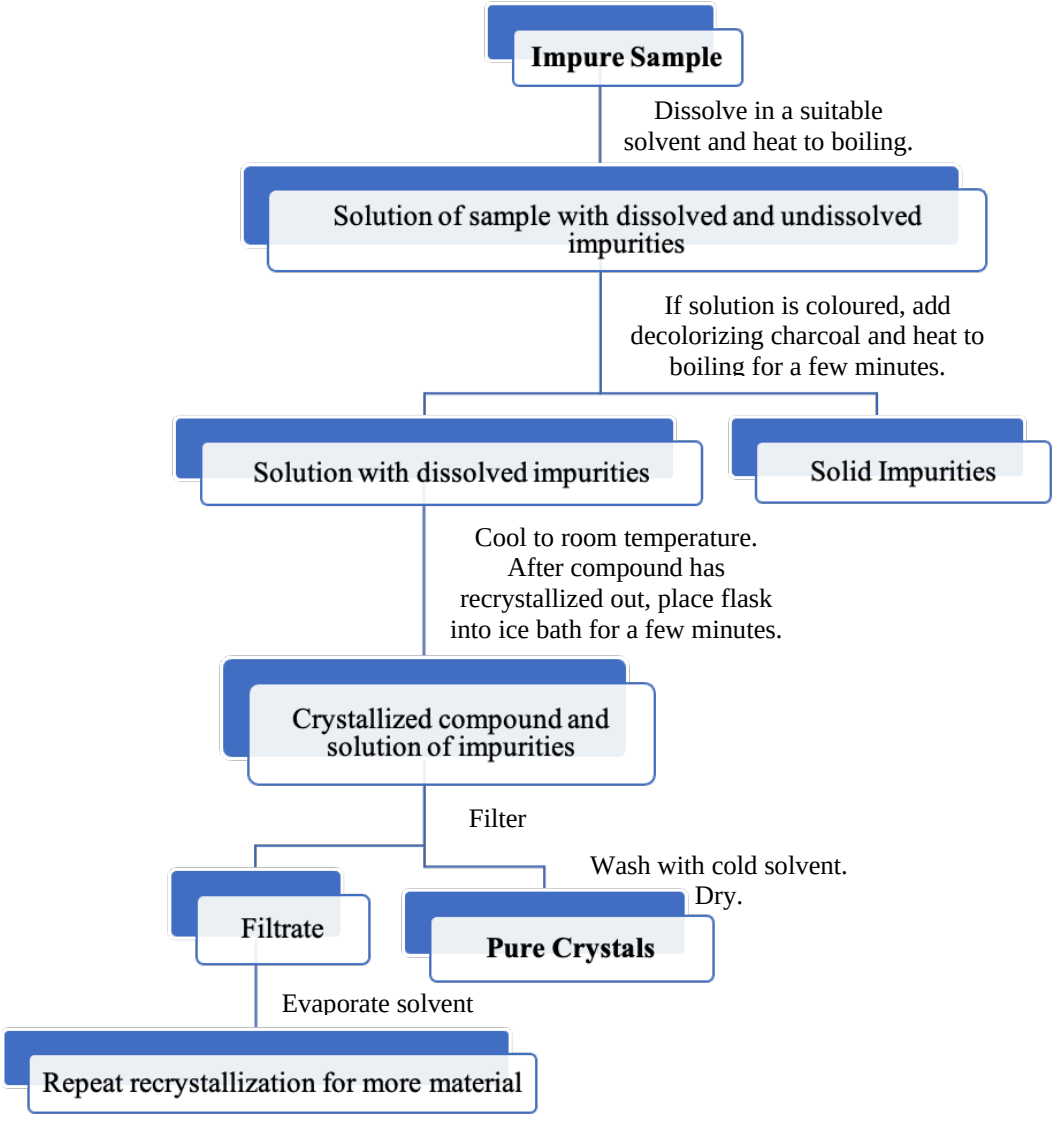
Organic compounds that are solids at room temperature can be purified by recrystallization. The general technique involves dissolving the material to be recrystallized in a hot solvent (or solvent mixture) and cooling the solution slowly. The solid that crystallizes out from the solution is the pure material.

During the recrystallization process, solid impurities (such as dust, filter paper, etc.) that do not dissolve in hot solution are normally eliminated through filtration. The dissolved impurities remain in the cold solution while the pure compound recrystallizes out of the solution.

Pre-Lab Reading/Discussion

- Melting point of crystals
- Crystallization process

The general procedure for recrystallization is as shown in the flow chart below:



2. LEARNING OUTCOME

Purification of solid compound by recrystallisation

3. METHOLOLOGY

3.1. Materials

Glassware	**Chemicals**
Conical flasks	Benzoic acid
Filter funnel	Distilled water
Buchner flask	
Hirsch/Buchner funnel	
Watch glass	

3.2. Experimental Procedure

1. Weigh about 1.0 g benzoic acid into a 100 mL conical flask. Add 15 mL water and anti-bumping granules (3-5 pieces). Heat the mixture on a hot plate until the solvent boils. Add successive small volumes of water (2-3 mL) and continue boiling until all benzoic acid has dissolved (apart from insoluble impurities).

   If the solution is coloured, remove the solution from the hot plate. Cool the solution to room temperature and add decolorizing charcoal (0.2-0.3 g). Mix thoroughly and boil the mixture for several minutes.
2. While waiting for the solution to boil, prepare the fluted filter paper and put it in the funnel. Put the funnel fitted with fluted filter paper in a conical flask. Add a little water and anti-bumping granules into the conical flask and heat on a hot plate.
3. Filter the hot mixture of benzoic acid through a fluted filter paper into the heated conical flask. If the filtration is done in batches, keep the remaining solution hot throughout the filtration process. If crystallization occurs on the filter paper, add a minimum volume of boiling water to re-dissolve the crystals, and allow the solution to pass through the funnel. Add hot solvent in small volumes until all crystals are dissolved. After filtration, boil the filtrate to produce a more concentrated solution.
4. Cover the conical flask with a watch glass and allow the solution to cool to room temperature, then in an ice-bath after the crystallization has occurred. If no recrystallisation occurs at this stage, it may be due to the fact that too much water was used. Concentrate the solution by heating on the hot plate and cool.
5. When all the benzoic acid crystals have crystallized out, filter the crystals through a Hirsch/Buchner funnel at the suction/water pump. Transfer all the crystals in the flask into the funnel by rinsing the flask with some of the filtrate. Wash the crystals with a little cold water and air dry.
6. Place the crystals in a watch glass to air dry or dry the crystals rubbing between two filter papers. Let the crystals dry completely before taking the melting point. Weigh the pure benzoic acid recovered, calculate the percentage yield.

4. QUESTIONS

1. Explain why anti bumping granules are added before any solution is heated?
2. What is the purpose of the recrystallisation process?
3. Why is suction filtration favoured over gravitational filtration when separating pure crystals from its supernatant liquid after the recrystallisation?
4. Explain how the washing of crystals is carried out.
5. Why it is necessary to remove all the solvent before the melting point of the pure compound can be determined?
6. In general, water is not a good solvent for the recrystallisation. Explain this statement.

5. REFERENCES

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2. Skoog, D.A.; West, D.M.; Holler, F.J.; Crouch, S.R. Fundamentals of Analytical Chemistry, Eight Edition. Brooks/Cole Cengage Learning, US, 2004.

Experiment C2 Organic Chemistry

SEPARATION BY CHROMATOGRAPHIC TECHNIQUES

1. INTRODUCTION

"Chromatography" literally means colour graphing and this technique was first used by a Russian botanist in the early 1900's to describe the separation of coloured plant pigments by passing a plant extract down a column of calcium carbonate and washing it with petroleum ether. Today, chromatography is a procedure which is commonly used to separate components in mixtures.

In general, chromatographic method involves putting a mixture to be separated on a stationary phase and a mobile phase is then passed through the stationary phase. The stationary phase can be of porous solids or a layer of liquid covering the surface of a suitable solid support while the mobile phase can be either a liquid or a gas. The adsorbents that are normally used as stationary phase are sucrose, cellulose, starch and inorganic carbonates but most separations are carried out using silica gel or alumina. The solvent used as the mobile phase usually has low boiling point and low viscosity, such as petroleum ether, ligroin, diethyl ether, dichloromethane, ethyl acetate, acetone, ethanol and methanol.

The principle of chromatographic separation relies on that fact that the different components in the starting mixture are adsorbed by the stationary phase and desorbed back into the mobile phase to different degrees, as they are moved with the mobile phase. The difference in the adsorption-desorption properties result in each compound passing through a given amount of stationary phase (often a column) at a different time (retention time). Thus, the components of the mixture are separated.

There are a variety of chromatographic techniques, which may be used depending upon what types of compounds are present in a mixture:

Thin layer chromatography (TLC) is carried out on a very thin layer of chromatographically active material dispersed on the surface of an inert support such as plastic or glass. A small volume of a solution of the mixture to be separated is 'spotted' onto the stationary phase, near the bottom of the paper or plate (the location is marked for later reference). The solvent from the solution is evaporated and TLC plate is then placed in a 'tank' containing the mobile phase. The level of the mobile phase should be below the location of the spot. The mobile phase then moves up the TLC plate and the separation is stopped when the mobile phase has nearly reached the top of the stationary phase. The mobile phase boundary is then marked and the mobile phase is allowed to evaporate. The locations of the different components in the mixture are then identified either visually (for coloured compounds), by UV light, or by a chemical development (iodine vapour, ceric nitrate spray, sulfuric acid spray, *etc*.). The R_f of each component (distance travelled by component divided by distance travelled by the mobile phase) is then calculated. The R_f is a constant for a given compound under a fixed set of conditions (mobile and stationary phase). By comparing with R_f values for known compounds (standards), the components in the sample mixture can be identified.

In a column chromatography, the stationary phase (typically alumina or silica) is held in a column (usually glass) and the mobile phase is passed down this column by aid of gravity. A column used is typically 5-50 cm long and 5-50 mm wide and usually has a stopcock (tap) on the bottom to halt the flow of the mobile phase. The stationary phase is usually held in place with an inert material at bottom (sintered glass, cotton or glass wool etc.) and is often protected at the top with fine sands or some inert powder. The column is usually established by pouring the slurry absorbent into the column. The solvent is then allowed to pass through the column until the level is just above the top of the stationary phase. The mixture to be separated is then added to the top of the column (in a solvent) and solvent is allowed to flow until the stationary phase is just covered with solvent. The mobile phase is then added to the top of the column and is allowed to pass through the column in

order to separate the mixture components. Fractions are collected from the bottom of the column and analysed so as to locate and identify the individual components.

In this experiment column chromatography is used to separate various components in a mixture and thin layer chromatography (TLC) is used to test the purity of the compounds separated.

Pre- Lab Reading/Discussion

- Hydrophobicity and hydrophilicity
- Molecular structure of silica
- Hydrogen bonding & polarity

2. LEARNING OUTCOME

1. Separation of compounds by column chromatography
2. Visualisation of separation by TLC using UV-detection

3. METHOLOLOGY

3.1. Materials

Glassware	**Chemicals**
Column	Mixture of *m*-nitroaniline and pyrene
Chromatographic plate (2.5 cm x 10 cm)	Silica gel
Test tube, capillary tube	Hexane
Chromatographic tank	Ethyl acetate
Glass wool	Anhydrous sodium sulphate
Beaker	
Conical flask (50 mL)/test tube	

3.2 Experimental Procedure

3.2.1. Column chromatography

3.2.1.1. Packing the column: The slurry method

1. Stir hexane with silica gel (4.0-4.5 g, depending on the length of the column) into a thin, homogeneous slurry mixture in a conical flask.
2. Pack the column by filling the column half full with the eluent solvent mixture of hexane/ethyl acetate (EA) (2:1). Place a loose plug of glass or cotton wool at the bottom of the column using a long glass rod. Ensure all entrapped air has been forced out. Pour a small amount of anhydrous sodium sulphate (or fine sand) into the column so that a small clean layer is formed on top of the glass wool. Tap the column to level the surface of the sodium sulphate. Wash down any sodium sulphate that adheres to the side of the column with a minimum volume of solvent. The sodium sulphate layer forms a base that supports the column adsorbent and prevents it from washing through the stopcock.
3. To pack the column with the slurry of adsorbent prepared earlier, open the stopcock of the column and allow the solvent to drain slowly into a beaker. Pour the slurry in portions into the column. While pouring, tap the column constantly and gently at the

side with a pencil fitted with a rubber stopper or with a rubber tube. Continue tapping until all the material has settled. Drain the solvent until it is just level with the top of the absorbent. Do not let the column run dry. Add anhydrous sodium sulphate to the surface of the adsorbent to protect the surface of the adsorbent from been disturbed.

3.2.1.2. Separating the sample on the column

1. Apply the mixture of *m*-nitroaniline and pyrene carefully around the circumference of the adsorbent using a capillary pipette so that a layer of the mixture is formed evenly on to top of the sodium sulphate covering the absorbent. Care should be taken not to disturb the surface.
2. Drain out the solvent until to bring the liquid level to the top of the absorbent again. Pipette a little eluent around the inside of the column to rinse down any inherent sample and once again, drain out the solvent until the liquid level to the top of the absorbent again.
3. Carefully add the eluent and begin collecting the eluate. Continue adding the eluent to ensure that the liquid level is nearly constant throughout the elution. Collect the eluate in a clean, dry flask.
4. When the first component in the mixture has been completely eluted, change to another flask and begin collecting the second component in the mixture. Continue elution until the yellow band is completely drained from the column.
5. Concentrate both the colourless and yellow eluates by heating on the water bath. The purity of both the compounds separated is then tested by TLC.

3.2.2. Thin layer chromatography

3.2.2.1. Spotting the plate

1. Dissolve the *m*-nitroaniline and pyrene fractions that have been collected in **3.2.1.2**. in dichloromethane.
   Note: Dissolve remaining mixture in dichloromethane and use for reference purpose.
2. Using a pencil, draw a straight line on the thin layer chromatography plate and mark two spots on the plate as shown in Diagram 1. The line with the spots must be high enough (about 1 cm from the bottom) to ensure that the compounds placed on it will not be exposed to the reservoir of the developing solvent (mobile phase) and dissolve in it.
   Note: Better prepare three (3) marks. The central mark is for the original mixture. This enables a comparison of the fractions with the original mixture.
3. Fill a capillary tube with the solution prepared in (1) by dipping one end of the tube into the solution. Capillary action fills the tube. Empty the tube by touching it lightly to the thin layer plate on the marked spot. When the tube touches the plate, the solution is transferred to the plate as a small spot. It is important to touch the plate very lightly so that the adsorbent layer on the surface of the plate is not scratched or broken.

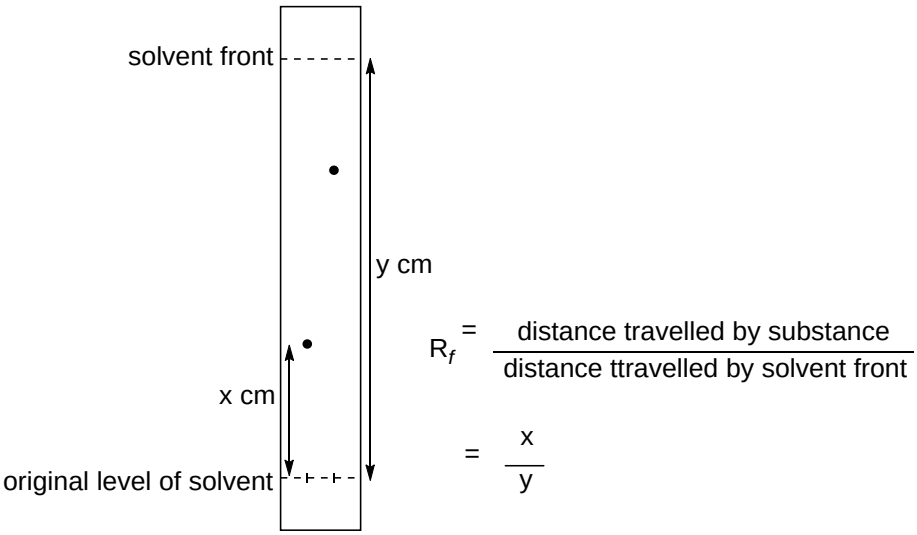


Diagram 1. Preparing the TLC plate

3.2.2.2. Developing the TLC plate

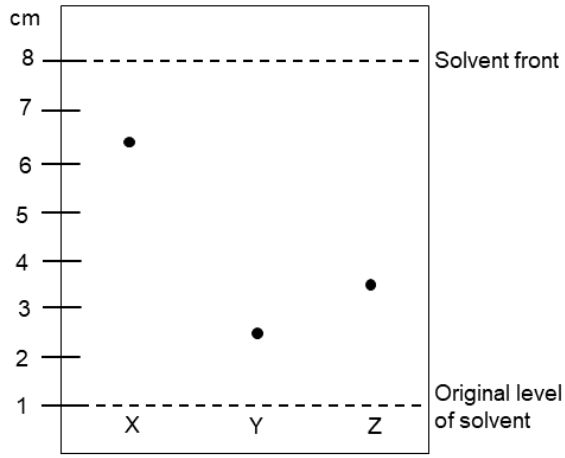
1. Line the inside of the developing tank/jar with a piece of filter paper. Pour the eluent solvent (hexane/ethyl acetate 2:1) into the developing tank/jar to a depth of a few millimeters and cap the developing tank/jar. Before the development, make sure the filter paper inside the tank thoroughly moistened with the eluent solvent. Once the filter paper liner is saturated, adjust the level of developing solvent in the bottom of the tank to a depth of about 5 mm. Cover the tank until it ready for use.
2. Place the spotted plate vertically (the end where the spots are, is at the bottom) into the developing tank and replace the cap. The eluent level must be below the spots. The solvent will rise slowly in the absorbent by capillary action. As the solvent rises, the plate becomes visibly moist. Do not move or open the chamber during the developing process. When the solvent has reached within 5 mm of the end of the coated surface, remove the plate and immediately draw a line across the plate to mark position of the solvent front with a pencil. Allow the solvent to evaporate from the plate.

3.2.2.3. Visualization

If the substance in the sample is coloured, they may be observed directly. If not, they can be visualized by shining an ultraviolet (UV) light on the plate. Mark the visible spots with a pencil and calculate the R_f values for both compounds. Colourless spots on the TLC plate may also be exposed to iodine vapour for visualization.

4. QUESTIONS

1. Explain the following terms in relation to chromatography.
   i. Mobile phase
   ii. Stationary phase
   iii. R_f value
2. Calculate the R_f value for compounds X, Y and Z in TLC plate given below.



3. Suggest one suitable chromatographic technique which can be used to separate a mixture of amino acid.
4. What are the advantaged of column chromatography over TLC?
5. Arrange the following compounds in the order of increasing polarity.
   ortho-nitroaniline, *para*-nitroaniline, *meta*-nitroaniline

5. REFERENCES

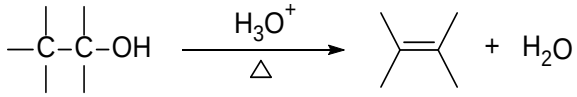
1. McMurry, J. Organic Chemistry. Brooks/Cole Cengage Learning. 2008
2. Skoog, D.A.; West, D.M.; Holler, F.J.; Crouch, S.R. Fundamentals of Analytical Chemistry, Eight Edition. Brooks/Cole Cengage Learning, US, 2004.
3. Robinson, J.W. Undergraduate Instrumental Analysis, Fifth Edition, Revised and Expanded. Marcel Dekker, Inc. 1995.

Experiment C3 Organic Chemistry

PREPARATION OF CYCLOHEXENE FROM CYCLOHEXANOL

1. INTRODUCTION

Alkenes can be prepared from alcohols by heating the alcohol in the presence of an acid. Two of the common methods used are heating the mixture of alcohol with sulphuric acid or phosphoric acid or passing the alcohol vapour over activated alumina at high temperature. The latter is an industrial method. In both reactions, water is eliminated and hence the reaction is known as dehydration.



In this experiment, the students will perform the dehydration of cyclohexanol, by heating the cyclohexanol in the presence of sulphuric acid. The acid catalyses the reaction by protonating the hydroxyl group, making it a good leaving group. Elimination of water from the protonated alcohol produces an alkene.

According to Le Chatelier's principle, elimination of one species from a product mixture will shift the equilibrium to the side that favours the formation of the product. Therefore, in this reaction, the cyclohexene and water formed are distilled out once they are formed. The elimination of these products shifts the equilibrium to the right and increases the yield of cyclohexene produced.

After washing and drying of the crude product, distillation gives pure cyclohexene. The technique used in the washing of a liquid is essentially an extraction process.

Pre-Lab Reading/Discussion

- Le Chatelier's Principle
- Dehydration of alkane

2. LEARNING OUTCOME

1. Purification of liquids by distillation
2. Purification of compounds by solvent-solvent extraction
3. Acid catalysed elimination of alcohols

3. METHOLOGY

3.1. Materials

Glassware	**Chemicals**
Flat round bottomed flask	Cyclohexanol
Fractional distillation column	Concentrated sulphuric acid
Still head	10% sodium carbonate solution
Thermometer	Anhydrous calcium chloride
Receiving adapter	
Separating funnel	
Conical flask	
Condenser	
Pear shaped flask	

3.2. Experimental Procedure

1. Place cyclohexanol (20.0 g, 21 mL) and concentrated sulphuric acid (2 mL) into a 100 mL round flat bottom flask. Shake the contents carefully by wrist motion and add a few anti-bumping granules.
2. Fit in the fractional distillation column complete with Still head, thermometer, condenser, receiving adapter and a receiving flask to collect the product.
3. Heat the reaction mixture slowly using a hot plate so that cyclohexene and water formed will distill out through the fractional column. Continue distilling until only a small volume of residue is left in the flask (not to dryness). Ensure that the temperature at the top of the column does not exceed 100°C during the distillation process.
4. Pour the distillate (crude product) into a separating funnel and wash carefully with a 10% aqueous solution of sodium carbonate (1-2 mL).
   (Note: evolution of carbon dioxide)
5. Transfer the organic layer into a conical flask (clean and dry) and add anhydrous calcium chloride. Leave for 15-20 minutes to dry.
6. While waiting for the crude product to dry, set up the distillation apparatus. Filter the crude product into the distillation flask and distill using a water bath. Record the boiling point and the mass of the cyclohexene.

4. QUESTIONS

1. Give two advantages of using phosphoric acid over sulphuric acid in the dehydration reaction of an alcohol.
2. The by-product formed in the dehydration of cyclohexanol is dicyclohexyl ether. Write the mechanism for the formation of this by-product.
3. If 2-methylcyclohexanol undergoes dehydration process, what is/are the alkene/s formed?
4. Why do you need to wash the distillate with a solution of sodium carbonate?
5. Why can the temperature at the top of the condenser in the fractional distillation **not** exceed 100°C?

5. REFERENCE

McMurry, J. Organic Chemistry. Brooks/Cole Cengage Learning. 2008

Experiment C4 Organic Chemistry

STEREOCHEMISTRY

1. INTRODUCTION

Molecular models are valuable tools to visualise the geometry of molecules and particularly stereochemistry. Significant costs of the modelling kits limit the application for students. Therefore, this experiment provides an opportunity to use physical models to enhance the understanding of organic stereochemistry. An alternative to physical models can be found in specific software package that enable 3D displays of molecules. Suitable non-licenced software can be found for computers (Windows) and tablets with various operating systems.

2. LEARNING OUTCOME

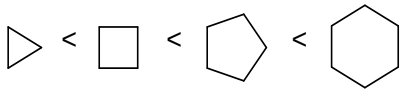
Visualisation of the 3-dimensional structure of organic molecules

3. METHOLOGY

3.1. Geometry and conformations

Molecular building kits are in limited supply in the laboratory. If required, students are advised to share.

1. Construct a model of methane, the simplest organic compound. Note that all the hydrogens are as far apart as possible. What is the angle between any two hydrogen atoms, as measured through the carbon atom?
2. Construct a model with 2 saturated carbon atoms and the appropriate number of hydrogens. How many hydrogens are needed to complete all the covalences in the C2 model?
3. (a) Construct a model of pentane. Observe that the chain is not straight and note the tetrahedral geometry of each carbon.
   (b) Make molecular models of the other two constitutional isomers for the five-carbon alkane: isopentane (or 2-methylbutane) and neopentane (or 2,2-dimethylpropane). Identify all the primary, secondary and tertiary hydrogens in all three C5 isomers.
4. Make a model of bicyclo[3.2.1]octane. Draw its structure.
5. Using Newman projections draw all conformations that result when 2-methylbutane is rotated around the C2—C3 bond. Draw a graph of energy versus dihedral angle for the different conformational isomers.
6. The relative stabilities of cycloalkanes are as follows. Why?



   (a) Construct models of cyclopropane, cyclobutane and cyclopentane.
      (i) Would you expect the molecules to be planar?
      (ii) Are there angle strains in the above molecules?

(b) Construct a model of cyclohexane.

(i) Do the carbons in the ring all lie in one plane?

(ii) By rotating some of the bonds you should be able to make your model look like the figure below. The model should sit firmly on the desktop with three hydrogens serving as legs. This model is the chair conformation of cyclohexane.

(iii) Look straight down each of the carbon-carbon bonds in the molecule. Are the bonds staggered or eclipsed?

(iv) Does the chair conformation of cyclohexane possess any torsional strain?

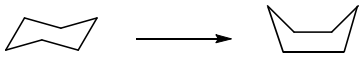
(v) Do any of the bonds in the chair conformation appear to be strained or bent?

(vi) What are the bond angles in the chair conformation?

Note that in the chair conformation, three of the hydrogens point straight up and three point straight down. These hydrogens are said to be in axial positions. The other six hydrogens radiate outward along the perimeter of the ring. These hydrogens are in equatorial positions.

(v) Draw the chair conformation of cyclohexane and label all of the hydrogens as equatorial or axial.

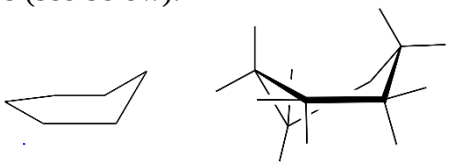
Grasp one of the carbons with an axial hydrogen pointing down and force it to point upwards (bond rotations are required). You should be able to get your model to look like a boat. (See figure below.) This unstable conformation is known as the boat conformation.



(vi) Are the C-C bonds all staggered in the boat conformation? If not, draw the boat and indicate which bonds are eclipsed.

(vii) Does the boat conformation of cyclohexane possess any torsional strain?

Other conformations of cyclohexane include the half chair and the twist conformations (see below).

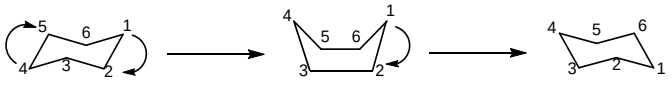


half chair twist

(c) Consider chair conformations of the cyclohexane ring only.

Remove an equatorial hydrogen from the cyclohexane model and add a CH_3 group in its place to form methylcyclohexane. Draw the structure represented by the model.

Now grasp and invert C-1 (the carbon with the methyl group) to form a boat conformation. Then take C-4 and invert it to remake a chair conformation. This process inverts the chair conformation and is called chair flipping.



(i) After the chair flip, what is orientation of the methyl group?

(ii) Draw the structure represented by the model.

(iii) Which chair conformation of methylcyclohexane is more stable? Why?

7. Conformations of Disubstituted Cyclohexanes
   (a) Replace the equatorial hydrogens on C-1 and C-2 of cyclohexane with methyl groups to make a model of **1,2-dimethylcyclohexane**. Are the methyl groups in this model *cis* or *trans*?

   Flip the ring to its other chair conformation.

   (i) What are the orientations of the two methyl groups now?
   (ii) Are the methyl groups in this model *cis* or *trans*?
   (iii) Which of the two chair conformations is more stable? Explain.

   (b) Now make a model of 1,2-dimethylcyclohexane with one of the methyl groups in an equatorial position and the other in an axial position. Are the methyl groups in this model *cis* or *trans*?

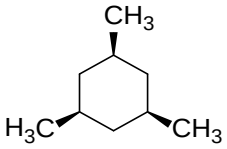
   Flip the ring to its other chair conformation.

   (i) What is the orientation of the bonds that connect the methyl groups to the ring?
   (ii) Are the methyl groups in this model *cis* or *trans*?
   (iii) Which of the two chair conformations is more stable? Explain.

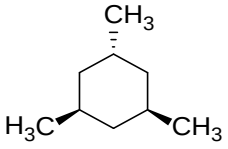
   (c) Now make models of *cis* and *trans* 1,3-dimethylcyclohexane. Find the most stable chair conformation of each.
   (i) Draw the most stable conformation of the cis isomer.
   (ii) Draw the most stable conformation of the trans isomer.
   (iii) Which isomer is more stable, cis or trans? Why?

   (d) Now make models of cis and trans 1,4-dimethylcyclohexane. Find the most stable chair conformation of each.
   (i) Draw the most stable conformation of the cis isomer.
   (ii) Draw the most stable conformation of the trans isomer.
   (iii) Which isomer do you think is more stable, cis or trans?

8. There are two stereoisomers of 1,3,5-trimethylcyclohexane, all-*cis* and *cis-trans-trans*. Make models of these molecules.



all-*cis*



cis-trans-trans

   (a) Determine the more stable isomer.
   (b) Draw the molecule in its most stable (chair) conformation.

3.2. Stereoisomers

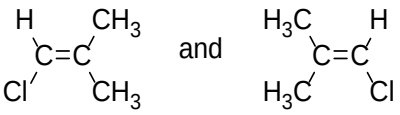
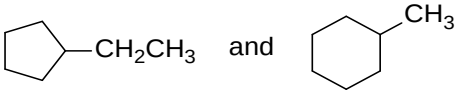
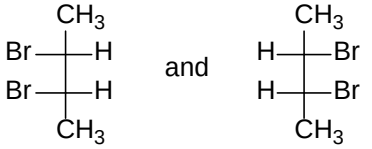
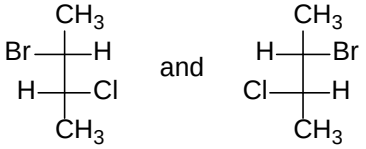
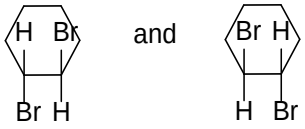
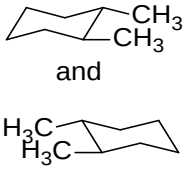
1. Construct a model of compound with four different groups attached to a central carbon (the stereogenic centre). Make its mirror image. Manipulate the two models to convince yourselves that they are not superimposable. Make a similar model that contains only three different groups. Show that these are superimposable.

2. Construct a model of 2-chloropropane and its mirror image.
   (a) Does the molecule have a stereogenic centre?
   (b) Are the two models identical?
   (c) Are they enantiomers?

3. Construct a model of 2-butanol and identify its stereogenic centre. Make its mirror image.
   (a) Are the two models superimposable?
   (b) What is the relationship between the two models?
   (c) Draw the two models and name them using the (*R*), (*S*) convention.

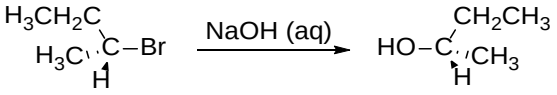
4. Construct a model of 3-bromo-2-butanol.
   (a) How many stereogenic centres are present in the molecule?
   (b) How many stereoisomers are possible for this molecule? Determine the stereochemical relationship between each pair of molecules.

5. Consider the following pairs of structures. Identify their relationship as representing molecules of the same compound, constitutional isomers, diastereomers, enantiomers or meso compounds.

(a)		(b)	
(c)		(d)	
(e)		(f)	

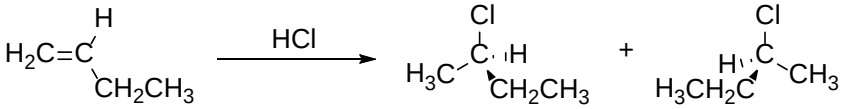
6. Construct models and draw Fischer projection formulas for the following.
   (a) (*R*)-1-Bromo-1-chlorobutane
   (b) (2*R*,3*R*)-2,3-Dibromopentane
   (c) A compound with a molecular formula of $C_2H_2(NH_2)_2Cl_2$ whose structures are
      (i) optically active
      (ii) optically inactive due to a plane of symmetry

7. The following reaction shows that an inversion of configuration has occurred.



   Construct models of the two molecules and show that both have different configurations.

8. The following reaction gives a racemic product. Explain.



   What is the stereochemical relationship between the two products?