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# **FIRST YEAR CHEMISTRY LABORATORY MANUAL**

**2018**

**CHEM10003 Chemistry 1**

*plus*

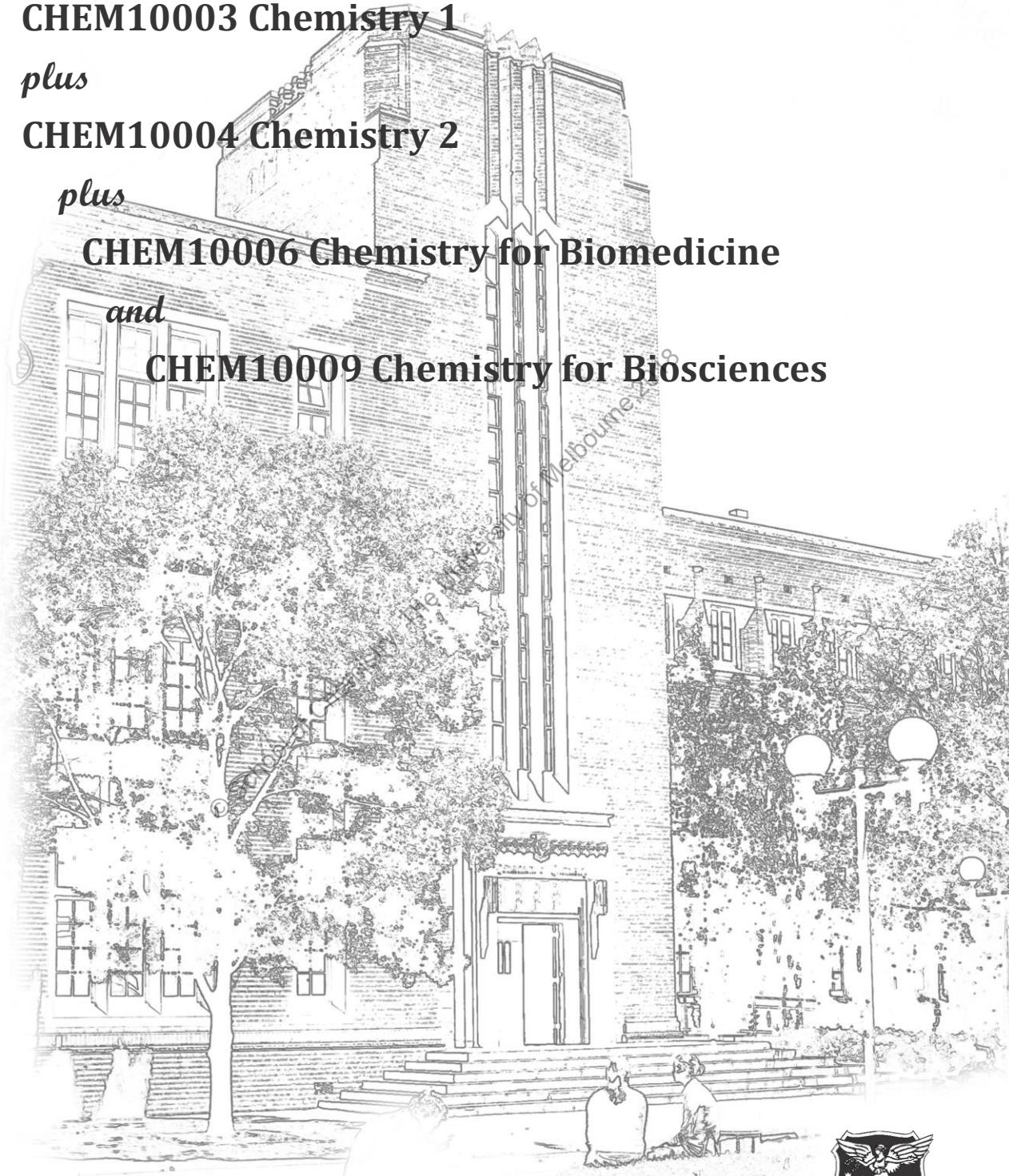
**CHEM10004 Chemistry 2**

*plus*

**CHEM10006 Chemistry for Biomedicine**

*and*

**CHEM10009 Chemistry for Biosciences**



**THE UNIVERSITY OF  
MELBOURNE**

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# CHEMISTRY LABORATORY PROGRAM 2018

This manual is required for any student undertaking the practical component of CHEM10003 and CHEM10004, the manual also satisfies the practical requirements of CHEM10006 and CHEM10009:

- CHEM10003 – Chemistry 1 (semester 1)  
Experiments 1 – 6.
- CHEM10003 – Chemistry 1 (semester 2)  
Experiments 1 – 6.
- CHEM10004 – Chemistry 2 (semester 2)  
Experiments 7 – 12.
- CHEM10004 – Chemistry 2 (summer semester 2019)  
Experiments 7 – 12.
- CHEM10006 – Chemistry for Biomedicine (semester 1)  
Experiments 2, 5, 7, 9, 11, 13.
- CHEM10009 – Chemistry for Biosciences (semester 1)  
Experiments 2, 5, 7, 9, 11, 13.

As set out in the following table, different textbooks will be prescribed for the different first-year subjects in Chemistry. Since both Burrows Chemistry<sup>3</sup> and the Blackman Chemistry textbooks will be supported by the library, suggested reading references are given to both books. It is NOT expected that students will have copies of both textbooks.

Subject Name	Subject Code	Prescribed Text
Chemistry 1	CHEM10003	<i>Chemistry</i> <sup>3</sup> , Burrows, Holman, Parsons, Pilling and Price 3 <sup>rd</sup> ed. 2017
Chemistry 2	CHEM10004	<i>Chemistry</i> <sup>3</sup> , Burrows, Holman, Parsons, Pilling and Price 3 <sup>rd</sup> ed. 2017
Chemistry for Biomedicine	CHEM10006	<i>Chemistry</i> , Blackman, Bottle, Schmid, Mocerino and Wille 3 <sup>rd</sup> ed. 2016
Chemistry for Biosciences	CHEM10009	<i>Chemistry</i> , Blackman, Bottle, Schmid, Mocerino and Wille 3 <sup>rd</sup> ed. 2016

# First Year Chemistry Laboratory – Practical Checklist

- Practical classes start PROMPTLY**
  - at 10:00 a.m. (morning sessions)
  - at 2:00 p.m. (afternoon sessions)
  - at 6:15 p.m. (evening sessions)
- CHECK timetable for your practical week, time and appropriate experiment**
- COMPLETE ChemCAL Prelabs for APPROPRIATE experiment before practical**  
**FILL OUT and sign the ChemCAL Prelabs receipt form**
  - reschedule practical for NOT COMPLETING the Prelabs
  - reschedule practical for completing for the WRONG EXPERIMENT
- BRING laboratory manual, notebook and a basic scientific calculator**
- WEAR shoes that enclose whole feet up to ankle (e.g. runners and boots)**
- BRING safety glasses or goggles**
- BRING laboratory coat (long sleeved and knee length)**
- TIED UP long hair and NO hat or cap in laboratory**
- DO NOT use mobile phones in laboratory**
- BORROWING safety glasses and laboratory coats**
  - limited number available
  - from 1<sup>st</sup> year enquiries counter
  - personal photo ID is required
  - same practical session return is required
  - maximum on TWO occasions
- RESCHEDULING practicals**
  - subject to availability of lab spaces
  - at 1<sup>st</sup> year enquiries counter
  - maximum TWO practicals may be rescheduled
- PRACTICAL EXEMPTION**
  - submit medical certificate at 1<sup>st</sup> year enquiries counter
  - submit relevant document at 1<sup>st</sup> year enquiries counter
  - maximum TWO practicals may be exempted
  - absence WITHOUT EXEMPTION will receive practical mark of 0/20

# Periodic Table

1		2		3		4		5		6		7		8		9		10		11		12		13		14		15		16		17		18	
alkali metals		alkaline earth metals		metals		nonmetals		metalloids		halogens		chalcogenes		pictogens		noble gases		2		He		He		He		He		He		He					
1	H	Li	Be	Na	Mg	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	Ar	Cl	S	P	O	F	Ne					
2	[1.008]	[6.94]	9.012	[22.99]	[24.31]	39.10	40.08	44.96	47.87	50.94	52.00	54.94	55.85	58.93	58.69	63.55	65.38(2)	69.72	72.63	74.92	78.97	[32.06]	[35.45]	39.95	[10.81]	[12.01]	[14.01]	[16.00]	19.00	20.18					
3	[138.9]	[227.0]	140.1	140.9	137.3	87.62	88.91	91.22	92.91	95.95	97.91	101.1	102.9	106.4	107.9	112.4	114.8	118.7	121.8	127.6	129.0	131.3	131.3	131.3	131.3	131.3	131.3	131.3	131.3	131.3					
4	[1.389]	[2.270]	1.401	1.409	1.373	1.8762	1.8891	1.9122	1.9291	1.9595	1.9791	2.011	2.029	2.064	2.079	2.112	2.148	2.187	2.218	2.276	2.329	2.389	2.431	2.474	2.511	2.557	2.591	2.621	2.651	2.681					
5	[89]	[227.0]	140.1	140.9	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3	137.3					
6	[1.89]	[2.27]	1.401	1.409	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373	1.373					
7	[223.0]	[226.0]	265.1	268.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1	271.1					
lanthanoid series	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86					
actinoid series	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	No	Md	Fm	Cf	Es	Fm	Md	No	Lu	Fr	Ra	Fr	Ra	Fr	Ra	Fr	Ra			

# First Year Chemistry Laboratory Information

## Student Lab Group Allocation and Practical Session

Student lab group allocation will be posted on Learning Management System (LMS) under Lab Information and on the notice board outside the first-year laboratory in the foyer of Chemistry building. This will normally be posted in the week prior to Week 1 of each semester and indicates the student ID/enrolled course/day/time/allocated group number for practical class.

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**All morning sessions start PROMPTLY at 10:00 a.m.**

**All afternoon sessions start PROMPTLY at 2:00 p.m.**

**All evening sessions start PROMPTLY at 6:15 p.m.**

*For safety reasons, student will NOT be permitted to enter the laboratory later than 10:10 a.m. (morning sessions) or 2:10 p.m. (afternoon sessions) or 6:25 pm (evening sessions). If you are unable to arrange to do the experiment at later time you will carry a grade of 0/20 for that experiment.*

For each practical session:

1. Check the practical timetable for the appropriate week number and experiment.
  2. Complete the appropriate ChemCAL Prelabs module for each experiment.
  3. Fill out and sign the ChemCAL Prelabs receipt form (at the back of lab manual).
  4. **MUST WEAR SHOES ENCLOSE ENTIRE FEET UP TO ANKLE (e.g. runners and boots).**
  5. Bring safety glasses and laboratory coat (long-sleeved and knee length).
  6. Bring lab manual, laboratory notebook and a basic scientific calculator.
- 

## Practical Timetables

Timetables for practical sessions will be posted on Learning Management System (LMS) under Lab Information and on the notice board outside the first-year chemistry laboratory.

**Notes: It is important to check for the CORRECT week number and APPROPRIATE experiment.**

## Attendance

**Students are expected to complete ALL SIX practicals.**

Each practical is given a mark out of 20. The final practical mark is an average over ALL SIX practicals.

Students who are absent will NOT be penalised if a medical certificate or any relevant documentation is submitted at the enquiries counter of the first-year chemistry laboratory. If possible these students will arrange a "make-up" practical at a time that does not clash with their timetabled classes. If it is not possible you will receive your average practical mark for your missed experiment.

## Rescheduling Practical Session

Please be aware that rescheduling a missed class is not always possible. It is subject to availability of lab spaces and depends on the number of sessions that any individual experiment still to run. Any request for these "make-up" practicals can be made at the enquiries counter outside the first-year laboratories. A total of two practical classes may be rescheduled in this way.

**Notes: If it is not possible to arrange for a "make-up" class and you do not have a validated medical or other reason for your absence then you will be given a score of 0/20 for any practicals you do not attend.**

## Notes

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# Practical Program

## General Information

The aims of practical work are to:

- Teach you manipulative skills.
- Enhance your skills of observation and deduction.
- Introduce you to report writing and data handling.
- Illustrate the experimental basis of lecture material.

**Attendance at laboratory classes is compulsory and will be monitored. Failure to pass the laboratory component of the subject will result in failure of the subject as whole, regardless of performance in the examination.**

**Note: Under normal circumstances students are expected to complete ALL SIX practicals.**

For each of the first-year Chemistry subjects the practical program consists of six experiments. The scheduling of these experiments will be posted on the LMS prior to the commencement of the laboratory program. Experiments are completed in rotation through the semester.

**Student practical class group allocation and timetable will be posted on LMS and on the notice boards at the beginning of semester – check the day, time and group number to which you have been assigned. (e.g. Mon/2pm/7 indicates practical class on Monday starting 2.00 pm, Group 7; Tue/10am/3 indicates practical class on Tuesday starting 10.00 am, Group 3).**

Classes begin promptly at either 10:00 am or 2:00 pm or 6:15 pm and finish promptly at 1:00 pm or 5:00 pm or 9:15 pm. It is essential that all students attend on time. Any student arriving more than 5 minutes late will be excluded from the session.

The "make-up" practical classes are conducted at different session times and are subject to availability of spaces. A total of two practical classes may be rescheduled in this way. Please be aware that rescheduling a missed class is not always possible and depend on the number of sessions that any individual experiment still to run. The procedure for requesting "make-up" practicals will be posted at the enquiries counter outside the first-year laboratories.

**If it is not possible to arrange for a make-up class and you do not have a validated medical or other reason for your absent then you will be given a score of 0/20 for any practicals you do not attend.**

## Absences from Practical Classes Due to Illness

Students who are absent from practical classes will not be penalised if a medical certificate\* is provided. If possible students will arrange a make-up experiment at a time that does not clash with other timetabled classes. If this is not possible you will receive your average practical mark for the missed experiment(s). Please note that

**ALL students must complete at least FOUR experiments to be eligible to pass the subject.**

\* **Medical Certificates:** It is important that all Medical Certificates be presented at the enquiries counter outside the first-year laboratories within three (3) working days from the date of absence so that your absence can be officially noted. In cases of ongoing illness, the medical certificate can be emailed chemistry first year at [chemistry-first-year@unimelb.edu.au](mailto:chemistry-first-year@unimelb.edu.au). Certificates received after this time will not normally be accepted.

## Absences from Practical Classes Due to Other Reasons

Make up practical sessions may also be arranged for non-medical reasons, such as accident or family circumstances. It is essential to provide documentation in such instances.

**Irrespective of medical or other reasons, students must complete at least FOUR out of the six experiments to pass the practical component of the subject.**

## Preparation for Laboratory Classes

Before you start the first practical class you will need to purchase:

- An A4 duplicate book for recording results and report writing.
- A basic scientific calculator.
- MUST wear shoes that enclose the ENTIRE feet up to ankle (e.g. runners and boots).
- A pair of approved safety glasses or goggles.
- A long-sleeved, knee-length laboratory coat.

It is essential to develop safe working practices in the laboratory. Please read the Safety in the Laboratory section of these notes (from page 13) before your first practical class.

**Before each practical class you are expected to:**

- Check the practical timetable for the appropriate week number and experiment.
- Read the notes, appropriate text references and relevant “Tips” posted on LMS under Lab Information.
- Complete the appropriate ChemCAL PreLabs module.
- Fill out and sign the ChemCAL Prelabs receipt form (at the back of lab manual).
- Make a note of the activities to be carried out during the session. Have a “plan of attack”.
- Prepare appropriate data tables, graph axes and possible answers to questions.
- Note any materials or resources such as graph paper or a basic scientific calculator which are to be brought along to the practical class.

## Chemcal Prelabs Online

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out each practical exercise.

You can access the ChemCAL PreLabs Online in the same way you use ChemCAL Online – from the Chemistry computer lab, from the public access computer labs on campus, or over the web from home. Use your web browser to go directly to <http://chemcal.chemistry.unimelb.edu.au> and then follow the links to the Online PreLabs index page.

To check that your computer is set up appropriately, run the early screens of the ‘Using ChemCAL Online’ module on the ChemCAL Online home page. Your personal username and password for ChemCAL Online and Online PreLabs are the same as for your university email account. You can use the ‘Usernames and Passwords’ page in this module, with its link to the ITS Computing Assistance to activate your account, check your username or change your password.

The ChemCAL PreLabs exercises and questions must be completed before you can begin the experiment. Set aside the necessary time before your lab class to complete them.

At the completion of all the questions in the PreLabs module you will be issued with a receipt number provided you score at least 80% for the module (you may, without penalty, attempt the Prelab module multiple times). Please record your personal receipt number, together with the other details on the ChemCAL Prelabs receipt forms at the back of this manual and hand the form to your demonstrator at the start of the practical class. This is your record of completion of the PreLabs exercises and contributes 2/20 marks to your grade for each experiment in the laboratory program.

**If you have not completed or completed for the wrong PreLabs you will not normally be permitted to carry out the experiment and rescheduling for a “make-up” practical at another session is required.**

## Laboratory Reports

All reports must be written up in the laboratory, in a bound duplicate A4 notebook or by using the proforma result sheet provided at the back of this manual. **The practical report should be written IN PEN.** Exercises associated with the experiments should also be attempted and recorded into your lab notebook or the proforma result sheets.

All experimental observations should be recorded directly into your book or the proforma result sheets and NOT onto pieces of paper or other extraneous places (e.g. hands, skin and laboratory coat). The record of your work may be highly abbreviated, provided essential steps are recorded. Much of the work can be recorded in tabular form. The description should be such as to allow someone else to repeat your work. Therefore, your report must make clear what you have done and the results you have obtained. The names and structures of all organic compounds should be included and where relevant an equation should be written for each reaction. The formulas of inorganic reagents should be included. For the preparative experiments, equations and theoretical yield calculations should be done before the class.

Your practical report (original) and the report cover sheet for each experiment (provided at the back of the laboratory manual) must be stapled together and handed to your demonstrator before you leave the laboratory at the end of each class.

**LATE REPORTS OR REPORTS OUT OF LABORATORY HOURS WILL NOT BE ACCEPTED.** Your reports will be returned to you at your next practical session. Each report will be marked out of 20, with marks allocated for completion of the ChemCAL PreLabs module and for your work in the laboratory and written report.

*Students need to be aware that the reports should represent their own work. All calculations and graphing of data collected during the experiment must be completed individually by student. Copying from other students or using another student’s report as a “guide” is considered as cheating and will not be tolerated. Students failing to observe this rule will receive ZERO for the practical report.*

## Guidelines for Writing a Laboratory Report

Your reports should contain the following sections. A guideline to the contents of each section is provided.

### Aim:

In this section, you must briefly state (**in your own words if possible**) the aims of the experiment. (*The length should be no more than 1 to 2 sentences.*)

### **Experimental Method:**

In this section, you describe the method or procedure used by you in the experiment. In almost all experiments it is sufficient to note: "Refer to First Year Chemistry Laboratory Manual, Experiment X, pages 15 to 18."

For preparative organic chemistry experiments (Experiments 2, 3, 7 and 8), you will be required to summarise the procedure in the style of leading chemical journals, e.g. the Australian Journal of Chemistry. An example is given with respect to each preparative experiment. You will be required to include the structures of organic compounds in a reaction scheme.

*(The length of this section should be no longer than two simple paragraphs of 3 to 4 sentences each.)*

### **Results and Discussion:**

Your results must be presented in a clear and accurate manner, so that it is clear what you have done and what results you have obtained. All numerical data obtained by you should be presented in tabular form, with appropriate descriptive headings and correct number of significant figures. Units must also be included where appropriate.

Below are given examples of a titration table and a weighing table.

#### **Mass of sodium chloride used to prepare standard solution**

Mass weighed	Mass recorded	Uncertainty
sample tube + sodium chloride	26.4673 g	± 0.0001 g
emptied (but not rinsed) sample tube	25.9876 g	± 0.0001 g
mass of NaCl added to standard flask	0.4797 g	± 0.0002 g

[Note: The uncertainties can be used in error calculations later in the experiment if required.]

#### **Titration of hydrochloric acid with sodium hydroxide**

Titre	Initial Burette reading	Final Burette Reading	Volume NaOH added
1	0.05 ± 0.02 mL	10.47 ± 0.02 mL	10.42 ± 0.04 mL
2	10.47 ± 0.02 mL	20.02 ± 0.02 mL	9.55 ± 0.04 mL
3	20.02 ± 0.02 mL	31.50 ± 0.02 mL	11.48 ± 0.04 mL
4	31.50 ± 0.02 mL	41.85 ± 0.02 mL	10.35 ± 0.04 mL

You **must** clearly document your data. It is totally meaningless to just write a result and not explain what that result is, or how it relates to the experiment as whole. Essentially, if you look at an old report it must be obvious at a glance what the results are.

*Your discussion should be a short and concise paragraph with no more than 5 sentences referencing to your results obtained in the experiment.*

### **Conclusion:**

This section is, as the name implies, a summary of the main results obtained from the experiment. You must include the main numerical results (if any, with proper units and correct number of significant figures) in this section. *If your conclusion is more than 3 sentences, then you are too wordy.* If you have anything you want to write about in detail – do it in the discussion, **NOT** in the conclusion.

## Safety in the Laboratory

The University of Melbourne has adopted the internationally recognised systems: Safety MAP and Environmental Management System (ISO14001), to ensure a safe and environment-friendly workplace for all staff, students and visitors. As a student of the University you are responsible for adopting safe work and study practices and you are required to comply with all relevant University and School of Chemistry rules and procedures.

Detailed information on University policy and procedures is provided in the Environment, Health and Safety Manual at: <http://www.unimelb.edu.au/ehsm-new>.

The Laboratory Rules and Safe Work Procedures set out in this practical manual must be adhered to at all time and the direction of School staff and demonstrators must be followed. If you have any concerns about the safety or environmental impact of any activity in School of Chemistry practical classes, please raise them with the staff members in charge of the class. All injuries, accidents or incidents must be reported immediately to a staff member.

If you have an allergy or medical condition that you think may be affected by the chemicals, materials or procedures to be used in these practical classes, please fill in a “Medical Status-Notification for Laboratory Classes Form” (can be obtained from your demonstrator) and give it to the staff member in charge, so that any risk can be assessed and the work procedures modified accordingly.

**The following rules apply to all laboratories in the School of Chemistry:**

- **Safety glasses with side-shields conforming to Australian Standard (AS) 1337 must be worn at all time in the laboratory. Prescription spectacles with polycarbonate lenses are acceptable provided they are fitted with side shields (available from optometrists).**
- **The wearing of contact lenses in the lab is strongly discouraged. If their use is unavoidable, then splash resistant chemical goggles (not just safety glasses) are mandatory. A chemical splash to the eye while wearing contact lenses can trap chemicals between the lens and the cornea, making eye washing more difficult.**
- **A long-sleeved knee length laboratory coat.**
- **Sensible Laboratory Dress: Participants in laboratory practical classes are reminded to wear sensible dress appropriate for the tasks being performed on the day. When the experiments involve highly corrosive or toxic substances, the wearing of very short pants and skirts is discouraged.**
- **Shoes which enclose the entire feet (e.g. runners and boots) MUST BE WORN in the laboratory. Thongs, sandals, ballets flats and open style shoes are prohibited.**
- **Long hair must be fastened securely and NO hat or cap in laboratory.**
- **Mobile phones, headphones, eating, drinking, smoking and chewing of gum are not permitted in the laboratory.**
- **Pipetting by mouth is prohibited. Safety pipette-filters are provided and must always be used.**
- **Chemical Hygiene: Lab users are reminded to practice proper chemical hygiene at all time. Gloves are often used to protect the skin from chemical exposure. Users must avoid touching other surfaces (door handles, taps, pens, phones, faces, etc.) while wearing gloves to prevent the spread of chemical residues. Failure to practice safe chemical hygiene defeats the purpose of wearing protective gloves.**

In any chemical laboratory, there is always a potential danger from accidental splashing or spillage of chemicals, cuts from broken glass, burns from touching hot apparatus or splashing hot liquids and fire. Part of your training in practical chemistry is to learn the procedures that minimise these dangers and allow safe working conditions.

Specific safety precautions relating to specific experiments are detailed in this laboratory manual. All laboratory glassware must be handled with due care. Hot objects should be allowed to cool before handling. If it is essential to handle a hot object (e.g. pouring a hot liquid from a beaker) use a cloth, an insulated glove or tongs to hold the object. In the case of a major chemical spillage or fire, evacuation of the laboratory may be required.

## Risk Assessment

In addition to observing the general safety rules in the laboratory a RISK ASSESSMENT must be carried out before commencing any experimental procedure.

For each experiment in this manual, the result of the risk assessment process has been documented on the risk assessment sheet.

The risk assessment process requires an examination of materials and processes as shown below:

1. The available information on all substances to be encountered during the experimental procedure has been examined and reviewed.
2. An assessment has been carried out of the risk to health using any hazardous substance or process under the experimental conditions proposed.
3. A decision as to the level of risk associated with the experiment has been made and an appropriate procedure decided on. Specific hazards and precautions have been entered onto the risk assessment sheet.

Students must read the experimental procedure and the Risk Assessment and sign off before they undertake a specific experiment. There is a tear-off slip at the back of this manual for submitting your receipt number for the ChemCal Prelabs Module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## Risk Assessment Information

Detailed risk assessment information can be found in:

1. School of Chemistry Safety Manual  
<http://safety.chemistry.unimelb.edu.au/ehs-procedures-and-processes/>
2. The School of Chemistry Safety Website  
<http://safety.chemistry.unimelb.edu.au/ehs-procedures-and-processes/risk-assessments/>
3. The University of Melbourne Health & Safety Website  
<http://safety.unimelb.au/hazard-topics/chemical-management>

## Levels of Risk

### Category 3 - minimal risk

The procedure does not involve ionising radiation, potential photonic radiation or laser exposure or the handling of chemicals except for spectroscopic or other measurements on small samples of non-hazardous material.

### Category 2 - low risk

#### a) Fume hood recommended:

Procedures involving exposure to low-risk chemicals e.g. small scale reactions, solvent transfers, drying and extraction, chromatography, refluxing.

#### b) Fume hood or Schlenk line essential for the following:

Procedures involving the small-scale use of chemicals known to be mildly toxic, irritant, corrosive or allergenic.

Small quantities of non-commercial compounds not yet classified where no data are available.  
(Could assume low risk based on personal experience of similar compounds.)

Reaction volumes is restricted to less than 500 mL of flammable solvent or if distilling, to less than 2 L of flammable solvent.

### Category 1 - significant risk

Special precautions will be required depending on the nature of the hazard:

#### a) Chemical hazards.

Any procedures involving chemicals which are classified as:

- strongly corrosive
- irritant
- pungent
- carcinogenic
- mutagenic (*agent that changes the genetic material, usually DNA, of an organism*)
- teratogenic (*an agent causing malformations of an embryo or fetus*)
- oxidising
- pyrophoric (*a substance that will ignite spontaneously in air*)
- highly flammable
- react violently with water
- highly toxic
- stench
- non-commercial compounds where high risk is assumed based on personal experience (no data available)

- b) Procedures requiring special location or facilities.
- large scale reactions, particularly involving solvent distillation
  - high pressure reactions
  - reactions in sealed tubes
  - radioactivity above the safe levels specified in the Health and Safety (Radiation) Regulations
  - potentially explosive reactions
  - reactions in glass or other containers under high vacuum

## Accidents and First Aid

### Chemicals

All chemicals must be treated with respect. Some (e.g. concentrated acids and alkalis) are corrosive (to skin and clothing), while others (e.g. cyanides) are poisonous. Many, particularly organic chemicals (e.g. phenols and aromatic amines) are toxic by skin absorption or breathing of vapours. Organic solvents (e.g. hydrocarbons, ether and alcohols) are volatile and highly flammable and must not be used in the presence of an ignition source (e.g. an electric hotplate or flame).

When a chemical substance is used for the first time you should ask a staff member about its properties, or consult a reference book. In this course, you will find specific safety precautions and procedures detailed in the notes.

The following general precautions always apply:

- Chemicals should never be touched, tasted or smelled. Always handle toxic or foul smelling chemicals in a fume cupboard.
- Spillage on the skin: If any chemical in contact with the skin immediately wash with running water from a tap, shower hose or shower. Organic chemicals (e.g. toluene, phenols and aromatic amines) are readily absorbed through the skin. After washing with cold running water for several minutes, wash the exposed area with warm water and soap.
- Clothing contaminated with a chemical should be removed immediately, placed in a plastic bag and later washed separately from other clothing.
- In the case of a chemical splash to the head or upper torso immediately remove contaminated clothing and wash the affected area under a safety shower. Report to the demonstrator who may advise seeking medical attention.
- Chemicals in the eyes: An accident involving a chemical splash into the eye must be regarded as serious. The immediate treatment is to wash the eye with cold running water at the eye-wash station. Then report to the demonstrator, who may advise seeking medical attention. The use of approved safety glasses or goggles greatly reduces the risk of injury to the eyes.

### Glassware

Glass is a very hard material but brittle and breaks readily under stress or strain. Handle all laboratory glassware carefully. Do not use chipped or cracked glassware. In the case of breakage of laboratory glassware, which results in a cut, any particles or splinters of glass in the wound must be removed. All cuts must be reported to the demonstrator, who will inspect the wound and may advise seeking medical attention.

Broken glass should be carefully cleaned-up using only a brush and dustpan and properly disposed. Check with your demonstrator if you need advice on correct disposal of broken glass. Replacement glassware may be obtained by asking at the service desk of the Preparation Room.

## **Fire**

Many chemicals are flammable and must not be used when an ignition source (e.g. an electric hot plate or flame) is present. You are required to know the location of the nearest fire extinguisher, fire blanket and safety shower.

If a person's hair or clothing catches fire, try to smother the flames with a fire blanket or laboratory coat, rolling the person on the floor if necessary. The safety shower can also be used. To prevent the flames from reaching the head do not allow the person to remain standing. Report to the demonstrator and who may advise seeking medical attention for burns.

The following general precautions always apply:

- Do not use any type of chemical fire extinguisher on a person.
- Never heat an organic liquid in an open vessel (e.g. a beaker or flask) on an electric hotplate or over a flame.
- Never distil a liquid to dryness (the vessel may crack) and always use anti-bumping granules during a distillation or reflux operation.

## **Evacuation Procedure**

**When the alarm sounds:**

- **STOP what you are doing and turn off electricity and any gas taps.**
- **Follow ALL evacuation instructions given by Emergency Wardens.**
- **Move quickly from the laboratory using the nearest, safe emergency exit – follow signs and DO NOT USE LIFTS. It is advised to take only personal belongings.**
- **Emergency Wardens will direct you to the building exit and the emergency assembly point outside Chemistry West entrance (the Macfarland Court, F15).**
- **Remain at assembly point until advised by Emergency Wardens or Emergency Services.**

**You MAY ONLY RE-ENTER the building when the Chief Emergency Warden gives the all clear.**

## **Waste Materials**

All chemical waste should be disposed of in a safe and environmentally responsible manner.

Chemical waste, other than non-hazardous aqueous solutions which have been neutralised, must not be washed down the sink.

Specific care must be taken in disposing of some chemical reaction residues. Follow the specific instructions given in the laboratory manual.

Waste chemical bottles will be provided in the laboratory. Ensure that only the type of chemical waste noted on the label of the waste bottle is put into it.

## Care of Benches and Apparatus

Each student is responsible for the section of laboratory bench allotted to him/her. If your bench is left in an unacceptable state at the end of the laboratory practical session, marks may be deducted from your report.

- Any chemicals or water spilled on the bench must be cleaned-up immediately.
- Concentrated acid spills should first be neutralised with sodium bicarbonate and then washed away with cold water.
- Your working area and the communal areas (e.g. reagent benches and fume cupboards) must be kept clean and tidy at all time. Untidy work areas invite accidents!
- Chemicals spilled on the floor must be washed away immediately with water and mopped.
- Wet floors are slippery and hence dangerous.
- Broken glass must be swept up. Mops, brooms, dustpans and brushes are available from the Preparation Room.

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# First Year Chemistry Laboratory – Report Checklist

## Aim:

- 1 to 2 sentences
- Brief and concise in your own words with references to the aim of experiment given in lab manual

## Experimental Method (for Experiments 2, 3, 7 and 8 ONLY):

- 1 to 2 concise paragraphs
- No more than 3 to 4 sentences each
- No bullet points or numbering in “Experimental Method”
- Use past tense
- Use passive voice
- Maintain good English language and use full sentences
- Avoid mention of glassware and equipment unless specialised
- No detailed descriptions of recrystallization, separation and extraction processes
- Include mass, number of moles or concentration in brackets AFTER each reagent
- Always include necessary details such as the solvent used and temperature
- Use TRUE and KNOWN values (such as time, mass, moles, temperature and volume) that correspond with your personal experiment. Do not just copy what is written in lab manual
- Always include product description, melting point, yield and % yield at the end of Experimental Method
- Include drawn reaction schemes

## Results and Calculations (where appropriate):

- Be neat with report writing
- 1 to 2 sentences describing product
- Include melting point range for product
- Include workings for the calculations of product theoretical yield and %yield
- Include observations and interpretations of test tube reaction
- Include data tables with appropriate headings
- Include graphs with appropriate titles, correct scales, axes labels and best fit lines

- Include workings to calculations for possible consequential report marks
- Use correct units
- Use correct significant figures
- Include calculations of errors when required

**Discussion (where appropriate):**

- 1 short and concise paragraph of no more than 5 sentences
- Comment on low product yield using known reason such as spillage, breakage, procedural mistake, product solubility and technique used
- Comment on purity of your product with references to:
  - Melting point range such as too wide or out of known range
  - Evidence of impure product from test tube reactions
  - The R<sub>f</sub> values determined using thin film chromatography
- Comment on known reasons affecting the accuracy and precision of practical results such as:
  - Accurate endpoint identification in titration
  - Errors involved in pipetting and burette readings
  - Errors involved in sample weighing, solution preparation and dilution
  - Errors involved in the preparation of ice-water baths of known temperatures
- Answer all questions and discussions given in lab manual
  - Precise answers of no more than 2 to 3 sentences each
  - Precise discussions of no more than 2 to 3 sentences each

**Conclusion:**

- 1 to 2 sentences
- Conclusion should be answering the aims and to restate important values such as:
  - The yield and purity of product
  - The concentration of measured solution
  - The verification of a given equation

# ANALYTICAL TECHNIQUES AND METHODS:

**1**

## AIMS OF THE EXPERIMENT

- To gain experience in the use of balances and important volumetric glassware.
- To perform a simple acid-base titration and become proficient with titrimetric techniques.

**Note:**

You will require a basic scientific calculator as well as your laboratory notebook. It is a good idea to bring these to all practical classes.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Concentrations of solutions and volumetric analysis: Section 1.5, pages 34 – 38;
  - Measurement and units: Section 1.2, pages 6 – 11;
  - Significant figures and rounding of decimals: pages 1308–1309.
- This laboratory manual:
  - Guidelines for Writing a Laboratory Report: page 11;
  - Safety in the Laboratory: page 13;
  - Techniques and instrumentation: from page 127;
  - Treatment of errors: from page 140.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## INTRODUCTION

For this experiment:

- Students work individually
- A Results Sheet is provided at the back of this manual (page 149)

Mastery of the techniques and instruments of experimental chemistry is essential to success in chemistry and the allied sciences - it should be your aim in this course. The use of instrumentation and the execution of analytical and synthetic procedures are daunting to some but apparently trivial to others. One thing is for sure, skill and confidence are attained only with practice - their rapid attainment will greatly enhance your performance in this course. Many other experiments you carry out this year will require analytical and titrimetric techniques. This introductory exercise is designed to enhance your skills in these areas so that you will be able to confidently approach these in later experiments.

### **Mass, Volume and Titration**

#### **(i) Mass**

The measurement of mass is one of the most important aspects of chemistry. It allows us, through the mole concept, to 'count' atoms or molecules and to thereby control and exploit stoichiometry in synthesis and analysis.

Mass ( $m$ ), a measure of the quantity or amount of matter in a sample, should not be confused with weight which is given by  $W = ma$  where  $a$  is the gravitational acceleration (strictly speaking weight is a force with a unit of kilogram-force, kgf). Unfortunately, it is common to speak of weighing a sample when determining its mass.

Balances are precision mechanical and electronic instruments designed and employed to measure mass. In this experiment, you will learn and gain practice in the use of a variety of balances. Your demonstrator will explain the correct method for the use of balances to you (see page 127). Remember to treat the balances with great care.

#### **(ii) Volume**

Quantitative analytical procedures depend on the accurate measurement of volume as well as mass. Therefore, special volumetric glassware has been designed to accurately control the volume of solution manipulated during analyses. This glassware, *e.g.* pipettes, burettes and volumetric flasks, should be scrupulously clean and handled with care and skill. Correct methods for the use of these items of glassware will be explained to you by your demonstrator (from page 129).

In this experiment, volumetric glassware will be employed in an acid-base titration.

Table 1 shows typical uncertainties for some laboratory instruments. These uncertainties indicate the range about the measured value likely to contain the true value, if there are no systematic errors or mistakes made during the measurement.

For example, a mass of  $10.32 \pm 0.01$  g, measured using a top loading balance, will lie between 10.31 g and 10.33 g, if the measurement is made by a skilled operator, using a well-maintained balance, under optimum conditions.

**Table 1.1 Operating range and precision of common analytical apparatus used.**

Instrument	Quantity	Uncertainty
Top loading balance	0 – 300 g	± 0.01 g
Analytical balance	0 – 120 g	± 0.0001 g
Beaker	200 mL	± 10.0 mL
Pipette	10 mL	± 0.01 mL
Burette	50 mL	± 0.02 mL
Volumetric flask	100 mL	± 0.1 mL
Measuring cylinder	100 mL	± 0.5 mL

These figures are useful for estimating the uncertainty in a single derived result from experimental measurements.

See from page 140 for a more detailed discussion of uncertainty and the treatment of experimental data.

## REPORT WRITING

Part of your training is to develop your ability to write clear, accurate and concise reports. This will be of significant future benefit to you. The aim of a report is for someone who has not done the experiment to be able to look at your report and immediately understand what you have done and what results you have obtained.

To make results clearer and more understandable you are encouraged to present them in a clear and easily legible manner. The use of tables for multiple data such as for weighing and titrations is highly recommended such that the reader is not forced to hunt through your report to see what you have done (see [Guidelines for Writing a Laboratory Report](#), page 11, for a guide).

For this experiment a result sheet is provided for you to record your experimental results. This should provide you with a model of how to record your results in future experiments.

## SAFETY



### Safety Warning:

**Caution: Take care with applying pipette filler onto the pipette.**

NaOH is a strong base. HCl is a strong acid. Concentrated solutions of both are corrosive and give off irritating vapours. Avoid skin contact at all time.

If spillage occurs, use water to dilute and wash away.

## Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the back of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Calibrating a pipette

*Note: When weighing the flask, it is important that the outside (including bottom) of the flask is dry, similarly the balance pan should also be dry. Why?*



Top-loading  
balance

1. Add 100 mL of distilled water to a small beaker.
2. Weigh an empty 50 mL or 100 mL conical flask on a digital top-loading balance (see page 127).
3. Pipette (see page 130) into the flask 10 mL aliquot of distilled water from the small beaker (don't forget to allow the correct draining time) and reweigh the flask.
4. Pipette three further aliquots of water into the flask, weighing after each addition.
5. Calculate the mass of each aliquot delivered from the pipette. Calculate the mean mass delivered (see page 140). The variation in your weights should be less than 0.3%. Repeat the process if your results are not reproducible.



Pipette

$$\% \text{ Variation} = 100 \times \frac{(\text{Difference between the highest and lowest mass})}{\text{mean mass}}$$

6. Convert this mass to a volume by using the appropriate density (your demonstrator has a table of water density as a function of temperature). Quote your answer to the appropriate number of significant figures.
7. Compare the volume delivered by your pipette with the manufacturer's volume.

### Part B: Volumetric analysis

In this analysis, you are provided with aqueous solutions of sodium hydroxide and hydrochloric acid. The concentration of the sodium hydroxide solution is accurately known (available from demonstrator) and you are asked to determine the concentration of the hydrochloric acid.

However, the concentration of the sodium hydroxide is approximately ten (10) times greater than the concentration of the hydrochloric acid solution. This means that 10 mL aliquot of sodium hydroxide would require approximately 100 mL of hydrochloric acid solution per titre for neutralisation. On the other hand, if you are titrated 10 mL aliquot of hydrochloric acid solution then you would require approximately 1 mL of sodium hydroxide solution per titre.

Since you have only a limited supply of hydrochloric acid (*ca.* 80 – 100 mL) the first method is not possible and the second method is not accurate enough (i.e. it is harder to deliver small volumes as accurately as larger volumes. **Why?**). The alternatives are to either concentrate the hydrochloric acid solution (very time consuming and impossible to control with any accuracy) or dilute the sodium hydroxide solution.

Diluting the sodium hydroxide solution is done simply by putting an aliquot of the solution into a volumetric flask and topping up the flask to the mark with distilled water. Since both the volume delivered by the pipette (determined in Part A above) and the volume of volumetric flask are known, you can accurately calculate the concentration of the diluted solution. Hence 10 mL aliquot of the

hydrochloric acid solution will require approximately 10 mL of the diluted sodium hydroxide solution for neutralization.

1. Dispense, via zippettes, the solutions of sodium hydroxide and hydrochloric acid into separate, clean and DRY beakers.
2. Pipette a 10.00 mL aliquot of the standardized sodium hydroxide solution into your 100 mL volumetric flask (see page 130) which has been pre-rinsed with distilled water.
3. Add distilled water to your volumetric flask and make up the solution to the mark. Add the last 1 – 2 cm with a plastic dropping pipette (**why?**), mix the solution thoroughly.
4. Calculate the concentration of sodium hydroxide in the volumetric flask.  
**REMEMBER to use the pipette volume determined in Part A, not the nominal 10.00 mL.**
5. Rinse and fill the burette with the diluted sodium hydroxide solution.



Volumetric Flask

- Ensure the funnel used for filling is removed from the burette and the tip of burette is filled with solution when the initial reading is taken (why?).*
6. Use your pipette to transfer a 10.00 mL aliquot of the hydrochloric acid (HCl) solution to a 250 mL conical flask which has been pre-rinsed with distilled water.
  7. Titrate HCl (see page 131) with the diluted sodium hydroxide solution from burette using phenolphthalein as indicator (1 – 2 drops).



Volumetric  
techniques

- For burette readings, record values to two decimal places.**
8. The endpoint is reached when the colourless solution becomes a faint pink colour and the colour persists for more than a few seconds. After an extended period of times the solution can return to its colourless state due to absorption of carbon dioxide from the atmosphere.
  9. You should aim to get at least three titres that are concordant (concordant titres are those which are within 0.20 mL of each other).

Titres which can be identified as being in error, such as a first run in which the endpoint has been overshot, may be rejected when calculating the mean titre. However, if you know of no reason why a value should be wrong it should be retained in the calculation.

If you are unsure at this point, check with your demonstrator. Remember that an important objective of this exercise is to improve your practical skills, so seek advice if you are struggling to obtain concordant titres.

10. Calculate the concentration of the HCl including error (see from page 140). Quote your answer to the appropriate number of significant figures.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input checked="" type="checkbox"/> Pungent  | <input type="checkbox"/> Stench      |
| <input type="checkbox"/> Toxic  | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input checked="" type="checkbox"/> Minimal risk   |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

# THE PREPARATION OF PARACETAMOL:

**2**

## Preparation of 4-ACETAMIDOPHENOL

### AIMS OF THE EXPERIMENT

- To synthesise the drug paracetamol through acetylation of 4-aminophenol.
- To determine the melting point of the prepared paracetamol sample.

### READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - The yield of a reaction: pages 24 – 25;
  - Nucleophilic acyl substitution reactions of acid anhydrides: pages 1116 – 1118.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:
  - Stoichiometry, limiting reagents and percentage yield: page 93 – 99;
  - Nucleophilic acyl substitution: pages 1026 – 1027.
- Techniques and Instrumentation section of this laboratory manual:
  - Top-loading balances: page 127;
  - Laboratory equipment and glassware: page 129;
  - Filtration: page 132;
  - Purification of compounds: pages 133 – 136.

### PRE-LAB QUESTIONS

There is a compulsory ChemCAL PreLab module which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

### INTRODUCTION

For this experiment:

- Students work individually and require a basic scientific calculator
- Part A and Part B are to be written up in “Experimental Method” of report (refer to page 30)

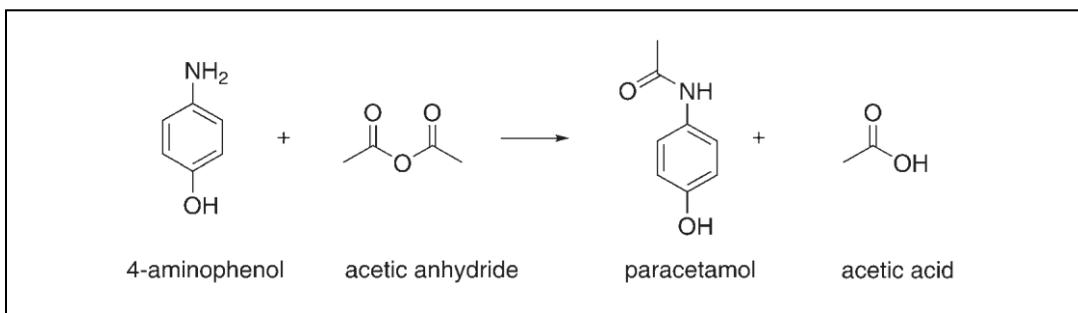
4-Acetamidophenol (acetaminophen), or paracetamol (para-acetamidophenol) as it is widely known, is a pain relieving and fever reducing drug. It is used to give temporary pain relief from minor complaints such as headache, muscular pains and backache.

The pharmacological effects of paracetamol were discovered by accident. In the late 19th century it was found in urine as a metabolite of two other less effective analgesics, acetanilide and phenacetin. However, it was not until the 1940’s that its analgesic effects were fully appreciated. Surprisingly, for such a simple molecular structure, it was not until 1955 that paracetamol was first marketed as a drug, initially as Tylenol. In 1956 it was marketed in the UK as Panadol, initially as a prescription

product. In 1963, it was turned to an over-the-counter drug, and its popularity as a pain- reliever grew rapidly.

Nowadays paracetamol is used in combination with other pain relievers such as codeine, dihydrocodeine and dextropropoxyphene (*e.g.* Panadeine), as well as oral decongestants in a range of products to relieve the symptoms of the common cold, flu and sinusitis.

Paracetamol can be prepared by acylation of 4-aminophenol with acetic anhydride as shown in Figure 2.1. This reaction constitutes the final step in the industrial synthesis of paracetamol.



**Figure 2.1** Reaction scheme for synthesis of paracetamol.

## SAFETY



### Safety warning;

4-Aminophenol is harmful if swallowed or inhaled. Prevent contact with the eyes (safety glasses) and skin (gloves). If spilt on the skin, wash off with copious amounts of cold water. 4-Aminophenol is very toxic to aquatic organisms and must be disposed of into the appropriately labelled waste container.

**Caution: The steam and hot surfaces of steam baths can cause burns.**

Acetic anhydride is an irritant and may burn the eyes and skin. Avoid breathing the vapour and avoid contact with the skin. If acetic anhydride is spilt on the skin, wash off with copious amounts of cold water.

### Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the back of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Preparation of Crude Product

- It is important to ensure that clean conical flasks are used for the preparation and recrystallization of paracetamol. Thoroughly wash both the conical flasks with water, followed by rinsing with small amount of distilled water and finally dry using paper towel before the start of the paracetamol preparation.
- Weigh 4-aminophenol (1.00 g, 0.00920 moles) using weighing paper and add into a clean small conical flask. Add 5 mL of water followed by acetic anhydride (1.2 mL, 0.013 moles) and heat the mixture on a hot water bath (see page 129) in the fume hood for 15 minutes, swirling frequently.



Top-loading  
balance

3. After heating the mixture, cool the conical flask and its contents in an ice-water bath. If no crystals appear within 5 minutes scratch the interior wall of the flask with a spatula until crystals begin to form (any large lumps of material present should be broken up with the spatula).
4. Collect the product on a Hirsch funnel (see page 132) by vacuum filtration. Wash the crystals with  $3 \times 2$  mL amounts of chilled water to remove acetic acid from the surface of the crystals.  
To partially dry the crude paracetamol, allow air to be drawn through the solid whilst pressing with a spatula.
5. Transfer the crude product to a clean 100 mL conical flask ready for recrystallisation.



Hirsch vacuum  
filtration

#### **Part B: Recrystallisation and melting point determination**

6. Add *minimum* amount of hot water (start off by adding a 4 to 5 mL portion) to fully dissolve the crude product in conical flask. The solution in the flask should be kept warm by heating on steam bath. If the crude product does not dissolve, add more hot water incrementally in 0.5 mL portion and continue the heating until all solid is dissolved (no more than 8 to 10 mL of hot water in total, see pages 133 – 134).
7. Once all the crude product has dissolved allow the solution to cool to room temperature undisturbed. When crystals start to form, complete the crystallization by cooling in an ice-water bath. If no crystals have formed after 10 minutes, ask your demonstrator for assistance.
8. Collect the recrystallised product on a Hirsch funnel. Wash the crystals with  $3 \times 2$  mL amounts of chilled water and then dry at the pump by allowing air to be drawn through the crystals for at least 5 minutes.
9. It is important to remove as much liquid as possible from the crystals by this method. Complete the drying of the crystals on a watch glass over a steam bath.
10. Whilst your product is drying, place a small amount on filter paper (see demonstrator) and “flash dry” on the edge of a steam bath. Set aside part of this small sample for testing in Part C and use the remainder to determine the melting point of your sample.
11. When the remainder of your product is dry, transfer it to a pre-weighed sample bag and re-weigh to determine the yield.
12. Calculate the percentage yield of paracetamol, based on the amount of 4-aminophenol you used.



MP determination

13. Submit the fully labelled (see Figure 2.2) sample bag to your demonstrator.

<b>STUDENT NAME (YOUR NAME)</b>
Date, Group Number, Day
4-Acetamidophenol (recrystallised) m.p. range
(e.g. m.p. = 129-130 °C) Percentage yield

**Figure 2.2 Sample label.**

### Part C: Colour-based spot test for organic functional groups

4-Aminophenol contains two functional groups: an –NH<sub>2</sub> group and an –OH group attached directly to the aromatic ring. Paracetamol is an amide, formed by the reaction of acetic anhydride and the –NH<sub>2</sub> group of 4-aminophenol. 4-Aminophenol gives an intense colour when treated with iron(III) chloride in dilute aqueous or ethanolic solutions. Hence this test can be used to confirm that 4-aminophenol has been converted to paracetamol.

14. Your demonstrator will supply you with test tubes.
15. In two separate test tubes add a few crystals of:  
(i) your sample of paracetamol and (ii) 4-aminophenol in 2 – 3 mL of ethanol.
16. To each of these solutions add two drops of 0.2 M iron(III) chloride solution and note any change of colour or solid formation.
17. *Comment on the test results and what they indicate about the purity of your product.*

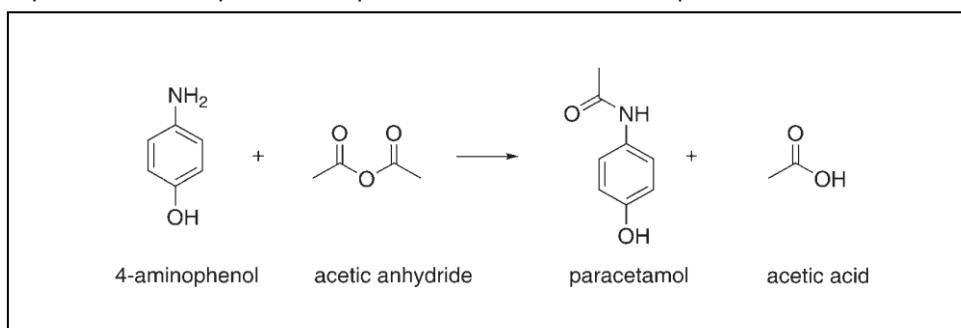
### WRITING UP OF PREPARATIVE EXPERIMENTS

Your report on the preparation of paracetamol, and any other preparative experiment, should follow the style used by leading chemical journals. Particularly, the report needs to be CONCISE:

1. The “Aim” should be 1 to 2 sentences
2. The “Experimental Method” should be no more than TWO short paragraphs of 3 to 4 sentences each:
  - Written in past tense and passive voice (“acetic anhydride was added to a solution of 4-aminophenol” NOT “I added acetic anhydride to a solution of 4-aminophenol”)
  - An equipment and glassware free description of the process (“the product was collected by vacuum filtration” NOT “the product was filtered using a Buchner funnel connected to the laboratory vacuum outlet”)
  - Included the structures of organic compounds in the reaction scheme
3. The “Result” should include all obtained experimental results and calculations
4. The “Discussion” should be ONE short paragraph of no more than 5 sentences
5. The “Conclusion” should be 1 to 2 sentences

For example:

#### Experiment 2: Preparation of paracetamol from 4-aminophenol



Acetic anhydride (1.2 mL, n mmol) was added to a solution of 4-aminophenol (1.00 g, y mmol) dissolved in water (5 mL) and the mixture was heated at 60–70°C for 15 min with frequent swirling. The solution was cooled on ice and the resultant precipitate was collected by vacuum filtration and recrystallized from water to give colourless plate-like crystals (0.89 g, x% yield), m.p. XX-XX °C

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |   |
|---|--|--|---|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input checked="" type="checkbox"/> Pungent  | <input type="checkbox"/> Stench                 |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input checked="" type="checkbox"/> Mutagenic  | <input checked="" type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic              |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |   |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |   |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input checked="" type="checkbox"/> Other: Exposure to hot surfaces (steam bath)            |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- |   |
|---|
| <input type="checkbox"/> 3 Minimal risk                                     |
| <input checked="" type="checkbox"/> 2a Low risk (Fume hood recommended)     |
| <input type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential)     |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)              |
| <input type="checkbox"/> 1b Significant risk (Special location or facility) |

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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# ISOLATION OF A NATURAL PRODUCT – II:

**3**

Including an examination of the SOLUBILITIES OF ORGANIC COMPOUNDS

## AIMS OF THE EXPERIMENT

- To isolate the yellow pigment lomatiol from seeds of the plant genus *Lomatia*, using the techniques of Soxhlet and liquid extraction.
- To explore some of the chemistry of lomatiol.
- To establish some of the principles of the solubility of organic substances.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - The yield of a reaction: pages 24 – 25;
  - Carbon frameworks and functional groups: pages 77 – 78;
  - Solvation: pages 305 – 306.
- Techniques and Instrumentation section of the laboratory manual:
  - Top-loading and analytical balances: page 127 – 128;
  - Laboratory equipment and glassware: page 129;
  - Filtration: page 132;
  - Solvent extraction using separating funnel: pages 137;
  - Soxhlet extraction: pages 139.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL PreLabs module which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## SAFETY



### Safety warning:

Diethyl ether, methanol, ethyl acetate and hexane are volatile and flammable liquids. Avoid contact with flames or electrical equipment. Many organic chemicals are toxic by skin absorption or breathing of vapours. Avoid breathing the vapour and avoid contact with the skin.

**it is important that you treat ALL chemical compounds as hazardous. Wear gloves at all time. If there is any spillage of chemicals on gloves, remove immediately and replace with new gloves.**

**Caution: The steam and hot surfaces of steam baths can cause burns.**

## Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLab module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## INTRODUCTION

For this experiment:

- Students work in pairs and the report is written individually in own words.
- You will require a basic scientific calculator.
- Part A and Part B are to be written as a report (refer to WRITING UP OF PREPARATIVE EXPERIMENTS, page 30).
- A Results Sheet is provided at the back of the manual for Part C (page 153).

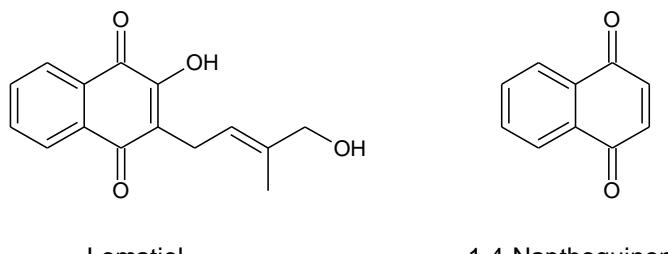
## EXPERIMENTAL PROCEDURE

### Introduction: The Isolation of a Natural Product

Much of Australia's organic chemical research has been and is still involved with natural product chemistry. This involves the extraction and identification (characterisation) of new substances from biological sources such as plants, marine organisms and animals. The total synthesis of the substances (in the laboratory) and their testing and application as, for example, drugs (antibiotics, antivirals, analgesics), biological control agents and growth regulators, follows their initial identification. This type of research has contributed immensely to organic chemistry as we know it today.

Trees belonging to the *Lomatia* genus in Australia have a bright yellow pigment on their seeds. The role of the pigment is open to speculation; it may for example, exhibit fungicidal properties thus protecting the seed from fungal attack.

Spectroscopic and chemical techniques have been employed to establish the structure of the pigment (Figure 3.1), which is called lomatiol. This compound is one of a series of natural products, the structures of which are based on the 1,4-naphthoquinone skeleton, shown below.



**Figure 3.1** Chemical structure of Lomatiol (a natural product) and its Naphthoquinone skeleton.

## Part A: Isolation of Lomatiol

- A1. The *Lomatia* seeds are supplied already ground. Weigh  $1.0 \pm 0.01$  g of the material onto a weighing paper.

• Record the mass and then place the powder into a Soxhlet thimble.

- A2. Extract the pigment in a Soxhlet extractor (your demonstrator will explain how to do this and see page 139) using ether (50 mL) as solvent.  
**(Notes.** Do not forget to add 3 to 4 boiling chips).

Clean DRY round-bottomed flask and Soxhlet extractor are supplied for the extraction.

- A3. When the extraction is complete (about 10 cycles is usually sufficient) transfer the cooled ether extract to a separatory funnel.

*Place the empty round-bottomed flask and Soxhlet extractor directly into the labelled containers in the fume hoods. DO NOT clean.*

- A4. Use a separating funnel (see page 137) to extract the ether solution with 2 x 10 mL of 10% NaHCO<sub>3</sub> solution as shown by your demonstrator.

Collect the first 10 mL aliquot of NaHCO<sub>3</sub> solution in 100 mL conical flask and extract the ether solution with a second 10 mL aliquot of NaHCO<sub>3</sub> solution. Combine the second NaHCO<sub>3</sub> extract with the first.



Soxhlet extraction



Separating funnel

*Immediately dispose of the ether residue into the ether residue jars in the fume hoods. DO NOT clean the separatory funnel.*

- A5. Place 2 mL of the NaHCO<sub>3</sub> extracts into 10 mL measuring cylinder and set this aside **for Part B**.

- A6. Carefully acidify the remainder of the extract with dilute HCl drop-wise. This acidification process is self-indicating in that when sufficient aqueous acid has been added the characteristic red colour associated with the conjugate base of lomatiol will be discharged.

Hence add only enough acid to achieve a yellow solution (make sure that as each portion of dilute HCl is added the contents of the flask are swirled thoroughly).

- A7. When the acidification step is complete, cool the conical flask and its contents on an ice-water bath and only scratch the interior wall of the flask with a glass rod if no crystals appear within five minutes.

If the precipitate is finely dispersed in the solution it may be coagulated by the addition of a small quantity (*ca.* 1 ml) of aqueous sodium chloride (see demonstrator).

- A8. Filter the resulting precipitates using a Hirsch funnel (see page 132) then dry the product on a watch glass over a steam-bath.



Hirsch vacuum filtration

- A9. Accurately weigh a clean, dry plastic bag using the analytical balance and transfer the solid to the bag. Re-weigh the plastic bag with the sample and record your yield.

• Calculate the percentage mass of lomatiol contained in *Lomatia* seeds.



Analytical balance

A10. Label the sample bag (see Figure 2.2) and hand it to your demonstrator. The crude product you have isolated would normally be purified by recrystallisation or sublimation.

### Part B: The Redox Reaction of Lomatiol

B1. Take 2 mL aliquot of the original basic  $\text{NaHCO}_3$  extract containing lomatiol that was set aside (from Part A) and treat it with freshly prepared dilute aqueous sodium hydrosulphite solution ( $\text{Na}_2\text{S}_2\text{O}_4$ ) drop-wise just sufficient to discharge the deep red colour.

B2. Leave aside to stand for some time.

- Interpret the colour changes you observe using the full equations for the reactions given by your demonstrator.

### Introduction: The Solubilities of Organic Compounds

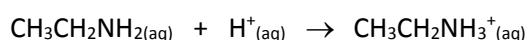
Dissolution is the process by which a solute (a solid, liquid or gas) becomes dispersed in a solvent (generally a liquid) to produce a homogeneous but physically separable (e.g. by solvent evaporation) mixture.

The solubility of a substance is defined as the maximum amount of solute that will dissolve in the solvent under specified conditions of temperature and pressure. The solubility of a substance depends on the degree of solvation, i.e. the extent of physical interaction between solvent and solute, and the magnitude of the forces in the solid state. The extent of solvation will be greatly affected by ionisation of the molecule by acid/base reactions.

Substances are generally soluble in like solvents. The solubility of molecular organic compounds depends primarily upon the polarity of the solute molecule relative to its size and the polarity of the solvent molecules. Hence short chain alcohols tend to be soluble in water (polar solvent) whereas long chain alcohols (e.g. decanol) are insoluble in water but soluble in non-polar solvent (e.g. heptane).

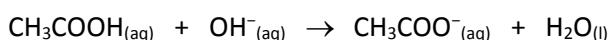
If a substance is soluble in acid, this is likely to indicate that there is a basic group in the molecule (e.g. an amine) able to accept a proton and thereby give the molecule a positive charge.

For example, when ethylamine is soluble in acid to give ethylammonium ion:



Conversely, if the substance is soluble in base, then this likely indicates that the molecule can be deprotonated to give a molecule with a negative charge.

For example, when acetic acid is soluble in base to give negatively charged acetate ion:



## Part C: Solubilities of Organic Compound

**Note:** It is essential that clean test tubes are used for each of these tests and clean spatulas are used for adding solid compounds. Any micro test tubes intend for heptane as solvent are rinsed with small amount of ethanol prior to use for tests.

- C1. Test the solubilities (at room temperature) of the following list of organic compounds in various solvents:
  - a) methanol
  - b) 2-octanol
  - c) 1-bromobutane
  - d) benzoic acid
  - e) propylamine
- C2. Use 0.10 mL (2–3 drops) or 5 mg (half a rice grain) of the test compound in approximately 1 mL of water. Should the compound do not dissolve immediately, mix it thoroughly using a plastic dropping pipette.
- C3. If the organic compound is **soluble in water**, place a drop of the solution onto the litmus paper using a plastic dropping pipette. Test the solution with both RED and BLUE litmus paper and note the colour change if any.

RED litmus turns BLUE in basic solution

BLUE litmus turns RED in acidic solution

- C4. Repeat above Step C2 for test compound in approximately 1 mL of 2 M HCl.
- C5. Repeat above Step C2 for test compound in approximately 1 mL of 2 M NaOH.
- C6. Repeat above Step C2 for test compound in approximately 1 mL of heptane.
- C7. Record your observations and comments on the solubilities of each compound on the Results Sheet provided (page 153 of this manual). Draw the structure and identify the functional group of all compounds used next to the appropriate name on the Results Sheet.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input checked="" type="checkbox"/> Other: Exposure to hot surfaces (steam bath)            |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

# VAPOUR PRESSURE OF A VOLATILE LIQUID:

**4**

## AIMS OF THE EXPERIMENT

- To use a vacuum line to measure the changes in equilibrium vapour pressure of a volatile liquid with temperature.
- To determine a value of the latent heat of vapourisation of a liquid.
- To gain experience in the safe handling of gases and refrigerants (ice and liquid nitrogen) and the operation of a vacuum line.

**Note:**

You will require a calculator (basic scientific), graph paper (2 mm ruling), a sharp pencil and a 30 cm ruler as well as your laboratory notebook.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - The gas laws and ideal gas equation: Sections 8.1 – 8.2, pages 345 – 352;
  - Vapour pressure: Section 17.1, pages 766 – 768;
  - Effects of temperature and pressure on phase transitions: Section 17.2, pages 776 – 781.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

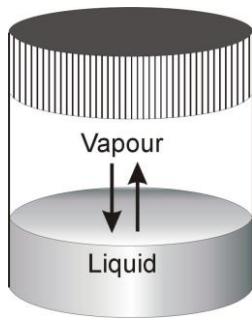
The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## INTRODUCTION

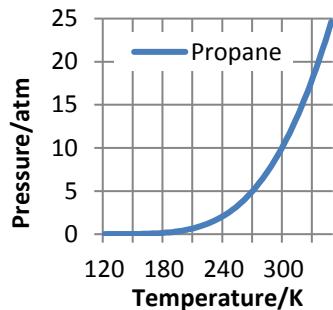
For this experiment:

- Students work in pairs
- A Results Sheet is provided at the back of this manual (page 155)
- The report is written individually in own words
- All calculations and graphing of data collected during the experiment must be completed individually by students

Evaporation takes place when molecules near the surface of a liquid have sufficient energy to escape from the attractive forces within the liquid and become gaseous molecules. The gas phase associated with a liquid is known as the vapour and the molecules in the gaseous phase exert a vapour pressure.



**Figure 4.1:** Dynamic equilibrium between a liquid and its vapour in a closed container



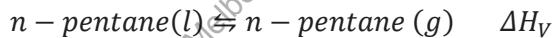
**Figure 4.2:** Saturated vapour pressure of a liquid as a function of temperature

At a specific temperature, in a closed container, as molecules continue to evaporate, the vapour pressure will rise but at the same time some of the gaseous molecules will strike the surface of the liquid and be recaptured. Eventually a stage will be reached where the number of particles leaving the liquid is exactly equal to the number returning. (*i.e.* a dynamic equilibrium is set up, see Fig. 4.1).

At this point, the rate of evaporation of the liquid is equal to the rate of condensation of the vapour and hence the number of particles in the vapour and the vapour pressure will remain constant. The vapour pressure at this point is called the *saturated vapour pressure*.

As the temperature ( $T$ ) of the liquid is increased, the saturated vapour pressure ( $\rho$ ) will increase as shown in Fig. 4.2. The liquid is said to be boiling when the vapour pressure is equal to the pressure of its environment.

For example, the process of evaporation for *n*-pentane, as for other liquids, is endothermic and we can write



where  $\Delta H_V$  (called the *molar latent heat of vapourisation*) is the enthalpy change when one mole of the liquid is evaporated.

The variation of  $\rho$  with  $T$  (Fig. 4.2) is given by the following equation:

$$\frac{d(\ln\rho)}{dT} = \frac{\Delta H_V}{RT^2} \quad (4.1)$$

where  $T$  is the absolute temperature,  $\Delta H_V$  is the molar latent heat of evaporation and  $R$  is the universal gas constant ( $8.314 \text{ Jmol}^{-1}\text{K}^{-1}$ ).

This relationship can be expressed mathematically in a linear form as:

$$\ln\rho = -\frac{\Delta H_V}{R} \left(\frac{1}{T}\right) + C \quad (4.2)$$

where  $C$  is a constant. Plotting  $\ln\rho$  versus  $1/T$ , over a limited range of temperature (for which  $\Delta H_V$  is constant), gives a straight line with gradient equal to  $-\Delta H_V/R$ . This allows  $\Delta H_V$  to be determined.

Integration of the above equation 4.1 gives

$$\ln\rho_2 - \ln\rho_1 = -\left(\frac{\Delta H_V}{R}\right) \cdot \left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad (4.3)$$

where  $\rho_1$  and  $\rho_2$  are the vapour pressures at absolute temperatures  $T_1$  and  $T_2$  respectively. If  $\Delta H_V$  is known, the vapour pressure ( $\rho_2$ ) at any other temperature ( $T_2$ ) or the temperature at any other pressure can be calculated.

## SAFETY

### Safety Warning:



Liquid N<sub>2</sub> is at a temperature of 77 K. It should not be brought into contact with the skin.

Liquid N<sub>2</sub> burns require first aid treatment.

The safety shield around the sample bulb is not to be removed under any circumstances.

### Disposal of Chemical Wastes:

Disposal of Liquid Nitrogen: Do not pour liquid nitrogen into sinks, disposal bottles, or on the floor. Leave it in the Dewar flask to evaporate.

There are no residues to be disposed of in this experiment. The very small amount of volatile liquid, which is lost from the vacuum line during this experiment, is safely vented to the outside of the building via a lower pressure extraction system.

### Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Vacuum line: Introduction

Gases can only be handled safely and satisfactorily using a vacuum line: this usually consists of a set of linked tubes (manifolds), storage bulbs, and pressure meters that can be thoroughly evacuated with a vacuum pump. Pure gases of various sorts can then be admitted to the line and their properties, free from the effects of any atmospheric contamination, can be studied.

Vacuum lines are usually made either of metal and/or glass. The vacuum pump in the present apparatus is a rotary oil pump, which is capable of reducing the air pressure to between one ten-thousandth and one hundred-thousandth of an atmosphere.

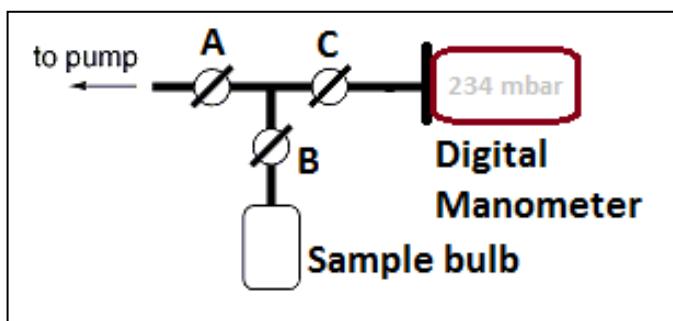


Figure 4.3: Schematic diagram of the vacuum line.

Pressures in vacuum lines are often expressed in torr: 1 torr = 1 mm Hg or 0.0013 atm or 1.33 mbar. The pressure in your vacuum line when the pump is working properly should be between 0.133 mbar and 0.013 mbar. The pressure in the line will be indicated by the digital manometer; it should therefore give a pressure reading close to 1 mbar.

A schematic diagram of the vacuum line is shown in Figure 4.3. Your demonstrator will familiarise you with the correct procedure for operating the vacuum line.

An important aid to handling gases is liquid nitrogen, which boils in air at a temperature of  $-196^{\circ}\text{C}$ , or 77 K. Most common substances are solids at this temperature and have very low vapour pressures. Liquid nitrogen is used as a refrigerant to freeze volatile substances (such as n-pentane) so that they condense leaving only air which can then be removed by pumping.

Before the start of the session, the liquid sample will have previously been degassed using the ‘freeze-pump-thaw’ method. This is necessary to remove all the O<sub>2</sub> and N<sub>2</sub> that were dissolved in the liquid sample. The volatile liquid contained in the sample bulb will be positioned on the vacuum line.

### ***Preparation of ice-water mixtures for vapour pressure measurements***

1. Add 250 mL of tap water to the glass beaker.
2. Add just enough ice to the 250 mL of tap water in glass beaker until the desired temperature (for example the  $2^{\circ}\text{C}$  ice-water mixture) is achieved. In each case, the temperature should be within half a degree of that specified.
3. Empty this ice-water mixture into the thermo-flask.  
Add metal stirrer and digital thermometer to the thermo-flask.
4. The temperature of the ice-water mixture should be accurately measured using a digital thermometer throughout measurements. Remember to frequently stir the bath for optimal thermal equilibration.

#### **Part A: Manometer Pressure Measurement**

**Be sure to record the identity of the given volatile liquid (e.g. n-pentane) in your laboratory notebook.**

At the start of the session:

1. The bulb containing the volatile liquid will be immersed in liquid nitrogen.  
The volatile liquid will be in a frozen state.
2. Note the pressure reading (in mbar) of the manometer and record directly into the Results Sheet provided at the back of laboratory manual.

#### **Part B: Vapour Pressure Measurements**

1. Remove the liquid nitrogen Dewar from the volatile liquid sample bulb.
2. Start thawing the liquid by immersing the sample bulb in a beaker of room temperature water.  
When the volatile liquid is completely melted, replace the beaker of water with the  $2^{\circ}\text{C}$  ice-water mixture (see above). Stir this mixture and note the manometer reading ( $P_m$ ).

To make sure you have reached a stable liquid-vapour equilibrium situation at a specified temperature:

- Tabulate your data directly into the Results Sheet (Part C) provided at the back of the laboratory manual.
- Record both the manometer reading ( $P_m$ ) and time ( $t$ ) at about one minute intervals (with the temperature kept constant), until the reading changes by less than 2 mbar in two successive readings. This may take about 5 – 10 minutes. If it takes you more than  $\sim 10$  minutes to get a stable reading (or if you don’t get a stable reading at all) see your demonstrator.

- Record the final temperature ( $T$ ) of this stable  $P_m$  manometer reading.
  - This last *data pair* is now your *equilibrium liquid-vapour pressure reading ( $\rho$ ) at a specified temperature ( $T$  °C)*. Record this into the *EQUILIBRIUM VAPOUR PRESSURE ( $\rho$ )* and *TEMPERATURE (°C)* columns of the Results Sheet (Part D) provided.
3. Repeat the equilibrium vapour pressure measurement with the ice-baths:
- at ~5 °C, ~10 °C, ~15 °C and tap water temperature (~18 – 20 °C).

**Discussion 1:**

*DO NOT TAKE THE TEMPERATURE ABOVE ROOM TEMPERATURE - WHY? (brief answer only)*

4. CAREFULLY freeze the volatile liquid for about 10 minutes using the liquid nitrogen remaining in the Dewar. Once the volatile liquid is completely frozen record the manometer reading.

**Discussion 2:**

*Is the manometer reading the same as at the start of the experiment? If not what do you think is the reason for the difference? How would this impact on your results? Can you suggest a method of correcting your data for effects of this sort? Include a discussion of these points in your report (in less than 5 lines).*

5. At the end of experiment:

- Remove the liquid nitrogen Dewar from the volatile liquid bulb and leave any remaining liquid nitrogen in the Dewar flask.
- Leave the vacuum system untouched.
- Empty water and ice down the sink.

**Part C: Treatment of Data**

1. Tabulate your raw data (see Part C of Results Sheet at the back of this manual)
  - $\rho$  = Equilibrium Vapour Pressure Measurement at Temperature  $T$  °C  
*(make sure to note the units for the pressure readings)*
  - Temperature  $T$  °C (and Kelvin equivalent)
2. Plot  $\ln \rho$  versus  $1/T$ , where  $\rho$  is the measured equilibrium vapour pressure of volatile liquid at the absolute temperature  $T$ .

*For good graphing technique:*

- *landscape format*
- *graph occupy > 80% of ordinate and coordinate range*
- *label axes with correct scales and units*
- *include a title*
- *draw the line of best fit*
- *show the data points used for gradient calculation*

3. From your graph derive a value for the volatile liquid's molar latent heat of evaporation,  $\Delta H$  (using equation 4.2). Draw the best straight line you can fit by eye when deriving your value for the gradient, and hence for  $\Delta H$ . Include units for the gradient and  $\Delta H$ .

A line of best fit is a straight line drawn through the maximum number of points on a graph balancing approximately an equal number of data points above and below the line.

4. Draw also two other lines of fit, one with the highest acceptable gradient for your set of point, and the other with the lowest acceptable gradient.

**NOTE:** Though it is NOT REQUIRED to do the calculations, should aware these two lines can be further used to place a confidence range on the value of the gradient, and consequently on the value of  $\Delta H$ .

#### Part D: Calculations

1. Using the value of  $\Delta H$  obtained from your graph and a pressure-temperature data pair  $(ln p_1, 1/T_1)$  from the graph, estimating the normal boiling point of your volatile liquid using equation 4.3.

**(HINT:** The normal boiling point ( $T_2$ ) will be at the atmospheric pressure,  $p_2 = 1013 \text{ mbar}$ .  
The universal gas constant  $R$  is  $8.314 \text{ JK}^{-1}\text{mol}^{-1}$ ).

2. Estimate the vapour pressure ( $p_2$ ) of your volatile liquid at  $-116^\circ\text{C}$  ( $T_2$ ).

**(HINT:** Using the value of  $\Delta H$  obtained and a pressure-temperature data pair  $(ln p_1, 1/T_1)$  from your graph accordingly to equation 4.3).

3. How much energy (in kJ) is required to vaporise 27.5 g of your volatile liquid?

**(HINT:** Consider the amount of volatile liquid in moles and the  $\Delta H_{vap}$  obtained has a unit of  $\text{kJmol}^{-1}$ ).

4. Calculate the volume of vapour that would be generated at 1 atm. pressure and  $40^\circ\text{C}$  by the evaporation of 27.5 g of your volatile liquid.

**(HINT:** Assume ideal gas behaviour,  $PV = nRT$ .  
The universal gas constant  $R$  is  $0.0821 \text{ L atm K}^{-1}\text{mol}^{-1}$ ).

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |                                       |  |                                      |
|---|---------------------------------------|--|--------------------------------------|
| <input type="checkbox"/> Corrosive  | <input type="checkbox"/> Irritant     | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input type="checkbox"/> Toxic  | <input type="checkbox"/> Carcinogenic | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric   | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |                                       | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |                                       | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |  |
|---|--|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions   |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?                       |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input checked="" type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |  |

### Special Precautions

- |   |  |   |
|---|--|---|
| <input checked="" type="checkbox"/> Special eye protection  | <input checked="" type="checkbox"/> Safety shield              | <input type="checkbox"/> Face mask                                  |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input checked="" type="checkbox"/> Any other – liq. N <sub>2</sub> |

*Handle liquid N<sub>2</sub> with extreme care. Possibility of cryogenic burns*

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

**Risk Assessed by:** Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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# GLASS ELECTRODE – BUFFER SOLUTIONS

5

## AIMS OF THE EXPERIMENT

- To titrate a weak base ( $\text{Na}_2\text{CO}_3$ ) with a strong acid (HCl) using
  - (a) an acid-base indicator, and
  - (b) a glass electrode.

From this titration, the  $pK_a$  for the bicarbonate ion and carbonic acid will be determined and the colour behaviour of indicators observed.

- To prepare and investigate the properties of a ‘buffer solution’ comprising sodium carbonate and sodium bicarbonate and to compare the measured pH with the calculated value.
- To investigate the response of the carbonate / bicarbonate buffer solution to the addition of a small amount of (a) strong acid and (b) strong base.

### Note:

You will require a basic scientific calculator, graph paper (2 mm ruling), a sharp pencil and a 30 cm ruler as well as your laboratory notebook.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Concentrations of solutions and volumetric analysis: Section 1.5, pages 34 – 38;
  - Measuring pH with glass electrode: Section 11.2, page 524;
  - Buffer solutions: Sections 7.3, pages 319 – 321;
  - pH changes in acid-base titrations: Section 7.4, pages 322 – 327;
  - Indicators: Section 7.5, pages 327 – 329.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:
  - Acid-base titration: pages 472 – 478;
  - The concept of pH: pages 438 – 441;
  - Acid-base indicators: pages 479 – 481;
  - Buffer solutions: pages 466 – 470.
- Techniques and Instrumentation section of this laboratory manual:
  - Pipettes: page 130;
  - Burettes: page 131.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL Prelabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the CAL module has been completed.

## SAFETY



**Caution: Take care with applying pipette filler onto the pipette.**

**Disposal of Chemical Wastes:** All solutions used in this experiment can be safely disposed of by flushing down the sinks. Indicator paper must be put in the waste bins, NOT in the sinks or runnels.

## Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLab module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## INTRODUCTION

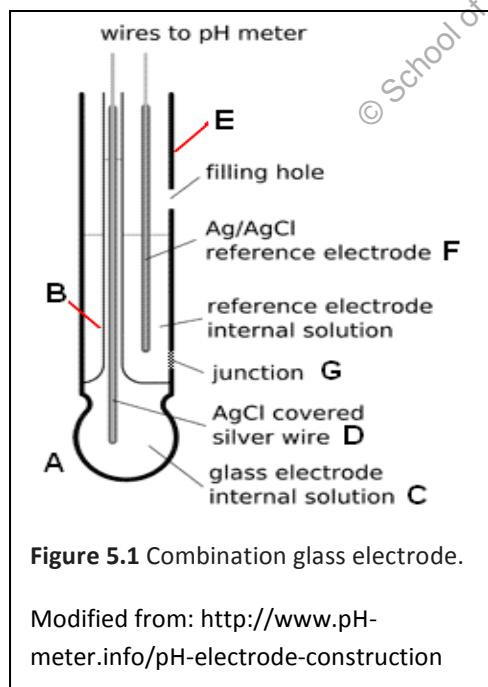
For this experiment:

- Students work in pairs.
- The report is written individually in own words.
- All calculations and graphing of data collected during the experiment must be completed individually by students.
- A Results Sheet is provided at the back of this laboratory manual (page 159).

## APPARATUS

### 1. Glass Electrode:

You will use a combination electrode (shown in Figure 5.1) in which the glass electrode is combined with a second reference electrode outside the glass membrane. It also has its own salt bridge to connect both electrodes to the solution being measured. In use, the combination electrode is immersed into the test solution.



A thin-walled bulb **A** is blown at the end of a glass tube **B**. This tube contains a chloride solution **C** of fixed pH in equilibrium with a silver-silver chloride electrode **D** (reference electrode 1).

**B** is sealed inside a second glass tube **E** which also contains a silver-silver chloride electrode **F** in a chloride solution (reference electrode 2). The second reference electrode is in contact with the test solution through the porous ceramic partition **G**.

A plastic shield protects the whole combination electrode. Electrical connections to the two electrodes are made through the two seals at the top.

The electrode potential of the glass electrode immersed in a solution of unknown pH at 298 K is:

$$E = E_{\text{const}} - 0.0592 \text{ pH},$$

where  $E_{\text{const}}$  is dependent upon the potential of the reference half-cell (in this case the potential of the Ag/AgCl half-cell) and the pH of solution **C**.  $E_{\text{const}}$  is a constant specific to a given electrode.

Note that at 25 °C the potential of a glass electrode decreases by 0.0592 V when the pH increases by one unit (*i.e.* when  $[H^+]$  drops tenfold).

*When not in use, the electrode must be stored in distilled water and must not be allowed to dry out.*

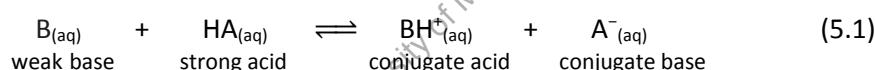
## 2. Potentiometer-pH Meter

The meter indicates potential in either volts or millivolts. It can also give pH directly after being standardized with a buffer solution. This calibration has been carried out prior to the practical session. It is important that the adjustment controls on the pH meter are not changed from their initial settings during the experiment.

### ACID-BASE TITRATION

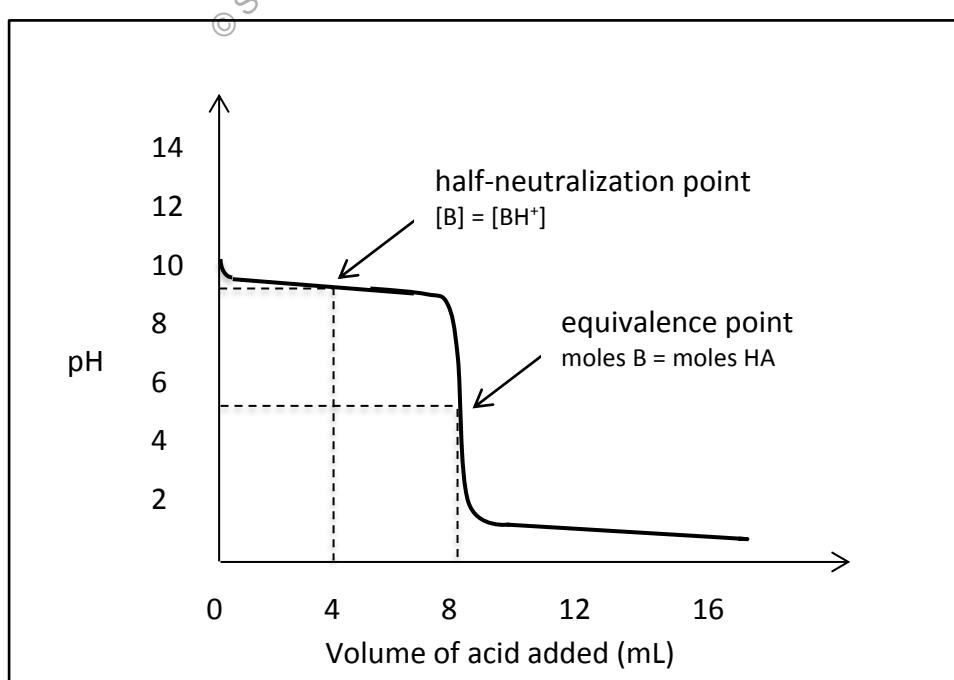
Titration is one of the most common laboratory methods of quantitative chemical analysis. It is used to determine the precise concentration of an *analyte* (such as a weak base  $Na_2CO_3$ ) in the conical flask with the delivery of a *titrant* (for example a strong acid  $HCl$ ) of accurately known concentration from a burette.

An acid-base titration can be monitored with an indicator or with a pH meter for the determination of *equivalence point* of the reaction. It is the point at which the number of moles of added acid ( $HA$ ) is stoichiometrically equal to the number of moles of weak base ( $B$ ).



An acid-base indicator undergoes colour changes with the pH of solution. If an indicator has been properly selected, it will provide an approximation of the *equivalence point*.

A pH meter, however, will allow a more accurate determination of the *equivalence point* of the reaction. It is used to measure the pH of solution providing a direct method of obtaining a titration curve. Titration curve is a plot following the change in pH of the analyte solution in the conical flask as the titrant is added from the burette.



**Figure 5.2** A typical titration curve for a weak base-strong acid titration.

A typical curve for this weak base-strong acid titration is shown in Figure 5.2 and the important regions of the titration curve are:

#### 1. The region of weak base

At the beginning of titration, the analyte solution (B) is a weak base with a pH greater than 9. As the titrant of strong acid (HA) is added, there is a quick drop in pH of solution as the concentration of weak base is reduced by the addition the strong acid.

#### 2. The region of equivalence point

As strong acid is continuously titrated into the solution, the point at which the number of moles of added acid (HA) is stoichiometrically equal to the number of moles of weak base (B) is called the *equivalence point* of the reaction. At this point is where the titration curve has the steepest slope with a very large change in pH over a small range of the added volume of acid. After this adding any more of the strong acid will rapidly make the solution acidic.

The pH of the solution at *equivalence point* is dependent on the strength of the acid and base used in the titration. The volume of added acid at the *equivalence point* is estimated to be at the mid-point of the "steepest section" of the titration curve. It can be used together with its known concentration to determine the number of moles of strong acid have been added to neutralize the weak base in reaction solution. This allows the determination of the concentration of weak base knowing its initial added volume where,

$$\text{moles of HA} = \text{moles of B} = [B] \times \text{Volume of B} \quad (5.2)$$

#### 3. The region of buffer solution and half-neutralization point

After the initial drop in pH, the titration curve levels off through the buffer region near the *half-neutralization point*. In this relatively "plateau" buffer region there is minimal change in pH of the solution upon addition of the strongly acidic titrant. The buffering region is about 1 pH unit on either side of the  $pK_a$  of the conjugate acid.

The *half-neutralization point* is when exactly half of the concentration of weak base (B) has been converted to its conjugate acid ( $BH^+$ ) and hence they have equal concentration. According, to Henderson-Hasselbach equation (equation 5.3, prefer to lecture notes and text book),

$$pH = pK_a + \log_{10} \frac{[B]}{[BH^+]} \quad (5.3)$$

the pH for a weak base at its *half-neutralization point* is therefore the  $pK_a$  of its conjugate acid.

$$pH = pK_a \quad (5.4)$$

The volume of added acid at this *half-neutralization point* is half of what it is at the equivalence point. Hence in the titration of a weak base with a strong acid the  $pK_a$  of the conjugate acid can simply be read off the titration curve as the pH at the *half-neutralization point*.

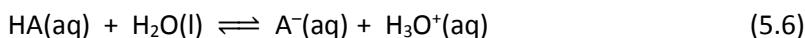
$$\begin{array}{lcl} \text{volume of added acid} & = & \frac{1}{2} \times (\text{volume of added acid} \\ \text{at half-neutralization point} & & \text{at equivalence point}) \end{array} \quad (5.5)$$

#### 4. The region of excess strong acid

As the titration continues beyond the equivalence point, the dominant species is the hydrogen ion of strong acid (HA) which now determines the pH of reaction mixture.

## BUFFER SOLUTIONS

Buffer solutions are used extensively in chemical and biological industry and especially within physiological environments. A buffer is an aqueous solution consists of a mixture of a weak acid ( $\text{HA}$ ) and its conjugate base ( $\text{A}^-$ ), or vice versa, establishing the equilibrium:



where

$$K_a = \frac{[\text{A}^-][\text{H}_3\text{O}^+]}{[\text{HA}]} \quad (5.7)$$

and with references to the Henderson-Hasselbalch equation given in equation 5.3,

$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{A}^-]}{[\text{HA}]} \quad (5.8)$$

An efficient buffer solution is when the ratio  $[\text{A}^-]/[\text{HA}]$  is close to 1. Buffer solution resists significant pH change upon the addition of strong acid (e.g. HCl) or base (e.g. NaOH) keeping the solution pH relatively stable. Maximum buffer capacity is attained when the  $\text{p}K_a$  of the weak acid used is closed to the desired pH range of the buffer solution.

## EXPERIMENTAL PROCEDURE

### PART A. Titration of a weak base with a strong acid: action of indicators

**Notes:** Make sure that the beakers to which the sodium carbonate and hydrochloric acid are added are clean and **completely** dry. Prepare your data table (using the result sheet on page 159) and graph axes prior to proceeding with the titration. **If you do not plot the pH-Volume graph as you proceed with the titration it is unlikely that you will finish the experimental work by the end of the session.**

For good graphing technique:

- landscape format
- graph occupy  $\geq 80\%$  of ordinate and coordinate range
- label axes with correct scales and units
- include a title
- consider the smooth titration curve
- show equivalence points and half-neutralization points

1. Empty the contents of the **reaction beaker**, being sure not to dispose of the stirrer bead, and rinse twice with distilled water.
2. Rinse a 10.00 mL pipette twice with distilled water and once with 0.1 M sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) solution.
3. Accurately pipette a 10.00 mL aliquot of the 0.1 M sodium carbonate solution into the reaction beaker; add 70 mL of distilled water and phenolphthalein indicator (2 – 3 drops).
4. Place the glass electrode and stirrer bead in the reaction beaker, checking that the electrode – including the porous frit – is covered and is clear of the moving stirrer; add more water if necessary. Record the pH.
5. Rinse and fill the burette with about 30 mL of 0.1 M HCl.  
*Ensure the funnel is removed from the burette and the tip of burette is filled with solution with no air bubbles before titration (why?).*



Pipette



Volumetric  
techniques

6. Titrate the sodium carbonate solution by adding 1 mL amounts of the 0.1 M HCl, recording the pH and the burette volume (to 2 decimal places) after each addition, until 7 mL to 8 mL has been added **or** when  $\Delta\text{pH} > 0.3$  in two successive readings. Note and record the intensity and colour of the indicator in the solution after each addition.  
**(NOTE: The equilibrating time between each addition of HCl is about 30 seconds.)**
7. Now add acid in  $\sim 0.20$  mL amounts, continuing to note the pH, burette volume and indicator colour until the pH change per addition is minimal ( $\Delta\text{pH} < 0.2$ ).
8. Add the Bromophenol Blue indicator and continue the titration with 1 mL additions until 16 mL to 17 mL of acid has been added **or** again when  $\Delta\text{pH} > 0.3$  in two successive readings. Note and record volume, pH and colour as before.
9. At this point reduce the amount of acid added each time to  $\sim 0.20$  mL, continuing to note volume, pH and any colour changes.
10. When the pH change per addition again becomes minimal when  $\Delta\text{pH} < 0.2$ , make 3 further additions each of 2 mL, recording the pH after each addition.

#### Question 1

Why are two indicators (phenolphthalein and bromophenol blue) used for this titration?

#### Question 2

Comment on the correlation between the change in colour of the indicators and the regions of rapid pH change on the titration curve.

#### Question 3

From the graph record the volume of titre **at both equivalence points**. Comment on the significance of equivalence point for an acid and base titration.

#### Question 4

The plateau regions of the titration curve:

- (i) *before* the first equivalence point and
- (ii) *between* the first and second equivalence points are buffer regions.

The equilibrium reactions in these regions can be represented using equation 5.6.

Write the ionic equation for acid dissociation for each of the reactions in regions (i) and (ii).

#### Question 5

Write the equilibrium expression ( $K_a$ ) for the acid dissociation reactions in Question 4:

- *before* the first equivalence point
- *between* the first and second equivalence points

#### Question 6

From the pH-volume graph, read off the pH at each of the half-neutralization points (*i.e.* refer to equations 5.3, 5.4 and 5.5). Use these half-neutralisation pH values and the equilibrium expressions from Question 5 to determine the value of the  $pK_a$  for the bicarbonate ion ( $\text{HCO}_3^-$ ) and carbonic acid ( $\text{H}_2\text{CO}_3$ ).

**Hint:** What is the relationship between  $[\text{CO}_3^{2-}]$  and  $[\text{HCO}_3^-]$  at the first half-neutralization point? What is the relationship between  $[\text{HCO}_3^-]$  and  $[\text{H}_2\text{CO}_3]$  at the second half-neutralization point?

## PART B. Buffer solutions containing carbonate and bicarbonate ions

**Notes:** Record ALL results and answers directly into the Results Sheet provided at the back of this laboratory manual on page 159.

### Step 1

- (i) Put 40 mL of 0.1 M sodium bicarbonate ( $\text{NaHCO}_3$ ) in a beaker.
- (ii) Add 40 mL of 0.1 M sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) to the bicarbonate solution and measure the pH.

### Question 7

Record your results.

### Question 8

Calculate the pH of the mixture in (ii) using equation 5.8 given that  $\text{p}K_a$  of the bicarbonate ion is 10.33 and equal amounts of carbonate ion and bicarbonate ion are used. Compare the calculated and experimental values.

### Step 2

- (i) Transfer half the solution containing carbonate ion ( $\text{CO}_3^{2-}$ ) and bicarbonate ion ( $\text{HCO}_3^-$ ) to another beaker.
- (ii) To one of these solutions add 1 mL of 0.1 M HCl, measure the pH (using the glass electrode) and record the change in pH due to the addition of the acid.
- (iii) Put about 40 mL of water into another beaker, measure the pH, add 1 mL of 0.1 M HCl to the water and record the change in pH.

### Question 9

Write a chemical equation for the reaction which occurs when HCl is added to the carbonate/bicarbonate solution.

### Question 10

Does the ratio of  $[\text{CO}_3^{2-}]/[\text{HCO}_3^-]$  increase, decrease or remain the same when the HCl is added to the carbonate/bicarbonate solution?

### Question 11

How is the addition of HCl affecting the pH of water?

### Question 12

Was the pH change produced by adding HCl to water larger than the pH change produced in the carbonate/bicarbonate solution?

**Step 3**

- (i) To the rest of the carbonate/bicarbonate solution add 1 mL of 0.1 M NaOH, measure the pH (using glass electrode) and record the change in pH.
- (ii) Put about 40 mL of water into another beaker, measure the pH, add 1 mL of 0.1 M NaOH and record the change in pH.

**Question 13**

Write a chemical equation for the reaction which occurs when NaOH is added to the carbonate/bicarbonate solution.

**Question 14**

Does the ratio of  $[\text{CO}_3^{2-}]/[\text{HCO}_3^-]$  increase, decrease or remain the same when the NaOH is added to the carbonate/bicarbonate solution?

**Question 15**

How is the addition of NaOH affecting the pH of water?

**Question 16**

Was the pH change produced by adding NaOH to water larger than the pH change produced in the carbonate/bicarbonate solution?

**Question 17**

Write a statement explaining why the carbonate/bicarbonate buffer solution shows only a small change in pH when strong acid or strong base is added.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input type="checkbox"/> Toxic  | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input checked="" type="checkbox"/> Minimal risk   |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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# A POLYIODIDE SALT: SYNTHESIS & ANALYSIS:

**6**

## AIMS OF THE EXPERIMENT

- To synthesise a polyiodide salt.
- To determine the chemical formula of the polyiodide using analytical methods.
- To introduce important synthetic and analytical methods and techniques.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Concentrations of solutions and volumetric analysis: Section 1.5, pages 34 – 39;
  - Relative formula mass and chemical formulae: pages 18 – 20;
  - Interhalogen compounds of Group 17 elements: pages 1246 – 1247.
- Techniques and Instrumentation section of the laboratory manual:
  - Balances: pages 127 – 128;
  - Volumetric flasks and pipettes: page 130;
  - Burettes: page 131;
  - Filtration: page 132.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL PreLabs module which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## INTRODUCTION

For this experiment:

- Students work individually
- You will require a basic scientific calculator.
- A Results Sheet is provided at the back of this manual (page 161)

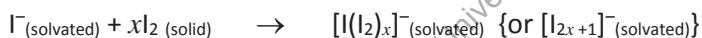
The group 17 elements or halogens – fluorine, chlorine, bromine and iodine – are among the most reactive of the elements. Accordingly, only their compounds, materials produced upon their reaction with other elements, are found in nature. Their reactions, particularly those involving fluorine and chlorine, are often vigorous and extremely dangerous. The halogens are toxic, corrosive substances, which can attack the eyes, mucous membranes and skin and cause death by asphyxiation. The gaseous and liquid halogens are particularly dangerous.

In this experiment, you will prepare and analyse a compound of the relatively safe halogen, iodine (a solid). Iodine and polyiodide compounds are used as catalysts in the rubber industry, as nutritional supplements and in the production of dyestuffs, pigments, pharmaceuticals, disinfectants and photographic film.

The Law of Definite Proportions states: with a given compound, the constituent elements are always combined in the same proportions by mass. Through the mole concept, the knowledge of the mass percentage composition of a compound permits the determination of its empirical formula. Experimentally, chemical and/or spectroscopic analyses are employed to establish the mass percentage composition of the compound, and this information is then used to deduce the formula. The formula of a compound is a vital piece of chemical information. In this experiment, you will synthesise and chemically analyse a polyiodide salt, and thereby determine its chemical formula.

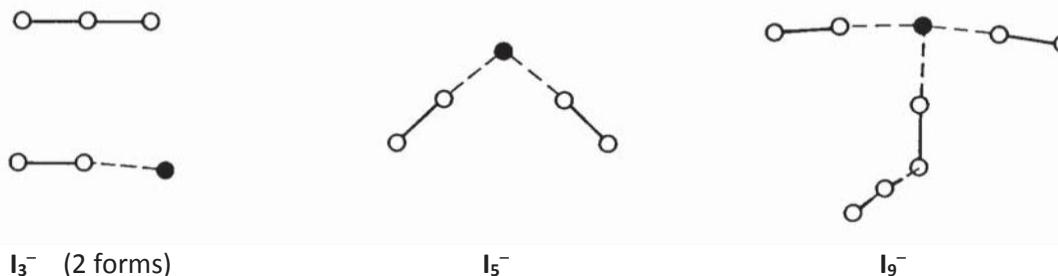
### Polyiodide Compounds

You are no doubt aware of the existence of the common halide anions,  $X^-$ : fluoride, chloride, bromide and iodide. These readily formed and very stable anions result from the completion of the  $(ns)^2(np)^6$  octet by addition of one electron to an  $(ns)^2(np)^5$  halogen atom. You may, however, be surprised to learn that the halogens form a wide variety of polyhalide anions, such as  $[ICl_2]^-$ ,  $[ICl_4]^-$ ,  $[Br_5]^-$ ,  $[IF_6]^-$ ,  $[I_7]^-$ , etc. Iodine, although only slightly soluble in water and alcohols, readily dissolves in solutions containing iodide due to the formation of polyiodide anions.



Indeed, under certain conditions it is possible to isolate from such solutions a variety of compounds having the general formula  $[A][I(I_2)_x]$  or  $[A][I_{2x+1}]$ , where A is a cation. Take for example  $[NMe_4]^+[I_{11}]$  in this case A is the tetramethylammonium cation,  $NMe_4^+ = N(CH_3)_4^+$ , and x equals 5.

Most of these beautiful and intriguing compounds were prepared over a century ago but only after the advent of X-ray crystallography were their striking structures revealed (Figure 6.1). This experiment will employ a titrimetric analysis to determine the percentage by mass of 'free' iodine (open circles in Figure 6.1) in the compound synthesised. Combined with carbon, hydrogen and nitrogen analyses, a full formulation for the compound may be established.



**Figure 6.1** The structures of some polyiodide anions in ionic salts of  $NMe_4^+$ : Iodide anions (filled circles) weakly interact with iodine molecules (open circles). The polyiodide you will synthesise is a salt of one of these anions.

## SAFETY



### Safety Warning

I<sub>2</sub> is corrosive, avoid contact with skin.

Methanol is a flammable liquid and toxic by skin absorption or breathing of vapours. Avoid contact with flames or electrical equipment. Avoid breathing the vapour and avoid contact with the skin. It should be handled in the fume hood.

**Wear gloves at all time. If there is any spillage of methanol on gloves, remove immediately and replace with new gloves.**

**CAUTION:** Hotplates have hot surfaces and can cause burns  
Take care with applying pipette filler onto the pipette.

### Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the back of this manual for submitting your receipt number for the ChemCAL PreLab module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

Enter your results on the Results Sheet provided at the rear of this manual.

### Part A: Preparation of the Polyiodide Salt

The following materials are provided:

Material	Appearance	Mass
Tetramethylammonium iodide	White solid	Measure approx. 0.25 g using scoop Weigh accurately
Iodine solution in methanol	Black/purple solution	0.825 g/10 mL

1. Rinse 100 mL conical flask with a small amount of methanol (to remove any water).
2. Weigh out (using weighing paper) and add the **known** amount of tetramethylammonium iodide solid into the conical flask and then add the iodine solution (10 mL).
3. Place a small watch-glass over the mouth of the flask and bring the mixture to a gentle boil on a hot plate in the fume hood. As the solvent comes to the boil, remove the flask from the hot plate and swirl the contents vigorously for a minute, then bring the solution to the boil once again. Repeat over the 5 minutes of times until all solid has dissolved (look through the bottom of the flask, preferably with a torch, for undissolved solids).
4. Top the solution volume up to 10 mL with methanol ONLY if significant solvent evaporation takes place (*check with demonstrator as this is usually not necessary*).
5. Finally, allow the hot solution to cool undisturbed in the fume hood for about 15 – 20 minutes. Then, gently swirl the mixture to induce further crystallisation.

6. Let the mixture stand for a further 10 minutes with occasional swirling.
7. Vacuum filter the product (see page 132).

*Note that the filtration should be very rapid with the vacuum taps only just slightly turned on. A high vacuum tends to rapidly cool the mixture, precipitating solid on the filter paper, causing a blockage which is best avoided.*
8. Wash the crystals with ice-cold methanol (5 – 10 mL only) and dry at the pump by drawing air through them for about 10 minutes. Your demonstrator will explain how to do this.
9. Record the mass of product obtained.
10. Transfer your product into a sealable sample bag which should then be placed into a second bag to minimize escape of iodine vapour. Fully label the sample.



Hirsch vacuum  
filtration

#### Part B: Analysis of the Polyiodide Salt

1. The sample solution for the titrimetric analyses should be accurately prepared:
  - First, ensure that your sample is dry.
  - Using a top-loading balance, weigh into a small, clean and dry beaker about 0.2 g of your compound, then proceed to the analytical balance.
  - Tare the balance with the pan unloaded and then place your beaker containing the compound on the balance pan. Be sure the doors of the balance are closed during all weighing. **Record the combined mass of the beaker and compound.**
  - Transfer the compound to a 50 mL volumetric flask using a funnel. Using the ethanol/KI solution ensure all the solid on the funnel is washed into the volumetric flask. A transfer pipette may be used for this purpose.
  - **Do not wash the beaker.** It doesn't matter if there is still some solid left behind in the beaker.
  - Immediately re-weigh, on the same analytical balance, the beaker. **Record its mass.**
  - The mass of compound delivered to the receiving vessel is obtained by difference.



Analytical balance



Volumetric Flask

2. Add about 30 – 40 mL of ethanol/KI solution to the volumetric flask. Remove the funnel and agitate until all solids have dissolved, then fill the flask to the mark with ethanol/KI solution using a transfer pipette. Mix thoroughly and check that the last traces of compound have dissolved.

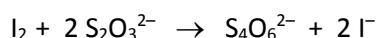
*The red-brown colour of the solution is due to the presence of iodine. Since iodine is volatile and may be slowly lost from solution, keep the flask stoppered when not in use.*

- Rinse and fill the burette with sodium thiosulfate solution. Ensure the funnel is removed from the burette and the tip of burette is filled with solution with no air bubbles before titration.

*Sodium thiosulfate is not a primary standard for analytical work since it is slightly efflorescent (the solid loses water of crystallisation) and its solutions slowly decompose. The thiosulfate solution will be standardised by Technical Staff and your demonstrator will supply you with the exact concentration.*

- Accurately pipette (see page 130) 5 mL aliquot of your polyiodide stock solution into a 250 mL conical flask, then add 25 mL of water using a measuring cylinder and 1 mL of 2 M H<sub>2</sub>SO<sub>4</sub>.

- The iodine in the sample is estimated titrimetrically (see page 131) with sodium thiosulfate accordingly to the following equation:



Volumetric  
techniques

- Titrate the free iodine until a light-yellow colour is produced.
- At this point in the titration add 1 mL of starch indicator solution, which will cause the solution to turn a deep blue colour.  
*If the starch solution is added too early, the remaining iodine in solution may appear as blue coloured lumps making the determination of the endpoint uncertain.*
- Continue the titration until the blue colour has completely disappeared.
- Record the burette reading (to 2 decimal places) at this endpoint of colourless solution.

- Repeat the titration until concordant results (readings within 0.10 mL to 0.20 mL of each other) are obtained.
- By following the steps given in the Results sheet, calculate the percentage of elemental iodine in your polyiodide sample (accurately weighed in above Step 1) and determine the appropriate value of x for the polyiodide salt (NMe<sub>4</sub>·I<sub>(2x+1)</sub>).

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input checked="" type="checkbox"/> Pungent  | <input type="checkbox"/> Stench      |
| <input type="checkbox"/> Toxic  | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input checked="" type="checkbox"/> Other: Exposure to hot surfaces (hotplate)              |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- |  |
|--|
| <input type="checkbox"/> 3 Minimal risk  |
| <input type="checkbox"/> 2a Low risk (Fume hood recommended)                       |
| <input checked="" type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential) |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)                     |
| <input type="checkbox"/> 1b Significant risk (Special location or facility)        |

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

# THE REDUCTION OF BENZOIN:

7

## The synthesis of *meso*-1,2-DIHYDROXY-1,2-DIPHENYLETHANE and an application of THIN LAYER CHROMATOGRAPHY

### AIMS OF THE EXPERIMENT

- To prepare *meso*-1,2-dihydroxy-1,2-diphenylethane by sodium borohydride reduction of benzoin.
- To purify *meso*-1,2-dihydroxy-1,2-diphenylethane by mixed solvent recrystallisation.
- To establish the purity of recrystallised *meso*-1,2-dihydroxy-1,2-diphenylethane using the technique of thin layer chromatography.

### READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Working out the yield of a reaction: pages 24 – 25;
  - Chromatography: Section 11.3, pages 528 – 532;
  - Nucleophilic addition reactions of aldehydes and ketones: pages 1061 – 1064.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:
  - Stoichiometry, limiting reagents and percentage yield: pages 93 – 99;
  - Reduction of aldehydes and ketones: pages 964 – 965.
- Techniques and instrumentation section of this laboratory manual:
  - Top-loading balances: page 127;
  - Laboratory equipment and glassware: page 129;
  - Filtration: page 132;
  - Purification of compounds: pages 133 – 134;
  - Criteria of purity of compounds: pages 135 – 136.

### PRE-LAB QUESTIONS

There is a compulsory ChemCAL Prelabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual. The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

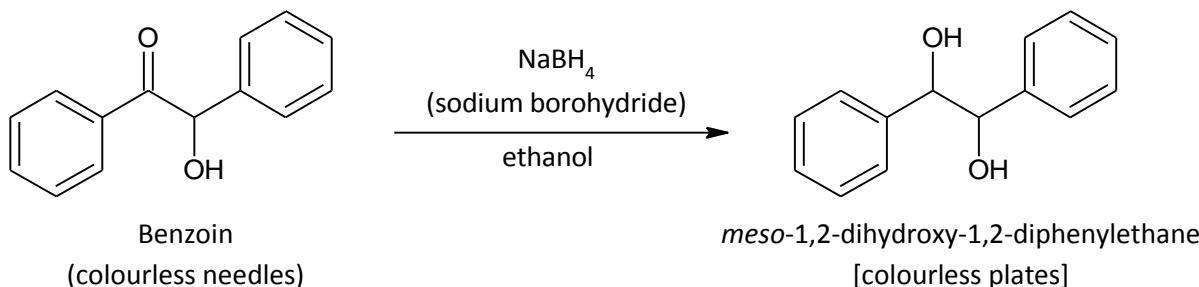
### INTRODUCTION

For this experiment:

- students work individually
- You will require a basic scientific calculator
- Part A is to be written up in “Experimental Method” of report (refer to page 68)

## Preparation of *meso*-1,2-Dihydroxy-1,2-diphenylethane

This preparation demonstrates the utility of hydride reagents for the reduction of organic compounds. Aldehydes and ketones are reduced rapidly and quantitatively to alcohols by the borohydride ion. In this specific example the rate of reaction depends on the rate of dissolution of the benzoin.



## Thin Layer Chromatography

Before the properties and/or structure of a compound can be examined, it must be pure. One very useful technique for establishing purity is thin layer chromatography (this being classed as one type of liquid-solid chromatography).

In the thin layer chromatography experiment the sample to be analysed (which will frequently consist of a mixture of the compound of interest and one or more impurities) is first adsorbed onto an inert substance called the stationary phase (in this experiment a thin layer of silica gel –  $\text{SiO}_2 \cdot x\text{H}_2\text{O}$  – on an aluminium backing) which has some polar groups. The stationary phase is then brought into contact with a less polar organic solvent (mobile phase). As the solvent moves past the point on the TLC plate where the mixture has been applied, the components of the mixture move different distances depending on their polarity.

Polar organic molecules, having a higher proportion of oxygen- and nitrogen-containing functional groups, are attracted more to the polar stationary phase and don't shift far from the point at which they were applied. Less polar compounds containing a higher proportion of carbon and hydrogen are attracted less to the polar stationary phase and move further from the point of origin.

## SAFETY

### Safety Warning

Ethanol, acetone, ethyl acetate and hexane are volatile liquids, which are flammable. Avoid contact with flames or electrical equipment. Phosphomolybdic acid is corrosive. Avoid contact with skin.



### Disposal of Wastes

Please ensure that all organic wastes are disposed of in the appropriate waste containers. Do not dispose of any organic materials down the sinks.

**Caution: The steam and hot surfaces of steam baths can cause burns.**

### Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### PART A. Preparation of *meso*-1,2-Dihydroxy-1,2-diphenylethane

Sodium borohydride is provided as a 5.27 M solution in water, which has been made alkaline to preserve the borohydride.

1. Rinse the 50 mL conical flask with small amount of distilled water and thoroughly DRY with paper towel.
2. Weigh out about 0.5 g of benzoin (to  $\pm$  0.01 g) using weighing paper and add directly into the DRY conical flask and then add 3.5 mL of ethanol from measuring cylinder. Then add 0.5 mL of the sodium borohydride solution to the suspension of benzoin in ethanol using a DRY dropping pipette.
3. Heat the mixture on the steam bath with swirling. Once the benzoin has dissolved, continue to heat for a further 1 – 2 minutes with occasional swirling. Dilute the hot solution with water (about 30 mL from wash bottle).
4. Chill the suspension in ice, filter off the product and wash it well with portions of chilled water.
5. The product is very soluble in hot ethanol and because the quantity of product is rather small you are to use a slight variation in the general technique of recrystallization (your demonstrator will instruct you about how to do this).
6. Transfer the crude product to a clean 50 mL conical flask and dissolve in minimum amount of hot ethanol (starts off by adding 1 – 2 mL portion, usually do not require more than 3 mL). If the solution is clear and colourless at this stage, add hot water drop-wise using a dropping pipette until crystals separate and just fail to redissolve on continued heating. Usually 8 – 9 drops (no more than 10 drops) of hot water are sufficient (see pages 133 – 134).
7. Allow the mixture to cool undisturbed for 2 – 3 minutes to room temperature, then chill in the ice-water bath. Filter off the purified product, wash with chilled water (small portion), then press dry at pump with spatula and dry on a watch glass over a steam bath.
  - Whilst the product is drying, place a small amount on filter paper (see demonstrator) and “flash dry” on the edge of a steam bath. Use this sample to determine the melting point ( $>120$  °C) of your product.
  - The *meso*-1,2-dihydroxy-1,2-diphenylethane, you obtain should crystallise as colourless glistening plates. Determine the yield of product and, based on the amount of reacted benzoin, calculate the theoretical and percentage yields of product. The percentage yield you obtain will be well below the theoretical yield because of the solubility properties of the compound.
  - Submit the fully labelled snap-lock plastic bag to your demonstrator:



Top-loading  
balance



Hirsch vacuum  
filtration



MP determination

Student Name (*i.e.* Your Name)

Date, Group Number, Day

*meso*-1,2-dihydroxy-1,2-diphenylethane (recrystallised)

M.P. Range (*e.g.* M.P. = 129 – 130 °C)

Yield, % Yield

Figure 7.1 Sample Label

## PART B. Thin Layer Chromatography

1. Take the TLC plate provided and, being careful not to touch the surface with your fingers, lightly draw a pencil line 1 – 2 cm from the bottom edge.
2. Prepare a solution of your *meso*-1,2-dihydroxy-1,2-diphenylethane by dissolving a few crystals of your recrystallized material in a few drops of acetone.
3. Use the capillary pipettes to carefully place one ***small spot*** with just sufficient amounts of:
  - a) the solution of your *meso*-1,2-dihydroxy-1,2-diphenylethane
  - b) the solution of a 1:1 mixture of benzoin and the diol reduction product (supplied) at the positions indicated in Fig. 7.2.



TLC setup

(Ensure that the spots are placed higher than the solvent level in the tank).

Try not to pierce the surface of the silica gel as you spot your sample. Ensure that the spots are kept as small as possible. Spotting can be done more than once to ensure enough material is applied to the plate, but if you do this allow drying between subsequent spotting.

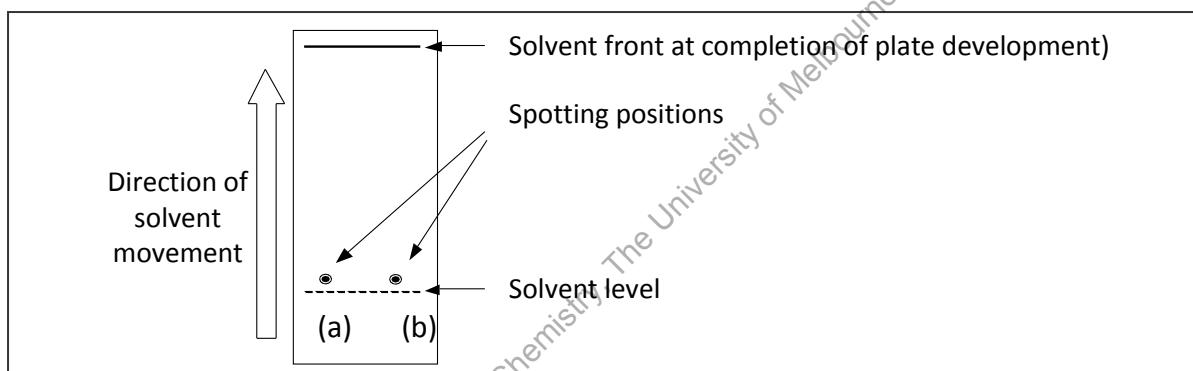


Figure 7.2 Thin Layer Chromatogram

4. Solvent tanks (in FUMEHOOD) containing a 1:3 (v/v) mixture of ethyl acetate and hexane are used to develop the TLC plates. Lower the plate into the solvent making sure that the spots are above the level of the solvent in the tank. Replace the tank lid, and allow the plate to develop undisturbed (do not walk around with the tank in your hands!) until the solvent is 1 cm or less below the top of the silica gel.
5. Remove the plate from the tank and immediately mark with pencil the position of the solvent front when the plate was removed from the tank. Allow the solvents to evaporate from the surface of the plate by leaving it face-up in the fume hood.
6. The next step in the TLC process involves visualisation of the plate. Place your dry plate underneath the ultra-violet (U.V.) lamp provided.
7. You should see a single dark circle (against a green background) in the upper left-hand section of the plate above the point at which the solution containing the mixture of benzoin and *meso*-1,2-dihydroxy-1,2-diphenylethane had been spotted.



Developing TLC plates

There should be no comparable dark circle on the plate above the spot where you applied a sample of your recrystallized product. Lightly outline the dark circle you see with a pencil.

This dark spot is due to the benzoin, which “shows up” under U.V. light because the molecule contains a chromophoric benzoyl ( $C_6H_5CO-$ ) unit. Your product molecule, spot 1, does not have such a grouping and is therefore not easily seen under the U.V. lamp (although it may appear as a much lighter spot). There are, however, ways of detecting so-called non-chromophoric materials on TLC plates. One technique employs dipping the plate into active solutions. In this experiment the solution used is phosphomolybdic acid (PMA) which is reduced to a blue-grey mixed oxide when it reacts with the organic compounds on the plate.

8. Dip the TLC plate (after you have looked at it under the U.V. lamp) in the 2.5% solution of phosphomolybdic acid (PMA) in isopropanol. Remove the plate from the solution and allow any excess liquid to run off the plate, carefully touch the plate to a tissue to remove any remaining surface liquid.
9. When the TLC plate appears to be dry gently heat it on a hotplate (CAREFUL: The hotplates are very hot!). The TLC plate will become discoloured and three dark spots should appear. Place the TLC plate in a labelled sample bag and attach to your lab report.

10. **Calculation of  $R_f$  values:**

If the preparation of the TLC plate and its development has been carried out under strictly constant conditions, the ratio of the distance travelled by the spot to the distance travelled by the solvent front should be constant for a given compound. This ratio is known as the  $R_f$  value for that compound and will always be in the range 0–1.

$$R_f = \frac{\text{distance travelled by spot from spotting position}}{\text{distance travelled by solvent front from spotting position}}$$

Do the measurement from the centre of spot.

- Calculate the  $R_f$  values for each of the spots on your plate.
- Comment on the purity of your recrystallized product. Does it contain any benzoin?

## **WRITING UP OF PREPARATIVE EXPERIMENTS**

Your report on the preparation of *meso*-1,2-dihydroxy-1,2-diphenylethane, and any other preparative experiment, should follow the style used by leading chemical journals. Particularly, the report needs to be CONCISE:

1. The “Aim” should be 1 to 2 sentences
2. The “Experimental Method” should be no more than TWO short paragraphs of 3 to 4 sentences each:
  - Written in past tense and passive voice (“acetic anhydride was added to a solution of 4-aminophenol” NOT “I added acetic anhydride to a solution of 4-aminophenol”)
  - An equipment and glassware free description of the process (“the product was collected by vacuum filtration” NOT “the product was filtered using a Buchner funnel connected to the laboratory vacuum outlet”)
  - Included the structures of organic compounds in the reaction scheme
3. The “Result” should include all obtained experimental results and calculations
4. The “Discussion” should be ONE short paragraph of no more than 5 sentences
5. The “Conclusion” should be 1 to 2 sentences

The report should be able to be repeated by a trained scientist who is not necessarily an expert in the area in their own laboratory using the relevant apparatus.

For example:

*Acetic anhydride (5 mL, n mmol) followed by conc. H<sub>2</sub>SO<sub>4</sub> (5 drops) were added to salicylic acid (3 g, y mmol) and the mixture was heated at 60 – 70 °C for 15 min with frequent swirling. Water (30 mL) was then added slowly with vigorous swirling and the mixture was cool to room temperature. The solid, which crystallized out of the solution, was collected by vacuum filtration and recrystallized from aqueous ethanol to give colourless needles (2.7 g, x% yield) m.p. 129-130 °C.*

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |                                       |  |                                      |
|---|---------------------------------------|--|--------------------------------------|
| <input type="checkbox"/> Corrosive  | <input type="checkbox"/> Irritant     | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric   | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |                                       | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |                                       | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input checked="" type="checkbox"/> Other: Exposure to hot surfaces (steam bath)            |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- |  |
|--|
| <input type="checkbox"/> 3 Minimal risk  |
| <input type="checkbox"/> 2a Low risk (Fume hood recommended)                       |
| <input checked="" type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential) |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)                     |
| <input type="checkbox"/> 1b Significant risk (Special location or facility)        |

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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**THE OXIDATION OF MENTHOL:****8****FORMATION OF MENTHONE****AIMS OF THE EXPERIMENT**

- To oxidise the secondary ( $2^\circ$ ) alcohol, menthol to the ketone, menthone.
- To prepare the crystalline semicarbazone derivative of menthone and then purify this compound by recrystallisation.

**READING**

- Chemistry<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Working out the yield of a reaction: pages 24 – 25;
  - Oxidising alcohols to carbonyls: pages 1064 – 1065;
  - Reaction with nitrogen nucleophiles: pages 1077 – 1079.
- Chemistry, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:
  - Stoichiometry, limiting reagents and percentage yield: pages 93 – 99;
  - Reduction of aldehydes and ketones: pages 964 – 965.
- Techniques and instrumentation section of this laboratory manual:
  - Top-loading and analytical balances: page 127 – 128;
  - Laboratory equipment and glassware: page 129;
  - Solvent extraction using separating funnel: pages 137;
  - Filtration: page 132;
  - Purification of compounds: pages 133 – 134;
  - Criteria of purity of compounds: pages 135 – 136.

**PRE-LAB QUESTIONS**

There is a compulsory ChemCAL Prelabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

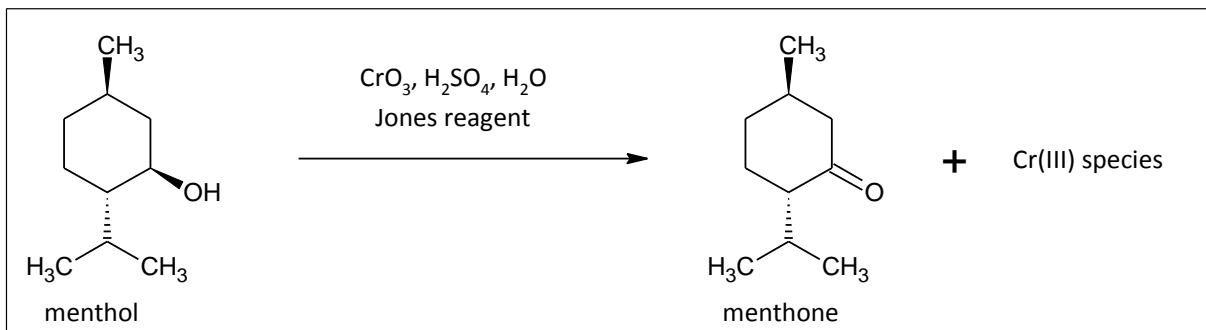
**INTRODUCTION**

For this experiment:

- Students work individually
- You will require a basic scientific calculator
- Part A and Part B are to be written up in “Experimental Method” of report (refer to page 75)

Menthol is a naturally occurring monoterpene isolated from peppermint, and other oils. It has commercial use in liqueurs, confectionery, perfumes, cough drops and nasal inhalers. It is also a mild local anaesthetic and counter-irritant. Menthone is another monoterpene found in volatile oils such as those from peppermint and geranium.

Menthone can also be prepared in the laboratory by the oxidation of menthol with chromic acid or sodium dichromate. *Common oxidising agents in the laboratory are  $KMnO_4$ ,  $CrO_3$  and  $Na_2Cr_2O_7$ .*



**Figure 8.1** Oxidation of menthol to menthone

The older type of ‘breathalyser’ is packed with  $Na_2Cr_2O_7$  crystals and an acid catalyst. When the drinker breathes his or her alcoholic ( $CH_3CH_2OH$ ) breath through the crystals the alcohol is oxidised and the Cr(VI) is reduced to Cr(III) *i.e.*, a colour change from yellow to green is observed. Consider the effect of menthol cough drops on one’s sobriety in the eyes of the traffic police!

## SAFETY

### Safety Warning:

**Caution: The steam and hot surfaces of steam baths can cause burns.**



Jones' Reagent is a very corrosive liquid. Semicarbazide hydrochloride is harmful if swallowed, inhaled or absorbed through the skin. Prevent contact these chemicals with the eyes and skin by wearing safety glasses and gloves.

Methanol, acetone and ethanol are volatile liquids, which are flammable. Avoid contact with flames or electrical equipment.

Methanol is toxic by skin absorption or breathing of vapours. Avoid breathing the vapour and avoid contact with the skin. It should be handled in the fume hood.

**Wear gloves at all time. If there is any spillage of methanol on gloves, remove immediately and replace with new gloves.**

### Disposal of Wastes

Please ensure that all organic wastes are disposed of in the appropriate waste containers. Do not dispose of any organic materials down the sinks.

### Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Preparation of Menthone

1. Dispense 10 mL of the menthol/acetone solution from the zippette (equal to 0.5 g menthol) into the conical flask provided and chilled the solution in an ice-water bath.
2. Add Jones' Reagent (**Important:** *Jones' Reagent is very corrosive: gloves must be worn*) in ca. 0.50 mL portions from a zippette. After each addition, swirls the flask in a bowl of ice water to cool and thoroughly mix the reactants.

*After each addition the mixture turns brown but this colour is soon discharged and a green sludge forms. The end-point (around 2 mL of Jones' reagent) is reached when the brown colouration persists for about ten minutes.*

3. Add enough sodium metabisulfite solution to discharge the brown colour into green colour (i.e., to destroy excess oxidant).
4. Dilute the mixture with water (30 mL) then transfer it to a separating funnel and extract the product into pentane (20 mL) (your demonstrator will explain how to do this, also refer to page 137).

Discard the *lower aqueous layer* into the beaker and wash the organic layer twice with water (ca. 20 mL each time).



Separating funnel

5. If an emulsion is produced, then add a small amount of the saturated NaCl solution provided and shake the separating funnel to break the emulsion.
6. Pour the organic solution into 50 mL beaker, add 2 to 3 boiling chips and evaporate most of the solvent on a steam bath<sup>†</sup> in the fume hood. A colourless oil (up to 1 mL) remains.

### Part B: Preparation of the Semicarbazone Derivative

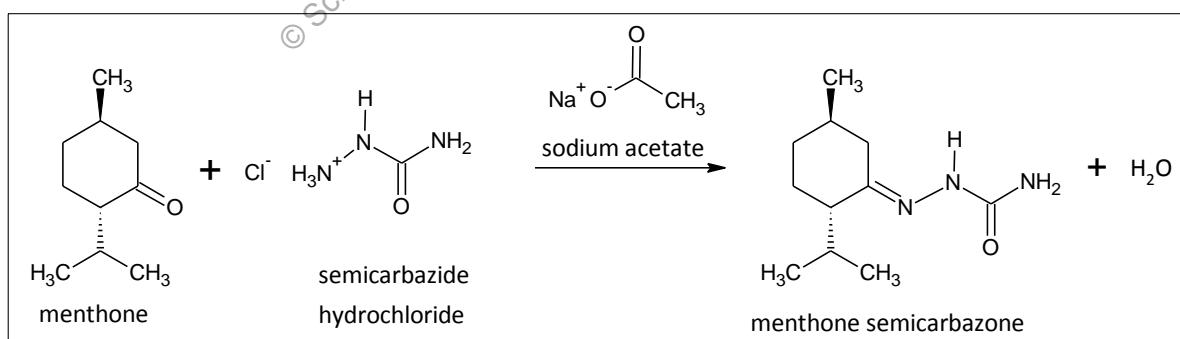


Figure 8.2 Formation of menthone semicarbazone

1. Add 5 mL of methanol to the beaker containing your crude menthone. Carefully transfer the resulting solution to the capped, clean plastic tube.

<sup>†</sup> It is important not to leave your solution of menthone in pentane on the steam bath for too long because significant losses of menthone (a relatively volatile compound) will occur. As a guide, if the boiling chips are no longer producing bubbles, then all the pentane has evaporated. Your demonstrator will give you guidance on this matter.

2. Into a second clean large test-tube dispense 5 mL of the semicarbazide hydrochloride aqueous solution from the zippette (equal to 1.0 g semicarbazide hydrochloride).

**Important:** gloves should be worn when handling semicarbazide hydrochloride solution).

Into the same test-tube dispense 2.5 mL of the sodium acetate trihydrate aqueous solution from the zippette (equal to 1.0 g of sodium acetate trihydrate).

3. Pour the solution into the capped plastic tube containing your crude menthone and methanol. Gently cap the plastic tube and shake it for ten minutes (*vigorously* for the first minute or two and release internal pressure), then allow it to stand at room temperature for ten minutes.
4. Finally, cool the plastic tube in ice-water bath for several minutes, quickly filter off the resulting crystals using a Hirsch funnel. Wash the crude product with a small volume of cold water and then dry it by pressing down on the crystals with a spatula.
5. Recrystallise (see pages 133 – 134) the crude menthone semicarbazone from hot ethanol (starts off by adding 6 – 7 mL but NO MORE than 8 mL) inside a test-tube. Swirl the test tube continuously and DO NOT allow the ethanol to boil and the recrystallisation process should take no more than 5 minutes.
6. Cool the test tube in ice-water bath and filter off the purified product, wash with small portions of chilled ethanol, press dry at pump with spatula and dry on a watch glass over a steam bath.
7. Whilst the product is drying, place a small amount on a filter paper and “flash dry” by heating on the edge of a steam bath for about two minutes. Use this sample to determine the melting point (>170 °C).
8. When dry, determine the yield of your product and calculate the theoretical and percentage yields based on the amount of menthol used. The percentage yield will be well below the theoretical yield due to the losses incurred during the two reaction steps and the recrystallisation.
9. Submit the product in a fully labelled snap lock plastic bag.



Hirsch vacuum  
filtration



MP determination

Student Name (i.e. Your Name)
Date, Group Number, Day
Compound name, structure or formula
M.P. Range (e.g. M.P. = 129 – 130 °C)
Yield, % Yield

**Figure 8.3** Sample label

**Question (to be included with your report)**

During the preparation of menthone, acetone (a ketone!) is used as a solvent. At the end of the procedure, the solvent is evaporated. If residual acetone is carried over to the next step, the semicarbazide may react with acetone as well as menthone, reducing the yield of the latter and potentially contaminating it with acetone semicarbazone. Draw the structure of acetone semicarbazone.

## **WRITING UP OF PREPARATIVE EXPERIMENTS**

Your report on the preparation of menthone semicarbazone, and any other preparative experiment, should follow the style used by leading chemical journals. Particularly, the report needs to be CONCISE:

1. The “Aim” should be 1 to 2 sentences
2. The “Experimental Method” should be no more than TWO short paragraphs of 3 to 4 sentences each:
  - Written in past tense and passive voice (“acetic anhydride was added to a solution of 4-aminophenol” NOT “I added acetic anhydride to a solution of 4-aminophenol”)
  - An equipment and glassware free description of the process (“the product was collected by vacuum filtration” NOT “the product was filtered using a Buchner funnel connected to the laboratory vacuum outlet”)
  - Included the structures of organic compounds in the reaction scheme
3. The “Result” should include all obtained experimental results and calculations
4. The “Discussion” should be ONE short paragraph of no more than 5 sentences
5. The “Conclusion” should be 1 to 2 sentences

The report should be able to be repeated by a trained scientist who is not necessarily an expert in the area in their own laboratory using the relevant apparatus.

*For example:*

*Acetic anhydride (5 mL, n mmol) followed by conc  $H_2SO_4$  (5 drops) were added to salicylic acid (3 g, y mmol) and the mixture was heated at 60–70 °C for 15 min with frequent swirling. Water (30 mL) was then added slowly with vigorous swirling and the mixture was allowed cooling to room temperature. The solid, which crystallized out of the solution, was collected by vacuum filtration and recrystallized from aqueous ethanol to give colourless needles (2.7 g, x% yield) m.p. 129–130 °C.*

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input checked="" type="checkbox"/> Other: Exposure to hot surfaces (steam bath)            |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- |  |
|--|
| <input type="checkbox"/> 3 Minimal risk  |
| <input type="checkbox"/> 2a Low risk (Fume hood recommended)                       |
| <input checked="" type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential) |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)                     |
| <input type="checkbox"/> 1b Significant risk (Special location or facility)        |

Risk Assessed by: Coordination: Sonia Horvat

Date: 6th January 2018

**RATES OF REACTION:****9****THE HYDROLYSIS OF *tertiary*-BUTYL CHLORIDE****AIMS OF THE EXPERIMENT**

- To determine the rate of a chemical reaction.
- To measure the kinetics of a first order reaction and to use the rate law to determine the rate constant for the reaction.
- To estimate the activation energy using the experimentally-determined temperature dependence of the rate constant of the reaction.
- To demonstrate that the conductivity of a solution can be used to follow the change in concentration of reactions that generate or consume ions.

**Note:**

You will require a basic scientific calculator, graph paper, a sharp pencil and a 30 cm ruler as well as your laboratory notebook.

**READING**

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Ions in solutions: Section 16.2, pages 730 – 733;
  - Rate equations: Section 9.4, pages 389 – 397;
  - Effect of temperature on the rate of a reaction: Section 9.7, pages 425 – 428.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:
  - Reaction Rates and factors that affect reaction rates: pages 642 – 647;
  - The integrated rate law: pages 655 – 658;
  - Half-life of first-order reactions: pages 658 – 659;
  - Activation energy and temperature effects-the Arrhenius equation: pages 665 – 670.
- Techniques and Instrumentation section of this laboratory manual: from page 127.

**PRELAB QUESTIONS**

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

**INTRODUCTION**

For this experiment:

- Students work in pairs
- The report is written individually in own words
- All calculations and graphing of data collected during the experiment must be completed individually by students

Tertiary-butyl chloride (*t*-BuCl) reacts with water (*hydrolyse*) to produce tertiary-butyl alcohol (*t*-BuOH) accordingly to Figure 9.1. The bulkiness of the reaction centre favours a unimolecular reaction path (*i.e.* dissociation of Cl<sup>-</sup> followed by entry of OH<sup>-</sup>).

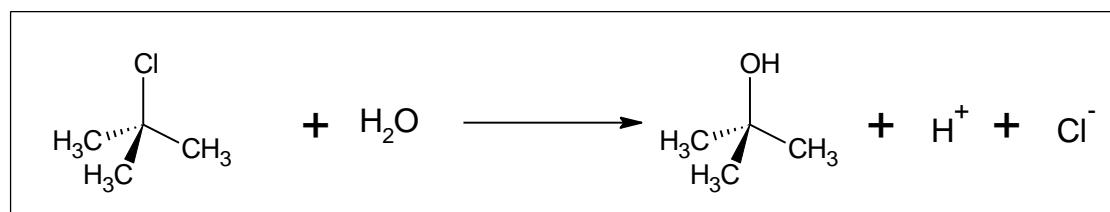
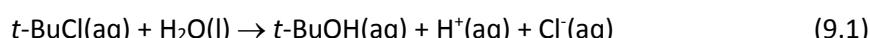


Figure 9.1: The hydrolysis of *tertiary*-butyl chloride (*t*-BuCl).

The equation for the hydrolysis can be written in shorthand form:



### Reaction rate:

The rate of the reaction may be followed by observing either the increase in concentration of H<sup>+</sup>, Cl<sup>-</sup> or *t*-BuOH or the disappearance of *t*-BuCl

$$-\frac{d[t\text{-BuCl}]}{dt} = +\frac{d[t\text{-BuOH}]}{dt} = +\frac{d[\text{H}^+]}{dt} = +\frac{d[\text{Cl}^-]}{dt} \quad (9.2)$$

The reaction (equation 9.1) is first order and its rate is accurately described by:

$$-\frac{d[t\text{-BuCl}]}{dt} = k[t\text{-BuCl}] \quad (9.3)$$

where ***k*** is the observed rate constant.

By integration this gives:

$$[t\text{-BuCl}]_t = [t\text{-BuCl}]_0 e^{-kt} \quad (9.4)$$

or, more conveniently,

$$\ln[t\text{-BuCl}]_t = \ln[t\text{-BuCl}]_0 - kt \quad (9.5)$$

where [t-BuCl]<sub>t</sub> is the concentration of *t*-BuCl at any time *t* during the reaction and [t-BuCl]<sub>0</sub> is the initial concentration of *t*-BuCl.

If a plot of ln[t-BuCl]<sub>t</sub> against *t* gives a straight line then the reaction is confirmed to be first order and the gradient of the line will be *-k* and the intercept, at *t* = 0, is ln[t-BuCl]<sub>0</sub>.

### Conductivity and kinetics

Hydrolysis of uncharged *t*-BuCl produces *t*-BuOH, H<sup>(aq)</sup> and Cl<sup>(aq)</sup>. Since the conductivity of the neutral molecules is zero the concentration of H<sup>(aq)</sup> and Cl<sup>(aq)</sup> can be determined by measuring the conductivity of the solution. (Conductivity is a measure of the ability of a substance to conduct electricity. Details of conductance and conductivity measurements can be found in Appendix 9.1 at the end of this experiment).

Initially, the conductivity,  $\kappa$  of the aqueous solution is very low and increases in proportion to the concentration of the  $H^+$  and  $Cl^-$  which are side products of the hydrolysis reaction. The conductivity reaches a maximum when the reaction is complete. At completion,  $t = \infty$ , all the  $t\text{-BuCl}$  present at the start of the experiment,  $[t\text{-BuCl}]_0$ , has reacted to form an equal amount of  $H^{(aq)}$  or  $Cl^{-(aq)}$ .

The conductivity at this infinity time,  $\kappa_\infty$ , is proportional to the amount of  $t\text{-BuCl}$  present at the start of the reaction,  $[t\text{-BuCl}]_0$ .

Since the conductivity,  $\kappa_t$  is proportional to the amount of  $t\text{-BuCl}$  which has reacted, we can write:

$$\begin{aligned}\kappa_\infty - \kappa_t &\propto [t\text{-BuCl}]_0 - [t\text{-BuCl}]_{\text{reacted}} \propto [t\text{-BuCl}]_t \\ \kappa_\infty - \kappa_t &= \text{Const} \times [t\text{-BuCl}]_t\end{aligned}$$

Thus

$$\ln(\kappa_\infty - \kappa_t) = \ln(\text{Const}) + \ln([t\text{-BuCl}]_t) \quad (9.6)$$

To apply equation 9.5, it is not necessary to know the actual *Const* or even the concentration of  $t\text{-BuCl}$  at any time, it is the rate of change of  $\ln(\kappa_\infty - \kappa_t)$  that gives the rate constant.

### Temperature effect on the reaction rate

It is generally found that the rate constant of a reaction increases with increasing temperature. The Arrhenius relationship (*Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 2<sup>nd</sup> ed. 2013: pages 421 – 424) describes this variation as

$$k_{(T)} = Ae^{-E_a/RT} \quad (9.7)$$

or

$$\ln(k_{(T)}) = \ln A - \left(\frac{E_a}{RT}\right) \quad (9.8)$$

where  $A$  is a constant and  $E_a$  is the activation energy.

At low temperatures, few molecules possess sufficient energy to react when they collide; however, at higher temperatures, many more molecules have enough energy and the reaction rate is consequently greater.

### Activation Energy $E_a$

The activation energy of a reaction is related to the enthalpy barrier for the transformation of the reactants to the products of the reaction. Each chemical reaction will have specific activation energy, where this can change accordingly to the environment, *e.g.* the polarity of the solvent.

Rearrangement of the Arrhenius equation (equation 9.8) shows that the temperature dependence of the reaction rate constant ( $k$ ) can be used to deduce the activation energy. Where a plot of  $\ln(k_{(T)})$  versus  $1/T$  should give a straight line with gradient equal to  $-(E_a/R)$ .

$$\ln(k_t) = \ln(A) - \left(\frac{E_a}{R}\right) \cdot \frac{1}{T}, \quad \text{i.e. gradient} = -\left(\frac{E_a}{R}\right) \quad (9.9)$$

[Note: use  $R = 8.314 \text{ Jmol}^{-1}\text{K}^{-1}$  for gas constant and the temperatures are in Kelvins].

Alternatively, if the rate constant is known at more than one, say two temperatures,  $T_1$  and  $T_2$ , then the activation energy can be obtained from the relationship,

$$\ln\left(\frac{k_{(T_2)}}{k_{(T_1)}}\right) = -\left(\frac{E_a}{R}\right) \cdot \left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad (9.10)$$

This relationship is commonly expressed as,

$$\ln(k_{(T_2)}) - \ln(k_{(T_1)}) = -\left(\frac{E_a}{R}\right) \cdot \left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad (9.11)$$

### Measurement of Conductivity

**Direct Reading Conductivity Meters:** The conductivity is measured at regular intervals during the reaction and should increase with time until all the *t*-BuCl has been converted to *t*-BuOH. The conductivity, in units of micro-Siemens per centimetre ( $\mu\text{S cm}^{-1}$ ), is displayed directly and the conductivity meters have been pre-calibrated.

**Conductivity Cell:** The conductivity cell used in this exercise consists of a probe which sits inside a glass vessel. The probe is fitted with a pair of specially prepared platinum electrodes, through which electrical contact with the solution is made. The probe may be removed for the purposes of rinsing and filling the conductivity cell.

## SAFETY

### Disposal of Chemical Waste:

All waste solutions from this experiment contain ethanol and *t*-butyl compounds and must be placed in the organic residue jars provided.

### Risk Assessment:

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the risk assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Reaction rate measurements

The rate of the hydrolysis reaction of *t*-BuCl depends on the polarity of the solvent and is inconveniently fast in pure water (the half-life of the reaction is about 20 seconds). The reaction rate is greatly slowed down in the presence of ethanol and we shall therefore study the reaction in a mixture of water, 61% and ethanol, 39% (by volume). *It is important that the composition of the solution used for your measurements does not change when you are transferring solutions between containers or to the conductivity cell.*

The rate of the hydrolysis will be measured by each pair of students at either 25 °C, 30 °C, 35 °C or 38 °C. Your demonstrator will let you know the temperature you are required to use for your experiment. The water bath for the hydrolysis reaction is nominally set to the required temperature however you should measure the actual temperature to the nearest 0.1 °C.

The set of solutions provided consists of:

- $10^{-2}$  M solution of *t*-BuCl in pure ethanol at room temperature.
- Ethanol-water solution (39% by volume of ethanol) in water bath adjusted to the required temperature.

**NOTE:** The conductivity cells are made of glass and must be handled with care.

Make sure no air bubbles are trapped in the cells when making conductivity measurements.

For good graphing technique:

- landscape format
- graph occupy >80% of ordinate and coordinate range
- label axes with correct scales and units
- include a title
- draw the line of best fit
- show the data points used for gradient calculation

## 1. Hydrolysis run at 0.5 °C

- i. The conductance measurements for a kinetics run carried out at a temperature of 0.5 °C is given in Table 9.1 which is given at the end of the experimental procedure. Use this data to calculate the rate constant  $k$  at 0.5 °C (equations 9.5 and 9.6). This value of the rate constant will be used in your estimate of the activation energy for the reaction.
- ii. It is strongly advised that you can calculate and graph the results of the 0.5 °C kinetics run BEFORE you come into the laboratory to do the experiment.
- iii. All calculations and graphing of data collected during the experiment must be completed individually by students.

## 2. Reaction mixture for your kinetic run

*Note: It is important to be able to obtain reliable kinetic results within a ¼ half-life. At higher temperatures, the half-life of the reaction is about 8 mins so the first reading is made at 2 minutes after mixing *t*-BuCl with water. It is important for students to work effectively together. The LabByte shows the preparation of the solution and filling of the conductivity cell and the flowchart, Figure 9.2, on the following page will help your planning of the experiment.*

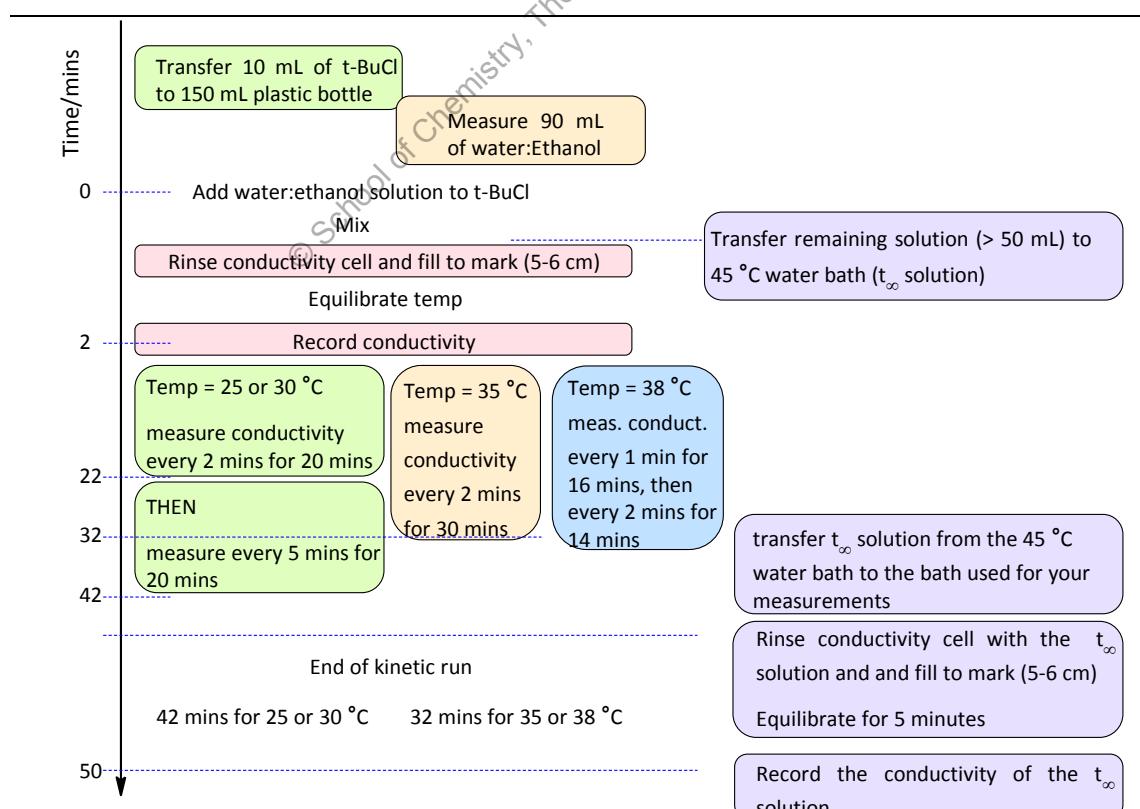
- i. Collect the cap and numbered, DRY 150 mL plastic bottle from pin-dryer. **Make sure you remember your bottle number.**
- ii. Dispense 10 mL of the *t*-BuCl stock solution, via the zippette, into the plastic bottle and cap.
- iii. Equilibrate the solution in your water bath for 5 minutes.
- iv. Using a measuring cylinder, add 90 mL of the ethanol-water mixture equilibrated at the temperature of your measurement to the *t*-BuCl solution and **start the stop-clock immediately. [Note: This is taken as the zero Time (t = 0 seconds) at the temperature (T) of your hydrolysis run.]**
- v. Shake well to mix.



Preparing  
Solutions

- vi. While your partner is preparing the solution (Parts iv. and v.), empty the conductivity assembly.
- vii. Then rinse the conductivity assembly twice with the reaction mixture (from Part v.). Approximately 5 mL is sufficient for each rinse.
- viii. Fill the cell with the reaction mixture well above the electrode and place it in the water bath at pre-set temperature.
- ix. **Cap the plastic bottle containing the remaining reaction mixture (should be about 50 mL) and place in the water bath set at 45 °C (this will be your  $t_\infty$  solution).**
- x. Make your first measurement of the conductivity at time = 2 minutes (the conductivity cell should have been equilibrating for about 1 minute). **Record your results in a pre-prepared table in your laboratory notebook.**
- xi. Make readings of conductivity,
  - 25 or 30 °C:** at two-minute intervals for the first 20 minutes, then at 5 minute intervals for a further 20 minutes.
  - 35 °C:** at two-minute intervals for the 30 minutes of the kinetic scan.
  - 38 °C:** at one-minute intervals for the first 16 minutes, then at 2 minute intervals for a further 14 minutes.

Overall the readings are taken over 40 minutes (25 or 30 °C) or 30 minutes (35 or 38 °C) of times period.



**Figure 9.2:** The flow chart for the hydrolysis run.

### 3. Readings of conductivity of your $t_\infty$ solution ( $\kappa_\infty$ )

Description of the measurement procedure and outline of the importance of conductivity at  $t_\infty$  ( $\kappa_\infty$ ) can be found in the LabByte, a discussion of the impact on the analysis is given in Appendix 9.2 at the end of this experiment on page 87.



#### $\kappa_\infty$ Measurement

- i. Once your  $t_\infty$  solution (the remaining reaction solution) has equilibrated in the 45 °C water bath for 30 minutes move the plastic bottle from the 45 °C water bath to the water bath used for **your** kinetic measurements (25 or 30 or 35 or 38 °C) and equilibrate for a further 10 minutes.
- ii. Empty the conductivity assembly upon the completion of the hydrolysis run. Rinse twice with the  $t_\infty$  solution. *Remember to gently open the plastic bottle as pressure can build up inside during the reaction time.*
- iii. Rinse the conductivity cell (twice, but less than 5 mL for each rinse) and fill the conductivity cell with your remaining  $t_\infty$  solution.
- iv. Once the temperature in the conductivity cell has stabilised, measure and record the conductivity ( $\kappa_\infty$ ). This usually takes no more than 5 minutes.

#### **Discussion 1:**

Comment on any difference between the measured conductivity of your  $t_\infty$  solution,  $\kappa_\infty$ , and the final conductivity measurement *made* at completion of your hydrolysis run.

### 4. End of experiment

- i. Leave the  $t_\infty$  solution and the probe in the conductivity assembly.
- ii. DO NOT rinse the measuring cylinder.
- iii. Rinse the 150 mL plastic bottle with 5 mL ethanol and place on the pin-dryer ready for next group to use.

#### **Part B: Graphing data**

1. For the data at each temperature (0.5 °C and your water bath temperature), use your  $\kappa_\infty$  and the successive  $\kappa_t$  value to plot  $\ln(\kappa_\infty - \kappa_t)$  against *time* (in seconds). You should construct separate graphs for each temperature (the scales should be different). This will provide more precise results for your rate constant determination.
2. Draw the line of best fit through your calculated data points. If you are excluding some of your measurements clearly indicate the data used to estimate the slope.
3. Use equations 9.5 and 9.6, together with your results to determine the first-order rate constant.

#### **Discussion 2:**

Inspect the two sets of data. Does the reaction rate increase with temperature?

### Part C: Reaction half-life ( $t_{1/2}$ ) and activation energy ( $E_a$ kJmol $^{-1}$ )

- What is the relationship between the rate constant  $k$  for a first order reaction and half-life ( $t_{1/2}$ )?  
Calculate the half-life for the reaction at:
  - 0.5 °C and
  - the temperature of your hydrolysis measurement.

*Rate constants measured at five different temperatures will be used in the determination of activation energy ( $E_a$ , equation 9.8) for the t-BuCl hydrolysis reaction.*

*This includes the rate constant derived from the analysis of the temperature = 0.5 °C data and rate constants at 25 °C, 30 °C, 35 °C and 38 °C determined by the different pairs of students in your practical group.*

- Calculate  $\ln(k_{(T)})$  and  $1/T$  (K $^{-1}$ ) for your kinetic run at allocated temperature and for your analysis of the temperature = 0.5 °C data.
- These  $\ln(k_{(T)})$  and  $1/T$  data pairs from your practical group will be listed on the whiteboard at the demonstrator station.
- Your demonstrator will plot all  $\ln(k_{(T)})$  versus  $1/T$  data pairs from your practical group using the available EXCEL plotting program on computer in laboratory. The best straight line will be drawn and its gradient will be determined using the fitting function of the plotting program.
- Derive the activation energy including correct unit from the determined gradient using equation 9.9 ( $-E_a/R$  with  $R = 8.314 \text{ Jmol}^{-1}\text{K}^{-1}$ ).

Time (sec)	$\kappa_t$ ( $\mu\text{S}/\text{cm}$ )	$\kappa_\infty - \kappa_t$ ( $\mu\text{S}/\text{cm}$ )	$\ln(\kappa_\infty - \kappa_t)$
0	2.99		
360	3.75		
720	4.59		
900	4.96		
1200	5.63		
1800	6.96		
2400	8.26		
3000	9.54		
3600	10.73		

**Table 9.1:** Data for hydrolysis run at temperature = 0.5 °C  
( $\kappa_\infty$  for this experiment is 80.00  $\mu\text{S}/\text{cm}$ ).

## APPENDIX 9.1

### Conductance and conductivity

A material conducts electricity to an extent described by its conductance,  $G$ , which is the reciprocal of its resistance,  $R$ . If a voltage  $V$  is applied to a piece of material and a current,  $I$ , flows (Fig. 9.3A), then Ohm's law states:

$$\frac{1}{R} = G = \frac{I}{V}$$

$G$  will depend on the dimensions of the material, particularly with its length  $l$ , and its cross-sectional area  $A$  (Fig. 9.3B); in fact,

$$G = \left(\frac{A}{l}\right) \kappa$$

The proportionality constant  $\kappa$  is known as the conductivity of the material. It is the conductance of a block, 1 m long and 1 m<sup>2</sup> in cross-sectional area.

$\kappa$  enables us to compare the intrinsic conducting power of one substance with another, free of the influence of sample size and shape.

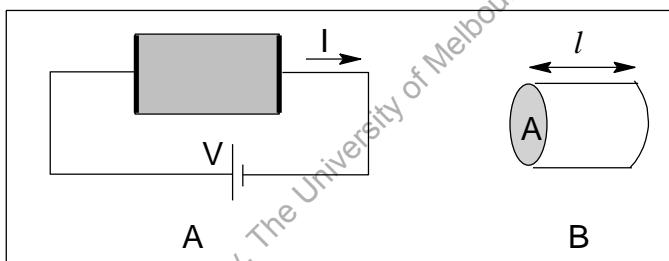


Figure 9.3: Conductance

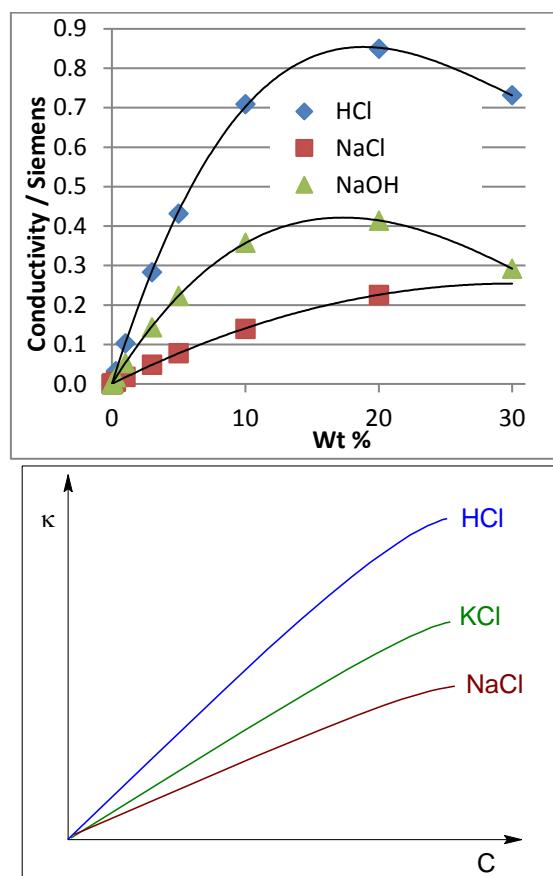
#### Properties of $\kappa$ for electrolyte solutions

Strong electrolytes, *e.g.* salts such as NaCl, when dissolved in water completely dissociate into ions and give highly conducting solutions. The conductivity of such a solution depends on the concentration of the ions, the number of charges on each ion, and the speed at which they move (mobility) when an electric field is applied.

The variation of conductivity  $\kappa$  with concentration,  $C$ , for several electrolytes is shown in Fig. 9.4. At very high concentrations the conductivity becomes non-linear, mostly a result of ion pairing. At lower concentrations, the graphs are nearly linear but of different slope for different electrolytes. To compare the conducting power of different electrolytes we need to factor out the effect of concentration by defining the molar conductivity or conductivity per unit concentration,

$$\Lambda = \frac{\kappa}{C}$$

Note that, as curvature in Fig. 9.4 implies,  $\Lambda$  is only approximately a constant for any electrolyte, and tends to fall off at higher concentration, *i.e.* the conductivity per unit concentration of ions is less in higher concentration solutions where the ions are moving less freely. The limiting value of  $\Lambda$  at low concentration is often listed in tables as  $\Lambda^\circ$  'the molar conductivity at *infinite dilution*'



**Figure 9.4:** Conductivity of various electrolyte solutions (top – experimental for concentrated solutions, bottom – schematic)

contributes to the total conductivity simply in terms of its concentration and mobility and is independent of the other ions present (while maintaining overall electrical neutrality).

### Conductivity Cell Calibration

As discussed earlier, the conductance of a solution will depend on the dimensions of the electrodes and their separation distance, whilst the conductivity is a measure of the intrinsic conducting power of the solution. To measure the conductivity of a solution rather than its conductance we need to know the ratio  $I/A$  for the conductivity cell (see Fig. 9.3). It is not easy to make conductivity cells of known dimensions; however, any cell can be calibrated easily. Cells of very accurately known dimensions have been used to measure precisely the conductivity  $\kappa$  of KCl solutions and these solutions are now used as international standards. The ratio  $I/A$  (see Fig. 9.3) for any cell is called the cell constant,  $K$ , and can be determined using a standard KCl solution.

$$K = \frac{\kappa}{G}$$

The unit of conductance  $G$  is the Siemen,  $S$  ( $\equiv \text{ohm}^{-1}$ ).

Conductivity  $\kappa$  has the unit  $\text{Sm}^{-1}$ , and molar conductivity  $L$  has the unit  $\text{S m}^2\text{mol}^{-1}$  (For KCl at 0.020 M,  $\kappa = 2.765 \times 10^{-5} \text{ S m}^{-1}$  at 25 °C).

Note also that the  $\kappa$  values for the three chlorides are all different;  $\text{HCl} > \text{KCl} > \text{NaCl}$ . The anion is common in all these solutions so the differences indicate that the cations have different mobilities, i.e.  $\text{H}^+ > \text{K}^+ > \text{Na}^+$ . (Similar observations would be made if you studied solutions of  $\text{NaNO}_3$ ,  $\text{NaCl}$ , and  $\text{NaBr}$ ; common cation, various anions).

Observations such as this led to the idea of the independent migration of ions.

The molar conductivity of an electrolyte can be expressed as the sum of contributions of its individual ions;

$$\text{e.g. for NaCl: } \lambda(\text{NaCl}) = \lambda_+ + \lambda_-$$

where  $\lambda_+$  and  $\lambda_-$  are molar conductivities of the sodium cation and chloride anion, respectively.

For a general electrolyte  $\text{A}_x\text{B}_y$ :

$$\lambda(\text{A}_x\text{B}_y) = x \cdot \lambda_+ + y \cdot \lambda_-$$

e.g. for aluminum sulfate,  $\text{Al}_2(\text{SO}_4)_3$ :

$$\lambda(\text{Al}_2(\text{SO}_4)_3) = 2\lambda(\text{Al}^{3+}) + 3\lambda(\text{SO}_4^{2-})$$

This means that in dilute solution each ion type

## APPENDIX 9.2

### The importance of having a good estimate of $\kappa_\infty$

The  $t_\infty$  solution is *your* reaction mixture after it has had time to react to completion and its conductivity is the infinite reading  $\kappa_\infty$ . This quantity is used for the calculations of  $\ln(\kappa_\infty - \kappa_t)$  in equation 9.6 (note that  $(\kappa_\infty - \kappa_t)$  is proportional to  $[t\text{-BuCl}]_t$ ).



$\kappa_\infty$  Measurement

**The impact of  $\kappa_\infty$  on the analysis** is most clearly shown by modelling the experimental data.

Programs such as *Excel* provide a are very convenient vehicle, consider the growth of a product from a reaction with a first-order reaction with  $k=0.008 \text{ s}^{-1}$  with  $\pm 0.5\%$  noise [*Excel* formula is given by:  $[\text{Product}] = 1-\text{EXP}(-0.008*\text{time})+0.02*(\text{RAND}()-0.5)$ ]. If the conductivity of the solution is:

**Constant** $\times$ [Product] – where the **Constant** is set to 100, for convenience. A plot of the data is shown in Fig. 9.5.

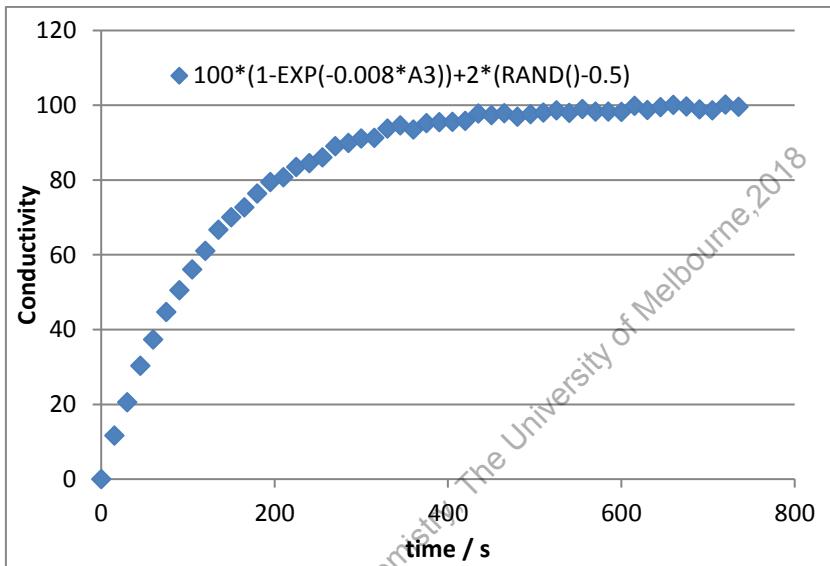


Figure 9.5: The graph of  $\kappa_t$  ( $\mu\text{Scm}^{-1}$ ) versus Time  $t$  (s) for a first order reaction.

This data is then used to calculate  $\ln(\kappa_\infty - \kappa_t)$  where the value of  $\kappa_\infty$  is exactly correct ( $\kappa_\infty = 100$ ) or 3% below or above the correct value ( $\kappa_\infty = 97$  and  $103$ , respectively). The resulting plots of  $\ln(\kappa_\infty - \kappa_t)$  vs. time are shown in Fig. 9.6.

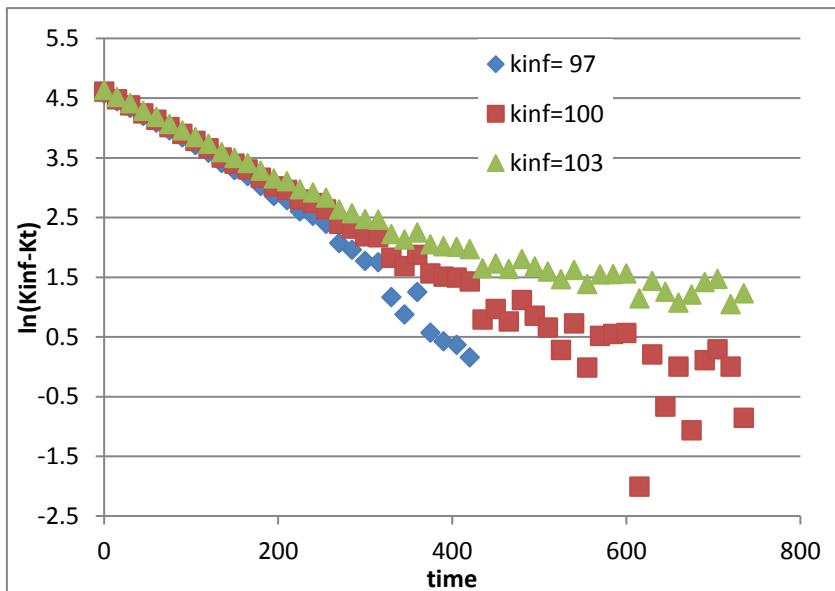


Figure 9.6: The graph of  $\ln(\kappa_\infty - \kappa_t)$  versus Time  $t$  (s) for a first order reaction.

The plot of  $\ln(\kappa_\infty - \kappa_t)$  versus time should be linear, but as  $(\kappa_\infty - \kappa_t)$  values approach to zero, any error in  $\kappa_\infty$  will introduce nonlinearities in the plot. This effect becomes significant when the measured value is comparable with the noise in the measurement. For the current experiment this occurs at times longer than about 3 half-lives (Fig. 9.6).

Notes:

- If a low or high value of  $\kappa_\infty$  is used then the plot of  $\ln((\kappa_\infty - \kappa_t))$  becomes non-linear and trends to lower or higher slopes at longer times (Fig. 9.6 at times longer than *ca.* 200 s).
- Even with an error of 3 – 5% the slope (rate constant) is quite well determined, but even if the  $\kappa_\infty$  is correct the data become noisy at longer times (this is expected – of course).
- While not shown in this analysis, students need to recognise that the data may be unreliable at short times (as the system comes to thermal equilibrium). In the order to extend the useful linear region of the graph it is important to prepare solutions rapidly and to ensure that the system quickly achieves thermal equilibrium. The instructions in the manual and the LabByte provide tips on how to do this. This issue is most important when there is a higher rate of reaction (higher temperatures).

### How to get a good estimate of $\kappa_\infty$ ?

At lower temperatures, such as 25 °C, about 10% of the t-BuCl will remain unreacted at the end of 45 minutes (3 – 4 half-lives). A further 60 minutes is required for 99% of the t-BuCl added initially to be hydrolysed. This makes it impossible to obtain  $\kappa_\infty$  from the actual solution used for your kinetics measurements in the allocated time during laboratory session.

The rate of reaction has been shown to vary directly with temperature according to the Arrhenius Equation (equation 9.7). In the experiment your t-BuCl reaction mixture is driven to completion within a 30 minute of period by warming the solution to 45 °C. This will produce a  $t_\infty$  solution. In the order to have a conductivity reading that is compatible with your kinetic measurements the temperature of the  $t_\infty$  solution must be equilibrated to the temperature of your kinetic measurements. Errors in the conductivity of the  $t_\infty$  solution could be attributed to:

- Change in the concentration of the hydrolysis products of *t*-BuCl as the result of solvent evaporation during reaction time at an elevated temperature.
- Dilution or contamination by any solvent being present in the conductivity assembly when measuring the solution conductivity.
- Poorly equilibrated or mixed solutions, e.g. insufficient time for infinity solution to reach thermal equilibrium at the pre-set temperature for the hydrolysis run.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input type="checkbox"/> Corrosive  | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input checked="" type="checkbox"/> Minimal risk   |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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**THE PARTICLE IN THE BOX:****10****PRACTICAL EVIDENCE OF THE “PARTICLE IN THE BOX” CONCEPT****AIMS OF THE EXPERIMENT**

- To use of a bench-top UV-Visible spectrophotometer to determine the electronic absorption spectra of different molecules.
- To develop an understanding of absorption spectroscopy and its relation to the colour observed in chemical compounds.
- To calculate the energy of electronic systems using the “particle in a box” approximation.
- To make a comparison between experiment and theory, and to develop a tangible understanding of some quantum mechanical concepts.

**Note:****You MUST BRING a virus-free USB stick (1 GB to 2 GB Memory is sufficient).**

You will require a computer, basic scientific calculator as well as your laboratory notebook.

**READING**

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:
  - Electromagnetic radiation and quantization: Section 3.2, pages 117 – 124;
  - Atomic spectra and the Bohr atom: Section 3.3, pages 124 – 131;
  - The nature of the electron: Section 3.4, pages 132 – 134;
  - Wave functions and atomic orbitals: Section 3.5, pages 134 – 142;
  - Molecular energies and spectroscopy: Section 10.2, pages 453 – 457.
  - The Beer-Lambert Law: Section 10.3, pages 460 – 462.
  - Ultraviolet-visible spectrophotometry: Section 11.4, pages 543 – 545.
- This laboratory manual:
  - Guidelines for writing a laboratory report: pages 11 – 12;
  - Safety in the laboratory: page 13;
  - Volumetric flasks and pipettes: page 130;
  - Treatment of error in experimental data: pages 140 – 144.

**PRELAB QUESTIONS**

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## REPORT SUBMISSION USING TURNITIN FACILITY ON LMS

For this practical report:

1. You will submit your report electronically using the TurnItIn facility on the Learning Management System (LMS). You will be given a date to view your report mark and feedback given by your demonstrator on Turnitin.

**The deadline for report submission will be given at your practical session. You are still allowed submitting the report after the due date but there will be a penalty of ONE MARK for each late day. There is NO allowance for re-submission of report on Turnitin.**

2. The report can be typed using your computer or hand written into your laboratory notebook.
3. Prepare your report including the signed Cover Sheet and graphs in **Portrait Format** and save as **ONE pdf file** before submission.
4. If your report is hand written:
  - you can scan your written report, signed Cover Sheet and graphs and save as **ONE pdf file** in **Portrait Format** before submission
  - you can take photographs pf your report together with the signed Cover Sheet and graphs, copy into a WORD file and save as **ONE pdf file** in **Portrait Format** before submission
  - **DO NOT submit as ZIP files**
5. Log onto LMS using your Username and Password
6. Go to 'Laboratory Information':
  - select 'Turnitin Experiment 10: Particle in the Box'
  - select your day of practical e.g. 'Monday'
  - select your session of practical e.g. 'AM sessions'
  - select your practical allocation e.g. 'Monday 10:00am / Group 1 (Demonstrator name)'
  - double click 'view/complete'
  - go to 'Author' and enter your name into appropriate boxes
  - follow the prompts to upload and submit your report
  - a 'Confirmation Number' will be issued for successful report submission
7. If you cannot submit your report using your own laptop particularly:
  - save the pdf file of your report to a USB
  - submit using another computer

**NOTE: If you have submitted your report into the wrong group or have difficulties with report submission, you can contact Dr. Alice Lamb at [aclamb@unimelb.edu.au](mailto:aclamb@unimelb.edu.au).**

## INTRODUCTION

For this experiment:

- Students work in pairs
- All calculations and graphing of data collected during the experiment must be completed individually by students
- You will complete the experimental aspects in the laboratory during practical session
- You can complete the calculations and preparation of your report outside your practical time slot and it **should take no more than 90 minutes**

### “Particle-in-a-One-Dimensional Box” (P1DB) model

Molecules (or atoms) of a coloured material absorb visible light photons causing an electron to be promoted in energy from the lowest energy electronic state (i.e. the “ground state” electron configuration) of the atom or molecule to a higher energy electronic state (the “excited state”). The absorbed wavelengths of light correspond to transitions between these electronic states that occur as the result of selective photon absorption. The colours observed from a given sample result from the wavelengths of light that are **not** absorbed by the sample i.e. the colours that are reflected by, or transmitted through the sample.

For a molecule to absorb a photon, the energy of the incident photon must be equal to the energy difference between the initial state and some higher energy state of the molecule. Various other factors also influence the likelihood of the absorption process. The difference in energy between the two energy levels is:

$$\Delta E (J) = E_{\text{upper state}} - E_{\text{lower state}} \quad (10.1)$$

$$= E_{\text{photon}} = h\nu = \frac{hc}{\lambda} \quad (10.2)$$

where  $h$  is Planck’s constant ( $6.63 \times 10^{-34}$  J s and  $1 \text{ J} = 1 \text{ kg m}^2 \text{ s}^{-2}$ ),  $\nu$  is the frequency of the radiation ( $\text{s}^{-1}$  or Hz),  $c$  is the speed of light ( $3.0 \times 10^8 \text{ m s}^{-1}$ ) and  $\lambda$  is the wavelength of the radiation (m).

Each energy level can be described quantum mechanically in terms of a “wavefunction”,  $\psi$ . The “Particle-in-a-One-Dimensional Box” (P1DB) model is useful for illustrating how Schrödinger’s wave equation:

$$\hat{H}\psi = E\psi \quad (10.3)$$

or

$$\frac{-\hbar^2}{2m} \frac{d^2\psi}{dx^2} = E\psi \quad (10.4)$$

can be set up and solved for a simple, well-defined system. The P1DB interpretation allows a hands-on experience of a molecular and chemistry-based approach to quantum mechanical concepts helping identify the terms involved in this equation and particularly, the concept of wavefunctions and their corresponding energies.

The problem is set up as follows with a particle assumed to be confined within a one-dimensional “box” of length  $L$ . Inside the box, potential energy is zero i.e. the particle has only kinetic energy inside the box. While outside the box, the potential energy is infinite and the particle does not exist outside the box. The solutions to this problem predict that the energy,  $E_n$ , corresponding to each wavefunction as a function of position within the box,  $\psi_n(x)$ , can be calculated using:

$$E_n = n^2 \frac{\hbar^2}{8mL^2} (J) \quad (10.5)$$

$$\psi_n(x) = \sqrt{\frac{2}{L}} \sin \left[ \frac{n\pi x}{L} \right] \quad (10.6)$$

where  $n = 1, 2, 3, \dots, \infty$  (i.e. the energy levels),  $m$  is the mass of the “particle” (kg) and  $L$  is the length of the “box” (m). This indicates that the energy of the levels increases as the square of the principal quantum number,  $n$ , and inversely with the square of the length of the box,  $L$ .

### Interpretation of energy levels

The difference in the energy,  $\Delta E$ , between any two levels,  $n \rightarrow n+1$ , can therefore be calculated for a given box length. For example, the energy difference between levels 1 and 2 would be:

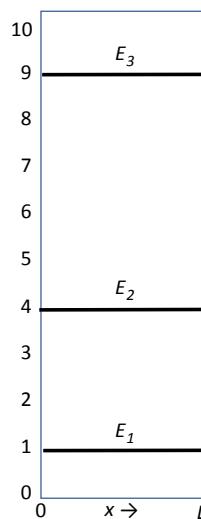
$$\Delta E_{1 \rightarrow 2} = E_2 - E_1$$

$$\Delta E = 2^2 \frac{h^2}{8mL^2} - 1^2 \frac{h^2}{8mL^2}$$

or between 1 and 3:

$$\Delta E_{1 \rightarrow 3} = 3^2 \frac{h^2}{8mL^2} - 1^2 \frac{h^2}{8mL^2}$$

The P1DB can be useful when describing *some* molecular systems. If only the particle-like nature of the electron is considered and a situation is envisaged where the electron is “delocalised” but constrained within a limited region of space, this lead to a situation analogous to the P1DB. A conjugated  $\pi$ -electron system, like the conjugated dyes used in this experiment, can be described approximately this arrangement.



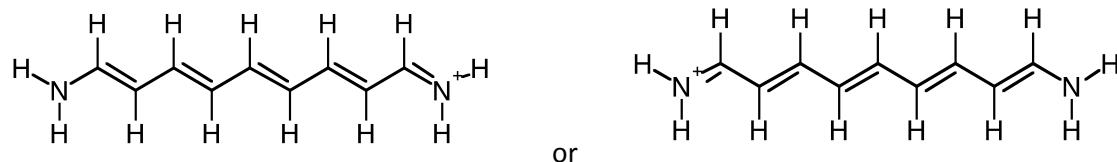
The Pauli exclusion principle limits the number of electrons in any given energy level ( $n$ ) to two, and these two electrons must have opposite spins: i.e.  $m_s = +\frac{1}{2}$  and  $-\frac{1}{2}$ . Using the Aufbau principle, in the ground state of a molecule with the *number of electrons*, these electrons will fill the lowest energy levels up to:

$$n = \frac{\text{number of electrons}}{2} \quad (10.7)$$

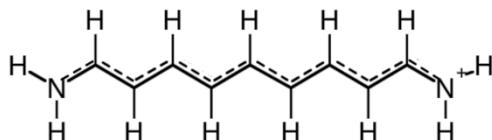
and all higher levels will be empty. For example, if the *number of electrons* = 6, electrons will fill up to  $n = 3$  energy levels, which is known as the highest occupied orbital. When the molecule absorbs light, one electron jumps from the highest filled level, i.e.  $n_1 = (\text{number of electrons}/2)$  to the lowest empty level, i.e.  $n_2 = [(\text{number of electrons}/2) + 1]$ . The energy change for this transition is:

$$\Delta E_{1 \rightarrow 2} = \frac{h^2(n_2^2 - n_1^2)}{8mL^2} = \frac{h^2(2n_1 + 1)}{8mL^2} \quad (10.8)$$

In a “polyene” molecule, each carbon atom participates in both  $\sigma$  and  $\pi$ -bonds, with each p-orbital containing one electron. One way of representing this is a series of alternating single and double bonds along the carbon atom chain:



However, this is better represented as a resonance structure, where each Carbon – Carbon bond shows “ $\frac{1}{2}$  bond” character or a Carbon – Carbon bond order of 1.5:



The electrons in the  $\pi$ -bonds of polyenes can therefore be considered to be delocalised over all the Carbon atoms of the conjugated chain, and can be thought of as moving somewhat freely along the length of the chain.

If the electrons are distributed into the various available orbitals in increasing energy, the lowest energy electronic transition will correspond to the promotion of the electron from the highest energy occupied orbital to the lowest energy unoccupied orbital. This somewhat mimics the P1DB model where the electrons represent the “particle” confined within the “box” that is defined by the conjugated molecular structure of the chain of Carbon atoms. If the “length of the box” could be changed, then the change in energy in going from level  $n \rightarrow n+1$  will differ and the sample should absorb different wavelengths of light (and thus appear a different colour).

Combining equations 10.2 and 10.8 gives:

$$\lambda = \frac{8mcL^2}{h(2n_1+1)} \quad (10.9)$$

This allows the calculation of the predicted wavelength of light absorbed ( $\lambda_{\text{predicted}}$ ). The equation directly relates the absorption wavelength ( $\lambda$ ) to the length of the box,  $L$ , the mass of the electron,  $m$ , and the *number of electrons* in the  $\pi$  electron system. Where  $c$  is the speed of light and  $h$  is the Planck’s constant.

This model is remarkably successful in modelling the quantum mechanical behaviour of many compounds such as polyenes and nanocrystals, and gives relatively good agreement with the experimentally observed absorption wavelength ( $\lambda_{\text{max}}$ ).

In this exercise the visible absorption spectra of a series of conjugated molecules of varying conjugation length will be examined, and the energies of the transitions observed experimentally will be compared to the one-dimensional quantum mechanical solutions given by the ‘particle-in-a-box’ approximation (equation 10.9).

## SAFETY

### Safety Warning:



Goggles or safety glasses must be worn at all time.

Nitrile gloves must be worn at all time.

**Methanol is highly flammable and is highly toxic. If there is any spillage of methanol on gloves, remove immediately and replace with new gloves.**

### Disposal of Chemical Wastes:

Empty any unused dyes solutions into waste container.

Empty the contents of cuvettes into waste container.

## Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

You will be provided with three conjugated dyes and their structures are given below in Figure 10.1. The *square brackets around the molecule below* shows the region of the “box”, where the electron is delocalised over the Carbon atoms between the two Nitrogen atoms.

### Number of participating Carbon atoms ( $p$ )

Each Carbon atom, between the two Nitrogen atoms, contributes  $1\pi$  electron to the conjugated system. These are termed participating Carbon atoms, and the number of participating Carbon atoms is designated  $p$ . For example, for 1,1'-diethyl-2,2'-cyanine iodide,  $p = 3$ .

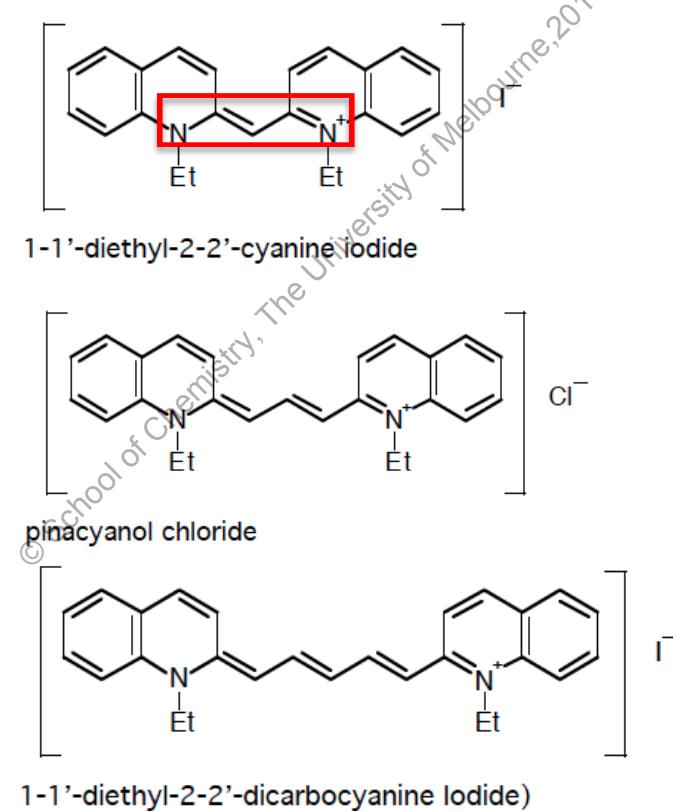


Figure 10.1: Conjugated cyanine compounds to be studied

### **Length of the box ( $L$ )**

The conjugated system is not linear but in fact "zigzag" and the displacement of the electron follows the zigzag path. The length of the conjugated chain can be used as an estimate for the length of the box ( $L$ ) and should therefore be taken as "*the length of the polymethine zigzag chain between the Nitrogen atoms plus one bond distance to either side*". The purpose of including a "bond distance" on either side of the Nitrogen atoms is to include the "distance" occupied by the lone pair of the Nitrogen.

To calculate the length of the box ( $L$ ), given that  $p$  is number of participating carbon atoms,  $(p + 1)$  will give the number of bonds between the Nitrogen atoms. Then add one extra bond on either side of the Nitrogen atoms and it gives:

$$L = (p + 3) \times (\text{average Carbon – Carbon bond length}) \quad (10.10)$$

assume an average value for Carbon – Carbon bond length = 139 pm (1 pm =  $10^{-12}$  m) and  $p$  is the number of participating Carbon atoms in the molecule.

### **Number of electrons in the box**

From this simple approximation of "Particle in the Box:" model, it can be assumed that each Carbon atom between the two Nitrogen atoms contributes one  $\pi$  electron to the system. In addition, each Nitrogen atom would contribute 2 electrons, but one electron has been lost to form the cation. Therefore, the two Nitrogen atoms together contribute three more electrons. Including the electrons contributed from the two Nitrogen atoms and the number of participating Carbon atoms ( $p$ ) in the molecule, the *number of  $\pi$  electrons* in the system is therefore:

$$\text{number of } \pi \text{ electrons} = p + 3 \quad (10.11)$$

### **Beer-Lambert Law**

The Beer-Lambert Law relates the absorption of light to its concentration of an absorbing species, providing the basis of using spectroscopy in quantitative analysis.

$$A = \varepsilon Cl \quad (10.12)$$

Where:  $I$  is the distance the light travels through the solution (cm) and  $C$  is the concentration of the absorbing species ( $\text{mol L}^{-1}$ ). The molar absorption coefficient or commonly known molar extinction coefficient ( $\varepsilon$ ) ( $\text{mol}^{-1} \text{L cm}^{-1}$ ) measures the effectiveness and strength of an absorbing species absorbing light. A high value of  $\varepsilon$  indicates a large amount of light is absorbed by the chemical species at a given concentration and wavelength.

For conjugated systems, such as the dye molecules, the wavelength ( $\lambda$ ) at which the absorbance is maximum becomes longer and its extinction coefficient ( $\varepsilon$ ) becomes larger as the conjugated system becomes longer, showing stronger absorption of light.

Refer to Chemistry<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:

*The Beer-Lambert Law: Section 10.3, pages 460 – 462.*

*Ultraviolet-visible spectrophotometry: Section 11.4, pages 543 – 545.*

## Determination of the absorption spectra of conjugated dyes

### Part A: Preparation of dye solutions of different concentrations

1. You will be provided with stock solutions of three conjugated dyes and their concentrations are given below in the table:

Conjugated Dyes		Stock Solution Concentration
1,1'-Diethyl-2,2'-cyanine iodide	DCI	$1.5 \times 10^{-5} \text{ mol L}^{-1}$
Pinacyanol chloride (1,1'-Diethyl-2,2'-carbocyanine chloride)	PCC	$1.2 \times 10^{-5} \text{ mol L}^{-1}$
1,1'-Diethyl-2,2'-dicarbocyanine iodide	DDCI	$1.2 \times 10^{-5} \text{ mol L}^{-1}$

2. For each of the given dyes, prepare solutions of three different concentrations by diluting their respective stock solutions with the solvent methanol:
  - a) Rinse each of the three 10 mL volumetric flasks with 2 x 1 mL methanol using a dropping pipette.
  - b) Add 8 mL of 1,1'-Diethyl-2,2'-cyanine iodide (DCI) stock solution, via zippette, into a DRY 50 mL beaker.
    - **Record the colour of dye.**
  - c) Pipette 4 mL of the stock solution using an auto-pipette into volumetric flask 1.
  - d) Pipette 2 mL of the stock solution using an auto-pipette into volumetric flask 2.
  - e) Pipette 1 mL of the stock solution using an auto-pipette into volumetric flask 3.
  - f) Add methanol to make up to the mark of each volumetric flask using a dropping pipette.
  - g) Stopper the volumetric flasks and mix thoroughly.
  - h) Calculate and enter the concentrations of these solutions into your laboratory notebook or computer.
  - i) On the completion of absorption spectra measurements for each set of dye solutions (refer to Part B),
    - empty solutions into appropriate waste container
    - rinse each volumetric flask with 2 x 1 mL methanol using a dropping pipette
  - j) Repeat Step (b) to Step (i) for Pinacyanol chloride (PCC).
  - k) Repeat Step (b) to Step (i) for 1,1'-Diethyl-2,2'-dicarbocyanine iodide (DDCI).

### Part B: Measurement of absorption spectrum using spectrophotometer

Use of spectrophotometer cuvette:

- The cuvettes should only be handled by top edge of the two ribbed sides; fingers must not touch the optically clear sides.
- Rinse cuvette three times with methanol and drain on a tissue.
- ALWAYS rinse cuvette twice with the solution to be measured.
- Fill the cuvette (only to two-third full) with the solution to be measured, away from the spectrophotometer and over a waste container.
- Carefully wipe the clear sides with a tissue to remove dust or liquid droplets.
- Ensure there are no air bubbles on the inside walls of the cuvette by gently tapping the cuvette on a hard surface.

- Check all sides are properly clean before inserting the cuvette in the spectrophotometer cuvette holder. Always position the cuvette so the light passes through the clear sides.
- When the experiment is concluded, rinse with methanol and drain on a tissue.

**NOTE:** Notify your demonstrator if any liquid is spilt inside the spectrophotometer.

1. A set of cuvettes, each with an optical path length ( $l$ ) of 1.00 cm, is provided for measurement.
2. The instructions to each model of UV spectrophotometer used are provided in the laboratory.

**Plug in your USB stick to spectrophotometer.**

3. Always use the “reference” solution to zero the spectrophotometer over the required wavelength range of 400 – 800 nm before taking any absorption measurement for the dye solution.

**Your demonstrator will provide you with a capped cuvette filled with methanol. This is your reference for the measurement of the absorption spectra. Make sure you return this cuvette to demonstrator upon completion.**

4. You will use the UV spectrophotometer to record the absorption spectra over the same wavelength ( $\lambda$ ) range of 400 – 800 nm as the reference for each prepared dye solutions.
5. Measure FIRST the UV-Vis absorption spectrum of the solution that has the lowest concentration of dye. If the absorbance maximum of any solutions is much greater than unity, your solutions should be diluted and the spectrum re-taken.
6. You will require saving your absorption spectra in CSV format to a USB stick.

### **Question 1**

**Why should the lowest concentration solution for each of three dyes be measured first?**

At the end of experiment:

- Unplug your USB stick from spectrophotometer.
- Empty all waste into appropriate waste container.
- Rinse all glassware and cuvettes with 2 x 1 mL methanol using a dropping pipette.

**NOTE: DO NOT USE WATER TO CLEAN GLASSWARE AND CUVETTES.**

### **Part C: Data analysis**

*EXCEL Plotting program*

- Install EXCEL plotting program onto computer (go to <https://portal.office.com> and follow prompts for free installation of Microsoft Office 365 and its EXCEL plotting program).
- Upload the CSV data files of absorption spectra of dye solutions onto computer.
- To find out gradient of graph:
  - go to ‘add trendline’ and a best fit line is now showed on graph through data points
  - go to ‘display equation on graph and display R squared value on chart’  
(the number given in front of ‘x’ of the displayed equation is the gradient)

- To overlay graphs:
  - x-axis values must be identical for each data set
  - copy the new set of data points (for y-axis) onto same spread sheet into the 3<sup>rd</sup> column and 4<sup>th</sup> column, if required
- Save and copy graph to your report for submission

1. Prepare a single plot overlaying the intensity normalised absorption spectra of the three dyes:
  - a) Only use the data for the dye solution that has highest concentration.
  - b) Plot the Absorbance (y axis) versus wavelength in nm (x axis).
  - c) Determine the wavelength ( $\lambda_{\max}$ ) at which the absorbance is maximum for each dye solution from the graph.

### Question 2

**What is the expected colour for each dye at the wavelength with maximum absorbance ( $\lambda_{\max}$ ) that you have determined for each dye? Do they agree well with the observed colour for each given dye?**

2. Determination of extinction coefficients ( $\epsilon$ ) for each dye:
  - a) Read off the absorbance at  $\lambda_{\max}$  for each prepared dye solution directly from their respective CSV data files.
  - b) On a single graph, plot the Absorbance (y axis) versus solution concentration (x axis) for each dye used.
  - c) Determine the extinction coefficients ( $\epsilon$ ) including unit for each dye compound using the Beer-Lambert Law, where the path length of light through the solution,  $\ell$ , is 1.00 cm.  
*(Hints: Consider the gradient of plot and using equation 10.12).*
3. Construct Table 1 summarising the results obtained above (i.e.  $\lambda_{\max}$  and  $\epsilon$  for each dye) from Step 1 and Step 2.

### Question 3

**What is the significance of extinction coefficient?**

### Question 4

**Comment on the correlation between the length of the conjugated dye, the wavelength of maximum absorbance ( $\lambda_{\max}$ ) and extinction coefficients ( $\epsilon$ ) of the three dye compounds you have given.**

4. Construct Table 2 summarising the following results from calculations:
  - a) Determine the number of participating Carbon atoms,  $p$ , for each of the three dyes (refer to Figure 10.1).
  - b) Calculate the length of the “box”,  $L$ , for each of the three studied dyes (equation 10.10).
  - c) Calculate the number of electrons that must be considered for each of the three conjugated dyes using equation 10.11.
  - d) Calculate the highest occupied level (i.e. the quantum number  $n$ ) for the ground state of each of the dyes using equation 10.7.

- e) Calculate the predicted wavelength ( $\lambda_{predicted}$ ) for each of the three dyes (using equation 10.9) where the “particle-in-a-box” model predicts their absorption maxima.

**Question 5**

*How could the estimate for the length of the “box” be improved?*

**Question 6**

*How well is the agreement between the experimental values for the wavelength with maximum absorbance ( $\lambda_{max}$ ) determined for each dye molecule with those above predicted values ( $\lambda_{predicted}$ )? Use the result from this comparison to discuss the suitability of the “particle-in-the-box” model in predicting the experimentally observed qualitative and quantitative trends with references to the predicted trends.*

5. On a single graph, plot the Absorbance (y axis) versus wavelength in nm (x axis) for each solution concentration of the dye 1,1'-Diethyl-2,2'-dicarbocyanine iodide (DDCI).

**Question 7**

*Do the absorption spectra change in shape as a function of dye concentration?*

*What could be the consequence of using too high a concentration for transmission-based measurements?*

**Part D: Questions**

**Question 8**

*When you look around yourself, many pure molecular substances are colourless.*

*Why is this so?*

**Question 9**

*Why can the colour of nanocrystals (e.g. of semiconductor nanomaterials such as CdSe) be described by a similar “Particle in the Box” approach?*

## WRITING UP OF REPORT

Your report for this experiment should be prepared **electronically**. Submit your report electronically using the TurnItIn facility on the Learning Management System (LMS). You can use the computer facilities provided in “The Labyrinth” or your own computer to prepare your report. The report should follow the style used by leading chemical journals and particularly, it needs to be CONCISE and written in the past tense and passive voice:

1. The “Aim” should be 1 to 2 sentences.
2. The “Experimental Method” should be sufficient to note “Refer to First Year Chemistry Laboratory Manual, Experiment X, pages 15 to 18.”
3. The “Result and Calculation” should include all obtained experimental results and calculations:

*For this report, graphs should be prepared in Portrait Format using a suitable computer plotting program, with both axes being appropriately scaled and clearly labelled including the units. Graph should be described with a title. Multiple spectra can be combined into a single plot where it is appropriate and clear, but must also include a legend.*

- Table 1 summarising the results for of name and concentration of dye solutions,  $\lambda_{\max}$  and absorbance at  $\lambda_{\max}$ .
  - Table 2 summarising the calculated values of p, L, number of electrons, n,  $\lambda_{\max}$  and  $\lambda_{\text{expected}}$ .
  - The single plot (Graph 1) overlaying the intensity normalised absorption spectra of absorbance versus wavelength (at the highest concentration) of each dye used.
  - The single figure (Graph 2) plotting the absorbance versus concentration of each dye used. Include the calculations of extinction coefficients.
  - The single figure (Graph 3) plotting the absorbance versus wavelength for the dye 1,1'-Diethyl-2,2'-dicarbocyanine iodide (DDCI) at three different concentrations.
4. The “Discussion” should include answers to Questions 1 to Question 9. The answers should be short, precise and of no more than 3 to 4 sentences each.
5. The “Conclusion” should be 1 to 2 sentences.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input type="checkbox"/> Corrosive  | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input checked="" type="checkbox"/> Highly flammable   | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input checked="" type="checkbox"/> Minimal risk   |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- |   |
|---|
| <input checked="" type="checkbox"/> 3 Minimal risk                          |
| <input type="checkbox"/> 2a Low risk (Fume hood recommended)                |
| <input type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential)     |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)              |
| <input type="checkbox"/> 1b Significant risk (Special location or facility) |

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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**ELECTROCHEMISTRY:****11****EMF MEASUREMENTS****AIMS OF THE EXPERIMENT**

- To set up an electrochemical cell (a device for producing electricity by means of a chemical reaction) and measure its potential. The  $\text{Zn}^{2+}|\text{Zn}$  and  $\text{Cu}^{2+}|\text{Cu}$  redox couples will be used.
- To use a reference electrode (a half-cell electrode which maintains a constant potential) to compare the oxidizing power of  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$ .
- To investigate the effect of changes in concentration on the cell potential.
- To compare the experimental results with the values calculated for half-cells using the Nernst equation.

**READING**

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:  
Electrochemical cells: Section 16.3, pages 735 – 746;  
Concentration dependence of  $E_{\text{cell}}$ : pages 749 – 751.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:  
Oxidation and reduction: pages 496 – 497;  
Galvanic cells and reduction potentials: pages 504 – 515;  
The Nernst equation: pages 522 – 523.

**PRE-LAB QUESTIONS**

There is a compulsory ChemCAL Prelabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

**INTRODUCTION**

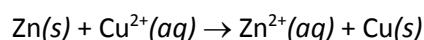
For this experiment:

- Students work in pairs.
- Students require graph paper and a basic scientific calculator.
- The report is written individually in own words with own graphs and calculations.

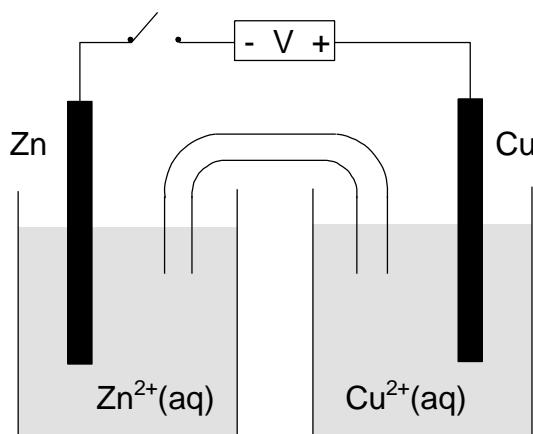
**1. Electrochemical Cells**

When zinc metal is dipped into a solution containing copper ions copper metal is plated out on the zinc. The spontaneous reaction occurring is the oxidation of the zinc metal producing electrons, which are then used in the reduction of copper ions to precipitate copper metal.

The overall reaction is:



This reaction can be carried out by setting up two half-cells as shown in Fig. 11.1.



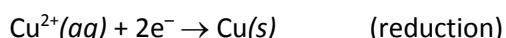
**Figure 11.1** An electrochemical cell

The right-hand beaker contains  $Cu^{2+}(aq)$  with a Cu metal electrode and the left-hand beaker  $Zn^{2+}(aq)$  with a Zn metal electrode. The electrodes are connected through a switch and voltmeter by wires which allow electrons to flow and the solutions are electrically connected by a salt-bridge, which allows charged ions to carry current between the solutions. When the switch is closed, small current flows and the voltmeter will indicate a potential of about +1.0 V with the polarity shown, meaning the oxidation is occurring in the left-hand beaker. Electrons produced are flowing from the Zn electrode via the external circuit to the Cu electrode where they are used in the reduction reaction, which produces Cu metal. Evidence for this is gained if the switch is left closed for several hours when the blue colour of  $Cu^{2+}$  solution diminishes and the Cu electrode increases in mass. In the other half-cell, the mass of the Zn electrode decreases and  $[Zn^{2+}]$  increases. Ions moving between the beakers via the salt bridge maintain electrical neutrality.

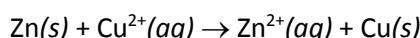
The cell diagram is represented as:



The half-cell reactions are:



and the overall reaction is:



which is the same as when the zinc metal is dipped in a copper solution but now the reaction is ‘producing electricity’

For an electrochemical cell,

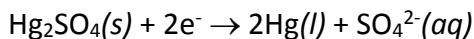
$$E_{\text{cell}} = E_{+\text{ve}} - E_{-\text{ve}} \quad (11.1)$$

Applying this to the cell in Figure 11.1:

$$E_{\text{cell}} = E_{\text{copper half cell}} - E_{\text{zinc half cell}}$$

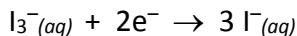
## 2. Reference Electrode

A reference half-cell is often used against which the potential of other half-cells can be measured. In this experiment the reference half-cell used is the mercury–mercury(I) sulfate electrode which is referred to as MMSE. It consists of liquid mercury and mercury(I) sulfate in solid form, since it is sparingly soluble in water, in contact with a saturated solution of potassium sulfate. The half-cell is represented as  $\text{K}_2\text{SO}_4(\text{sat'd})|\text{Hg}_2\text{SO}_4(s)|\text{Hg}(l)$  which at 298 K has a potential of 0.642 V. Its reduction reaction is:



In part A of the experiment the MMSE electrode is combined with a  $\text{Cu}^{2+}|\text{Cu}$  half-cell and a  $\text{Zn}^{2+}|\text{Zn}$  half-cell.

In part B of the experiment the MMSE is used to determine the half-cell potential for the reaction:



As both the oxidized and reduced species in this half-cell are ions in solution a platinum electrode is used as the potential probe.

## 3. Nernst Equation

For the reduction reaction:



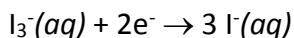
the Nernst equation (11.2) shows the relationship between the half-cell potential and the concentrations of the oxidized and reduced species.

$$E = E^\circ - \frac{RT}{nF} \ln \left( \frac{[\text{Red}]^b}{[\text{Ox}]^a} \right) \quad (11.2)$$

where  $E^\circ$  is the standard reduction potential for the specified system,  $R$  is the gas constant,  $T$  the temperature (in Kelvin),  $n$  the number of electrons transferred in the reaction and  $F$  the Faraday constant. A natural logarithm,  $\log_e$ , is written as  $\ln$  and  $RT/F$  has the value 0.0257 V at 298 K.

If a plot of  $E$  against  $\ln \left( \frac{[\text{Red}]^b}{[\text{Ox}]^a} \right)$  is a straight line, it will have gradient  $-\left( \frac{RT}{nF} \right)$  and intercept  $E^\circ$ .

For the reaction



The Nernst equation applied to this redox couple is:

$$E = E^\circ(\text{I}_3^-/\text{I}^-) - \left( \frac{RT}{2F} \right) \ln \left( \frac{[\text{I}^-]^3}{[\text{I}_3^-]} \right) \quad (11.3)$$

## SAFETY



### Safety Warning

Iodine is corrosive. Avoid contact with the skin. Wear gloves when necessary.

Avoid breathing vapour.

### Disposal of chemical wastes:

Do not pour any of the metal ion solutions or any of the triiodide-iodide ion solutions down the sinks. Discard only in the appropriate residue bottles.

## Risk Assessment

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

The cell potential is measured using the TPS pH Cube voltmeter with the settings:

- RED connector is the positive terminal.
- BLACK connector is the negative terminal.
- The cell potential is measured in millivolt ( $1 \text{ mV} = 10^{-3} \text{ V}$ ).

Between solutions:

- Rinse and dry the electrodes (use tissue).
- Rinse and dry the surfaces of the salt bridges.
- Wash beakers with distilled water and rinse with the next solution before taking your test sample.

### PART A. Half-cell potential of Metal|Metal ion electrodes

1. You are provided with a solution of 0.01 M copper sulfate and pieces of zinc and iron nail.
  - a) Expose a clean surface on the iron nail by light abrasion with emery (sand) paper, rinse with water and dry the metal surface before dipping it into 5 mL of the copper sulfate solution in a test tube. Allow to stand for 1 minute.
    - Record any changes you observe and write ionic equation for any overall reaction.
  - b) Repeat the procedure with the zinc metal.
    - Explain any changes you observe, writing ionic equation for any overall reaction.
2. Set up an electrochemical cell as follows:
  - a) To a small beaker add 20 mL of 0.01 M  $\text{ZnSO}_4$  and the zinc electrode.
  - b) To another small beaker add 20 mL of 0.01 M  $\text{CuSO}_4$  and the copper electrode.
  - c) Connect the two half-cells using a potassium nitrate salt-bridge.
  - d) Connect the copper electrode to the positive terminal of the voltmeter, the zinc to the negative terminal.
    - Measure the cell potential.

**Discussion:**

- i. Sketch the cell. If the cell is to discharge by connecting its electrodes with a piece of wire, clearly indicate the direction of electron flow.
  - ii. Write ionic equations for the half-cell reactions and the overall reaction.
  - iii. In which half-cell is oxidation occurring?
  - iv. Indicate the direction of movement of the ions in the salt bridge.
  - v. In your own words, describe the essential features of an electrochemical cell.
3. Measure the potential of a cell consisting of the MMSE and the  $\text{Cu}^{2+}|\text{Cu}$  half-cell as follows:
- a) Remove the salt-bridge and the two wires from the cell constructed in **Step 2**.
  - b) Put the MMSE electrode into the beaker containing the  $\text{Cu}^{2+}|\text{Cu}$  couple.
  - c) Connect the MMSE electrode to the positive terminal and the Cu electrode to the negative terminal of the voltmeter.
    - Record the cell potential.
4. Measure the potential of a cell consisting of the MMSE and the  $\text{Zn}^{2+}|\text{Zn}$  half-cell as follows:
- a) Remove the two wires from the Cu and the MMSE electrodes in the cell constructed in above **Step 3**.
  - b) Rinse and carefully dry the MMSE electrode. Put the MMSE electrode into the beaker containing the  $\text{Zn}^{2+}|\text{Zn}$  couple.
  - c) Connect the MMSE electrode to the positive terminal and the Zn electrode to the negative terminal of the voltmeter.
    - Record the cell potential.

**Discussion:**

- i. Write ionic equations for the half-cell reactions considered above (**3** and **4**) and the overall cell reaction occurring in each case.
- ii. In which half-cell is oxidation occurring?
- iii. If the MMSE has a potential of +0.642 V, what is the potential of the  $\text{Cu}^{2+}|\text{Cu}$  and  $\text{Zn}^{2+}|\text{Zn}$  half-cells you have prepared? [Use equation 11.1]. Which is the stronger reducing agent, Cu or Zn?
- iv. Will a reaction occur between  $\text{Zn}^{2+}(\text{aq})$  and  $\text{Cu}(\text{s})$ ? How will you know if it does? Try it.
- v. All half-cell potentials are expressed relative to the standard hydrogen electrode (SHE) which is accepted as having a potential of exactly zero (0.00 V) at 298 K.  
List the MMSE,  $\text{Cu}^{2+}|\text{Cu}$ ,  $\text{Zn}^{2+}|\text{Zn}$  and the SHE in increasing order of oxidizing power.

## PART B. Triiodide-iodide half-cell potential measurements

There are two important factors need to be considered when making electrical cell potential measurements, they are

- The liquid junction potential: This potential is hard to estimate accurately but could be as high as 0.05 V at the junction between the triiodide-iodide solution and the electrolyte in MMSE.
- The interaction between triiodide-iodide ions in solution.

1. Set up the cell: Pt | I<sup>-</sup>(aq), I<sub>3</sub><sup>-</sup>(aq) || MMSE

Determine the cell potential using the various solutions of triiodide/iodide ion supplied. Use about 20 mL of the solutions for each measurement.

Connect the MMSE electrode to the positive terminal of the voltmeter

Connect the platinum electrode to the negative terminal of the voltmeter.

Two sets of solutions are provided;

Set 1 has constant iodide (I<sup>-</sup>) concentration

Set 2 has constant triiodide (I<sub>3</sub><sup>-</sup>) concentration.

- Record the measurements ( $E_{\text{cell}}$  (V)) in tabular form **into laboratory notebook** as shown below.

Solution number	[I <sup>-</sup> ]/M	[I <sub>3</sub> <sup>-</sup> ]/M	$E_{\text{cell}}$ (V) (measured)	Potential for I <sub>3</sub> <sup>-</sup> /I <sup>-</sup> half cell		ln[I <sup>-</sup> ]	ln[I <sub>3</sub> <sup>-</sup> ]
				$E_{\text{exp}}$ (/ V)	$E_{\text{cal}}$ (/ V)		
Set 1	1	0.2000	0.0010				
	2	0.2000	0.0050				
	3	0.2000	0.0100				
	4	0.2000	0.0500				
Set 2	5	0.0500	0.0050				
	6	0.1000	0.0050				
	7	0.1500	0.0050				
	8	0.2000	0.0050				

2. Use the relationship;

$$E_{\text{exp}}(\text{I}_3^-/\text{I}^-) = 0.642 - E_{\text{cell measured}}$$

to calculate the half-cell potentials for the triiodide-iodide half-cell and enter the data in the table ( $E_{\text{exp}}$  (V)).

3. The Nernst equation (11.3) for this triiodide-iodide redox couple can be expressed to the form:

$$E = E^\circ(I_3^-/I^-) + \left(\frac{RT}{nF}\right) \ln[I_3^-] - 3\left(\frac{RT}{nF}\right) \ln[I^-] \quad (11.4)$$

$E^\circ(I_3^-/I^-)$  is constant, 0.534 V, relative to the SHE at 298 K,  $n$  is the number of electrons transferred in the reaction and  $(RT/F)$  has the value 0.0257 V at 298 K.

- i. Use the Nernst equation to calculate the half-cell potentials ( $E_{\text{calc}}$  (V)) for the solution concentrations investigated. You should have already completed part of these calculations in ChemCal Prelab module. Enter the calculated values,  $E_{\text{calc}}$  (V), in the table.
- ii. How well do the predicted  $E_{\text{calc}}$  using the Nernst equation compare to the experimental results ( $E_{\text{exp}}$ )?

**NOTE:** Your graph should be in landscape format, occupy > 80% of ordinate and coordinate range, label axes with correct scales and unit, include a title and show data points used for gradient calculation.

4. The experimental results should indicate that changing the concentration of the oxidant and reductant changes the half-cell potential. To establish a relationship, plot the results as follows:

- i. Using Set 1 data:  $[I^-]$  constant and Equation 11.4 becomes

$$E = \text{constant} + \left(\frac{RT}{nF}\right) \ln[I_3^-]$$

- Plot a graph showing  $E_{\text{exp}}$  (V) on the vertical axis and  $\ln[I_3^-]$  on the horizontal axis.
- Draw the best fit line and determine the ‘experimental gradient’ including unit.
- Calculate the ‘predicted gradient’  $\left(\frac{RT}{nF}\right)$  including unit.

- ii. Using Set 2 data:  $[I_3^-]$  constant and Equation 11.4 becomes

$$E = \text{constant} - 3\left(\frac{RT}{nF}\right) \ln[I^-]$$

- Plot a graph showing  $E_{\text{exp}}$  (V) on the vertical axis and  $\ln[I^-]$  on the horizontal axis.
- Draw the best fit line and determine the ‘experimental gradient’ including unit.
- Calculate the ‘predicted gradient’  $[-3\left(\frac{RT}{nF}\right)]$  including unit.

- iii. What is the value of the ratio of experimental gradients,  $\frac{\text{Set 2}}{\text{Set 1}}$ , calculated using the above results determined in Step (i) and Step (ii)?

- iv. What is the value of the ratio of predicted gradients,  $\frac{\text{Set 2}}{\text{Set 1}}$ , calculated using the Nernst equation for the triiodide-iodide redox couple as given in equation 11.4?

#### **Discussion:**

How well does the Nernst equation predict the experimental results?

Use the results obtained in Step 4(iii) and Step 4(iv) for comparison.

Write a short statement summarizing this comparison.

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input type="checkbox"/> Pungent   | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

# SYNTHESIS OF HEXAAMMINECOBALT(III)CHLORIDE

**12**

## AIMS OF THE EXPERIMENT

- To synthesize a typical coordination complex, hexaamminecobalt(III) chloride,  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$ .
- To illustrate the use of a redox reaction in chemical synthesis.
- To gain further practice in the skills necessary for chemical synthesis, especially recrystallization.

## READING

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:  
Coordination Chemistry: Sections 28.3 – 28.8, pages 1265 – 1296.
- Techniques and instrumentation section of this laboratory manual: from pages 127.

## PRE-LAB QUESTIONS

There is a compulsory ChemCAL Prelabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

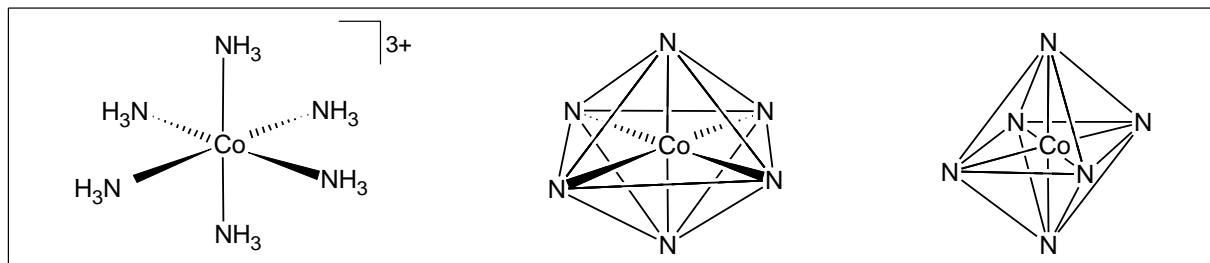
On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the end of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

## INTRODUCTION

For this experiment students work individually and require a basic scientific calculator.

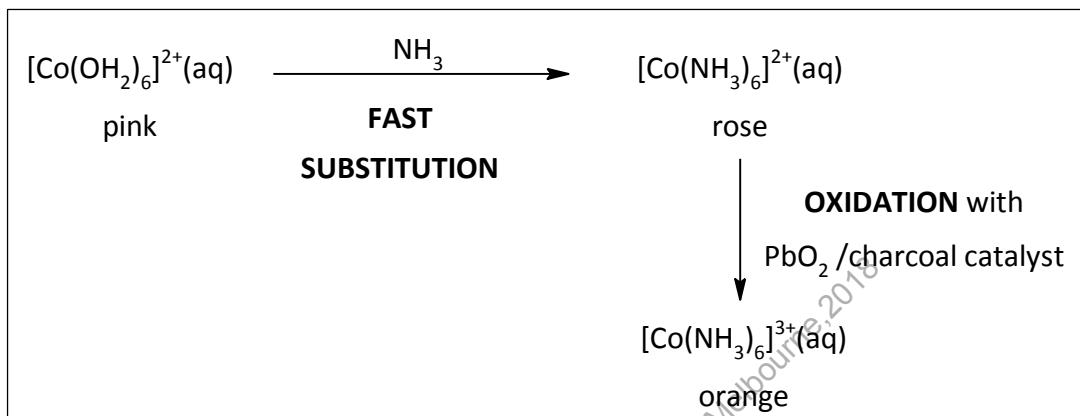
In this experiment you will synthesize a typical coordination complex, hexaamminecobalt(III) chloride,  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$ . Hexaamminecobalt(III) chloride consists of a central metal ion  $[\text{Co}(\text{III})]$  around which are six ammonia molecules arranged in octahedral coordination. Each ammonia molecule forms a bond to the cobalt ion via the lone pair of electrons on the nitrogen atom; the resulting entity is called a coordination complex. The charge on the complex cation  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$  is counterbalanced by three negative chloride ions, which are not covalently bonded to the cobalt ion. The complex cation  $[\text{Co}(\text{NH}_3)_6]^{3+}$  and the  $\text{Cl}^-$  anions form an ionic lattice. In a similar way solid NaCl consists of a lattice of  $\text{Na}^+$  and  $\text{Cl}^-$  ions. Different representations of the structure of the  $[\text{Co}(\text{NH}_3)_6]^{3+}$  cation is shown in Fig. 12.1.



**Figure 12.1** Different representations of the structure of the hexaamminecobalt(III) cation.

## **Chemical Aspects of the Synthesis**

For very many Co(III) complexes the starting material used in synthesis is a Co(II) salt. This is because the usual oxidation state of cobalt in its simple salts such as the sulfate is +2. The Co(III) oxidation state only becomes stable when cobalt is coordinated to certain ligands such as  $\text{NH}_3$  or  $\text{NO}_2^-$ . In addition, Co(II) complexes undergo substitution reactions (the replacement of one ligand by another) very rapidly whereas reactions of Co(III) complexes are very slow. Therefore, the preparation of a Co(III) complex very often takes place by a fast reaction between Co(II) and the ligand to give the Co(II) complex, which is then oxidised to the corresponding Co(III) complex. In the synthesis of  $[\text{Co}(\text{NH}_3)_6]^{3+}$  the key sequence of steps is as follows.



## SAFETY



**Caution:** Steam baths have hot surfaces and steam can cause burns.

## **Safety Warning;**

Concentrated aqueous ammonia solutions are corrosive and give off irritating vapours. Handle in the fumehood and wear safety glasses and gloves. Concentrated aqueous solutions of hydrochloric acid are corrosive and give off pungent vapours. Handle in the fumehood and wear safety glasses.

Ethanol is a volatile liquid, which is flammable. Avoid contact with flames or electrical equipment.

## Risk Assessment:

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Synthesis and Purification

#### 1. Preparation

- i. Into a 50 mL conical flask dispense, from the appropriate zippette, 1.5 mL of the cobalt(II) sulfate,  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , solution (750 mg, 2.66 mmol); 3.0 mL of the ammonium chloride ( $\text{NH}_4\text{Cl}$ ) solution (740 mg, 13.8 mmol) and 2 mL of the concentrated ammonia solution. Note the colour changes. To minimize the loss of ammonia, cover the flask with a watch glass. Using the scoops provided add lead(IV) oxide,  $\text{PbO}_2$ , (1 level scoop) and activated charcoal (1 level scoop).

**NOTE:** The following instructions must be followed carefully; see Fig. 12.2 for an outline of the procedures used in the preparation of the crude product.

- ii. Swirl the flask gently and let stand at room temperature for about 2 minutes to allow initial reaction without any loss of ammonia. Place the flask on a port of a steam bath for 15 minutes. Swirl the reaction mixture every 2 to 3 minutes. Throughout this process leave the watch glass in place. The steam bath should be operating on setting 10, which will maintain the temperature of the mixture at about 90 °C.
- iii. Remove the flask from the steam bath and chill it in ice, which will cause a solid product to separate. Break up the solid with a micro-spatula and then collect it by filtration through a Hirsch funnel. If any solid remains in the flask the filtrate may be recycled to rinse out the flask. (The filtrate can be used for this purpose but water cannot. Suggest the reason for this.)
- iv. The solid product is a mixture of the hexaammine cobalt(III) chloride, charcoal, lead(II) sulfate and excess lead(IV) oxide.



Hirsch vacuum  
filtration

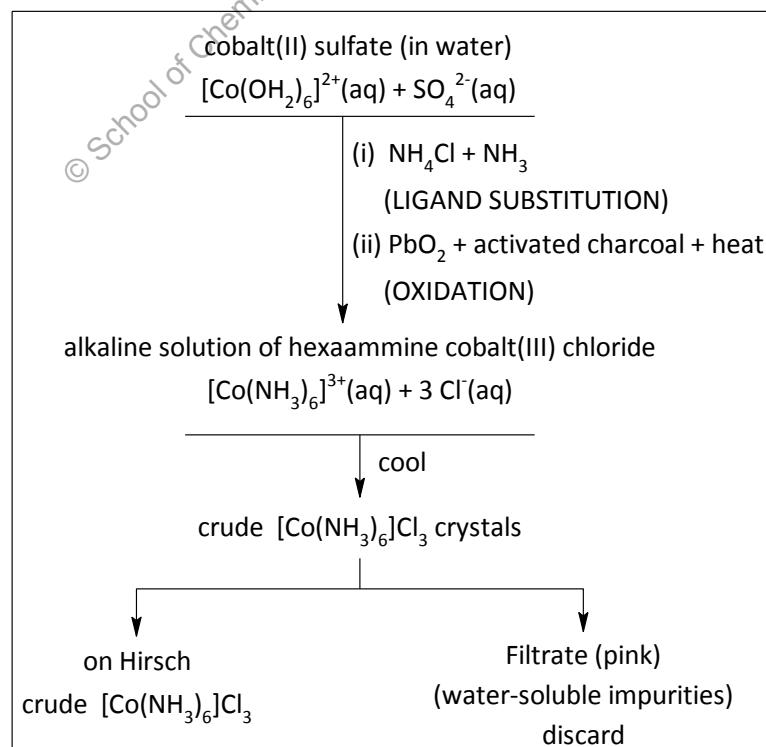


Figure 12.2 Preparation of crude  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$ .

## 2. Purification by Recrystallization

- i. The flow chart in Figure 12.3 outlines the procedures to be used in the purification of the crude  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$ .
- ii. Carefully transfer the crude product to a 50 mL conical flask and add 12 mL of water and then 0.4 mL of concentrated hydrochloric acid (via zippette). Check that the solution is faintly acid to litmus paper; if it is not, add 1 to 2 drops (dropping bottle) of concentrated hydrochloric acid.
- iii. Heat the mixture on a steam bath until all the orange crystals have dissolved (this can be checked by examining the base of the beaker with a torch.) There will still be some undissolved solid at this stage.
- iv. Filter the hot solution through a pre-heated Hirsch funnel (the funnel may be heated by inverting it over a port of the steam bath) and quickly transfer the filtrate to a clean 50 mL conical flask.
- v. Slowly add 2 mL of concentrated hydrochloric acid from a zippette whilst continually swirling the flask.
- vi. Heat the mixture on a steam bath until all the fine crystals has dissolved, this should take no more than 5 minutes (if necessary add 1 mL of water using a dropping pipette). Finally, allow the hot solution to cool undisturbed at room temperature for about 15 – 20 minutes.
- vii. Collect the crystals on a Hirsch funnel and dry at the pump.

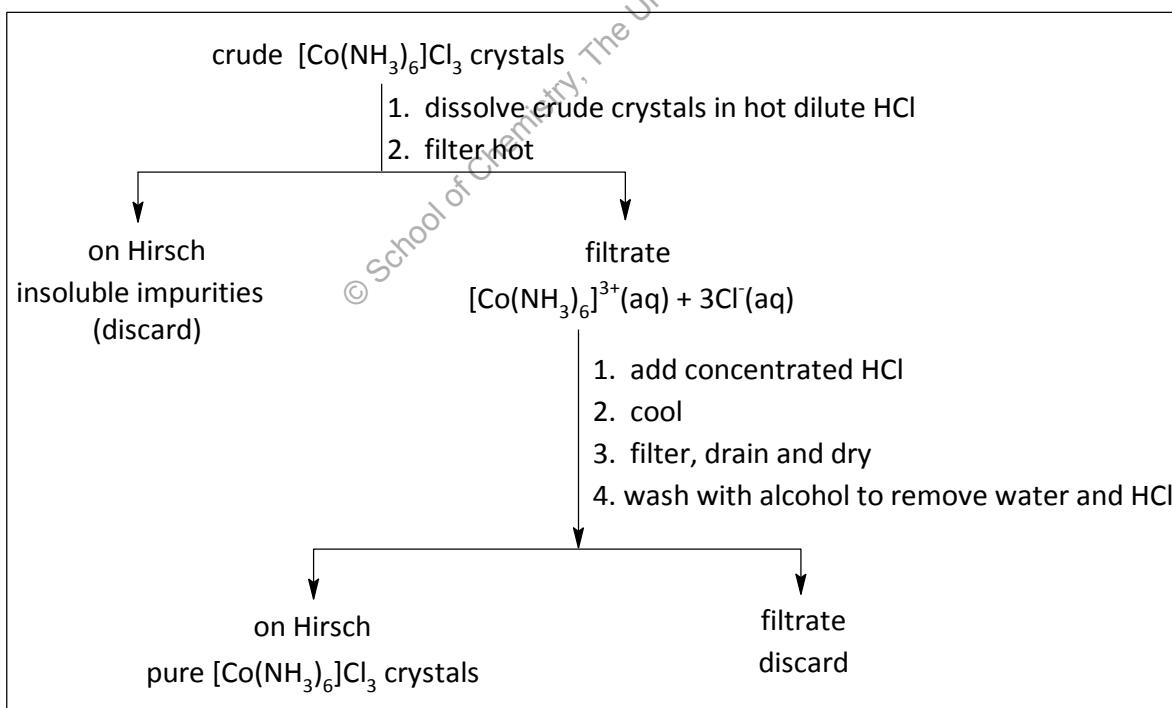


Figure 12.3 Purification of crude  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$  by recrystallization.

## 3. Product Yield

- i. Transfer the crystals to a pre-weighed snap lock plastic bag and reweigh the bag to obtain the yield. Calculate the theoretical yield of hexaamminecobalt(III) chloride based on the mass of  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$  used.
- ii. Express your actual yield as a percentage of the theoretical yield.



Analytical balance

## Part B: Test Tube Reactions of Co(III) and Co(II) Complexes

### 1. Reactions of Co(III) complex – $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$

When  $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$  is dissolved in water it dissociates into ions; what are these ions? Thus, in describing the reactions given below you should write ionic equations where appropriate.

Record your results in a tabular fashion into laboratory notebook under the following headings: REACTION, OBSERVATION and EXPLANATION AND EQUATION. i.e.

REACTION	OBSERVATIONS	EXPLANATION AND EQUATION

- i. Take a small amount of your solid product from Part A (the equivalent in volume to about one rice grain) and dissolve it in 1 mL of water in a test tube. Add 5 – 6 drops of 0.01 M  $\text{AgNO}_3$  solution. *Note the result and write the appropriate ionic equation.*
- ii. Take a small amount of your solid product from Part A (the equivalent of about two rice grains in volume) and dissolve it in 2 mL of water in a test tube. Warm the solution on the steam bath for 1 minute and test the vapours with moist red litmus paper. *Note the result. Does any reaction appear to have occurred?*
- iii. Cool the solution from (ii), add 5 to 6 drops of 2 M NaOH solution and repeat the above test for at least 1 minute of warming the solution. *Note the result and write the appropriate equation.*

### 2. Reactions of Co(II) complex – $[\text{Co}(\text{OH}_2)_6]^{2+}(\text{aq})$ ion: stabilization energy

Take 1 mL of the “test” solution of  $\text{CoSO}_4$  and add 1 mL concentrated HCl until the solution changes colour permanently. Cool the solution in the ice bath until a colour change occurs, heat in the steam bath and the colour should change back. The cycle may be repeated by heating and cooling the solution.

*Describe how heating and cooling affects the equilibrium position of reaction?*

*Record and explain your observations. Include appropriate equations.*

### 3. Reactions of Co(II) complex – $[\text{Co}(\text{OH}_2)_6]^{2+}(\text{aq})$ ion: concentration effect

Take 1 mL of the “test” solution of  $\text{CoSO}_4$  and add concentrated HCl dropwise with shaking until the solution changes colour permanently. Now decant approximately one-quarter of the solution to a clean test tube (retain the remainder of the solution for use in Part 4 below) and carefully dilute with water until a colour change occurs. The cycle may be repeated by adding more concentrated HCl to a small portion of the solution.

*Describe how addition of water ( $\text{H}_2\text{O}$ ) and HCl affects the equilibrium position of reaction?*

*Record and explain your observations. Include appropriate equations.*

### 4. Reactions of Co(II) complex – $[\text{Co}(\text{OH}_2)_6]^{2+}(\text{aq})$ ion: solvation effect

Decant approximately one-third of the remaining  $\text{CoSO}_4$  /HCl solution prepared in Part 3 (above) into a clean test tube and carefully dilute with water until a colour change occurs (as in the preceding part). Now add 1 mL of acetone to the solution.

*Describe how addition of acetone affects the equilibrium position of reaction?*

*Record and explain your observations. Include appropriate equations.*

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input checked="" type="checkbox"/> Pungent  | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input type="checkbox"/> Minimal risk  |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |   |                                       |   |                                |
|---|---------------------------------------|---|--------------------------------|
| <input checked="" type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|---|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |   |
|-------------------------------------|-----------------------------------|--|---|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input checked="" type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |   |

### Category of Risk (tick one)

- |  |
|--|
| <input type="checkbox"/> 3 Minimal risk  |
| <input type="checkbox"/> 2a Low risk (Fume hood recommended)                       |
| <input checked="" type="checkbox"/> 2b Low risk (Fume hood/Schlenk line essential) |
| <input type="checkbox"/> 1a Significant risk (Chemical hazard)                     |
| <input type="checkbox"/> 1b Significant risk (Special location or facility)        |

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

**SPECTROSCOPY:****13****DETERMINATION OF THE pK<sub>a</sub> OF AN ACID-BASE INDICATOR****AIMS OF THE EXPERIMENT**

- To determine the molar absorption coefficient,  $\epsilon$ , for the acid form of bromocresol green at the wavelength of maximum absorption,
- To verify the Beer-Lambert law at two wavelengths, and
- To determine the pK<sub>a</sub> for bromocresol green

**Note:**

You will require a calculator (basic scientific), graph paper, a sharp pencil and a 30 cm ruler as well as your laboratory notebook.

**READING**

- *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:  
Indicators: Section 7.5, pages 327 – 329;  
Electronic spectroscopy: Section 10.6, pages 491 – 492;  
Ultraviolet-visible spectrophotometry: pages 543 – 545.
- *Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 3<sup>rd</sup> ed. 2016:  
Acid-base indicators: pages 479 – 481;  
The colours of transition metal complexes: pages 574 – 575;  
Beer-Lambert Law: page 578  
UV/visible spectroscopy: pages 916 – 917.
- Techniques and Instrumentation section of this laboratory manual:  
Volumetric flasks and pipettes: page 130.

**PRELAB QUESTIONS**

There is a compulsory ChemCAL PreLabs module, which must be completed before you carry out this practical exercise. The details of how to access the module are on page 10 of this manual.

On completion of the ChemCAL module you will be issued with a receipt number. This number should be recorded, along with the other necessary details, on one of the “tear off” record slips at the back of this manual.

The completed slip must be handed to your demonstrator at the start of the session as evidence that the ChemCAL module has been completed.

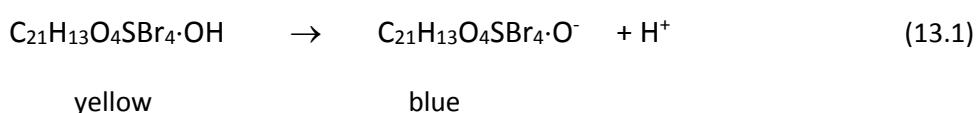
## INTRODUCTION

For this experiment:

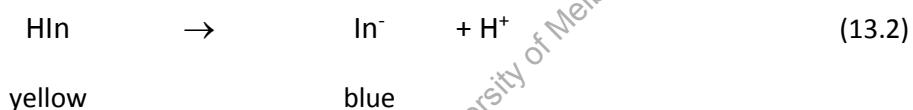
- Students work in pairs
  - The report is written individually in own words
  - All calculations and graphing of data collected during the experiment must be completed individually by students

The most common acid-base indicators are complex molecules that are themselves weak acids (represented by HIn). They exhibit one colour when the proton is attached to the molecule and a different colour when the proton is absent.

The indicator to be studied in this experiment is Bromocresol green, which has the formula  $C_{21}H_{13}O_4SBr_4 \cdot OH$ . Bromocresol green can dissociate as an acid:



For simplicity we shall represent the acid form (yellow in colour) as  $\text{HIn}$  and the basic form (blue) as  $\text{In}^-$ . The acid-base dissociation is then:



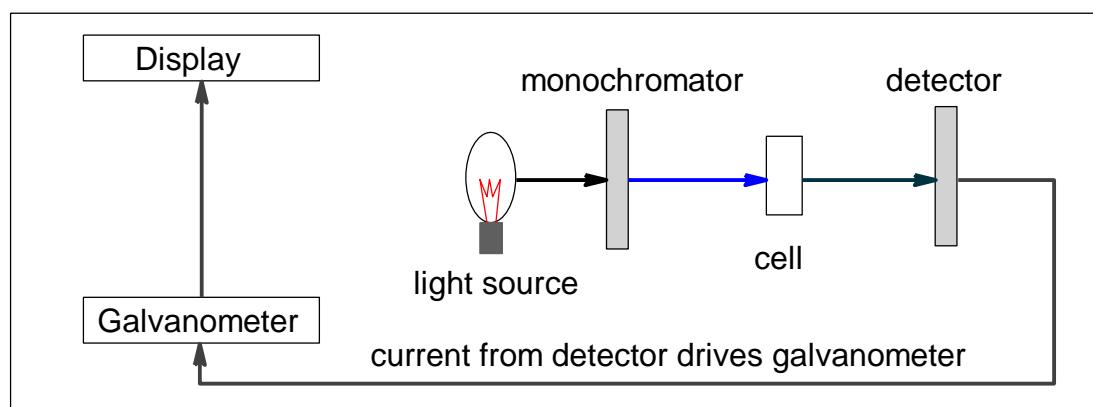
for which the equilibrium constant is:

$$K_a = \frac{[\text{H}^+][\text{In}^-]}{[\text{HIn}]} \quad (13.3)$$

This can be rearranged to give:

$$\frac{[K_a]}{[H^+]} = \frac{[\text{In}^-]}{[\text{HIn}]} \quad (13.4)$$

This ratio shows that the proportion of the two forms of the indicator in a solution is dependent on the concentration of H<sup>+</sup> present. If the solution is made suitably acidic, the indicator will be present mainly as the acid form (yellow). If the solution is made suitably basic, the indicator will be present mainly as the basic form (blue).



**Figure 13.1** Schematic diagram of the spectrophotometer

The concentration of either form of the indicator can be determined using a spectrophotometer to measure the absorbance of the indicator solution at a suitable wavelength of visible light.

In this experiment the ratio of the two forms of the indicator in a solution of known pH can be determined spectrophotometrically. This information can then be used to calculate the acidity constant,  $K_a$  of the indicator.

### Spectrophotometers

The NOVA and SHIMADZU spectrophotometers use the same basic principles for the measurement of absorbance. A simplified diagram of the operation of a spectrophotometer is shown in Fig. 13.1. White light passes through a spectrum wedge (monochromator) which selects a narrow band (35 nm); this monochromatic light beam passes through the sample (contained in the cell) and then impinges on a photoelectric cell detector coupled to a sensitive galvanometer.

A reference cell containing solvent is first placed in the light beam and the galvanometer (Absorbance scale) is adjusted to zero (0). This procedure corrects for any absorption by the solvent and cell. The sample is then placed in the path of the beam and the absorbance can be read.

### Spectrophotometer cells

A set of four cells, each with an optical path length of 1.00 cm, is provided. The cells should only be handled by the two matt sides; fingers must not touch the optically flat sides.

- Why? Would fingerprints on the reference cell increase or decrease the absorbance?

*After filling a cell with solution (only to two-thirds full), away from the spectrophotometer and over a beaker of water, carefully wipe the clear sides with a tissue to remove dust or liquid droplets. Check the sides are properly clean before inserting the cell in the cell holder, away from the spectrophotometer. Also, ensure that there are no air bubbles on the inside walls of the cell. Cells must never be placed in or removed from the cell-holder when the cell-holder is in the machine.*

*NOTE: You must always rinse the cells with the solution to be measured at least twice before filling the cells. Notify your demonstrator if any liquid is spilt inside the spectrophotometer.*

*To clean the cells, simply empty the contents into a beaker containing some water, and not the sink. Rinse the cells three times with distilled water (from a wash bottle) and drain on a cleaning tissue. When the experiment is concluded, wash and drain the cells and place them on the tissue in the box provided.*

### Beer-Lambert Law

The Beer-Lambert Law relates the absorption of light to its concentration of an absorbing species, providing the basis of using spectroscopy in quantitative analysis.

$$A = \epsilon cl \quad (13.5)$$

where  $\ell$  is the distance the light travels through the solution and  $c$  is the concentration of the absorbing species. The molar absorption coefficient ( $\epsilon$ ) measures the effectiveness and the strength of an absorbing species in absorbing light. A high value of  $\epsilon$  indicates a large amount of light is absorbed by the chemical species at a given concentration and wavelength.

## SAFETY



**Caution:** Take care with applying pipette filler onto the pipette.

**Safety Warning:**

HCl is a strong acid.

Concentrated solutions of HCl are corrosive and give off irritating vapours. Avoid skin contact at all times. If spillage occurs use water to dilute and wash away.

## Risk Assessment;

Before you undertake this experiment, you must read through the experimental procedure, including the Risk Assessment sheet. There is a tear-off slip at the end of this manual for submitting your receipt number for the ChemCAL PreLabs module. Please sign this slip to acknowledge that you have read and understand the information on the Risk Assessment sheet.

## EXPERIMENTAL PROCEDURE

### Part A: Determination of the absorption spectrum of the acidic form of bromocresol green.

**Note:** Ensure that you have two cells that are matched. That is, when water is placed into both and the spectrophotometer is zeroed using one cell as the reference then the absorbance reading of the other cell should be very close to zero. This operation should be performed at about 450 nm.

1. Using a zippette, place the stock solution of the indicator ( $3.00 \times 10^{-5}$  M bromocresol green in 0.025 M HCl) into a clean and DRY container. Using the disposable pipettes supplied, rinse a spectrophotometer cell and two-thirds fill with 0.025 M HCl (reference cell). Rinse and similarly fill another cell with the indicator solution.

**Question 1:**

Which is the main form of the indicator, HIn or In<sup>-</sup>, present in the solution?

2. Record the absorbance A of the HIn solution at 20 nm intervals from 400 nm to 640 nm. Record additional readings at 5 nm intervals in the region of maximum absorption (i.e.  $\pm 20$  nm from the absorption maximum).

*Note: Remember that every time you change the wavelength you need to ‘zero the spectrophotometer’ with the reference cell at the new wavelength. Note that the mechanical system can be subject to “backlash errors” these are avoided by making the final change of wavelength by driving the monochromators in the same direction – e.g. from lower to higher wavelength.*

3. Plot absorbance A (vertical axis) against wavelength  $\lambda$  (horizontal axis) and from your plot determine the wavelength  $\lambda_{\max}$  of the absorption maximum (band maximum).
4. Calculate the molar absorption coefficient  $\epsilon$  ( $\text{L mol}^{-1} \text{cm}^{-1}$ ) of the HIn molecule at  $\lambda_{\max}$  from the Beer-Lambert Law:

$$A = \epsilon cl$$

where  $l$  is the distance the light travels through the solution and  $c$  is the concentration of the absorbing species.

## Part B: Beer-Lambert Law – Investigation of molar absorption coefficient ( $\epsilon$ )

1. Using a zippette, place the stock solution of  $3.00 \times 10^{-5}$  M bromocresol green in 0.025 M HCl into a clean and dry container. Prepare two other indicator solutions by diluting the stock solution 2.5 and 5 times with 0.025 M HCl as follows:
  - i. pipette 10.00 mL of stock solution into a 25 mL volumetric flask then add 0.025 M HCl to make up to the mark (see page 130).
  - ii. pipette 5.00 mL of stock solution into a 25 mL volumetric flask then add 0.025 M HCl to make up to the mark.
2. Measure the absorbance of the undiluted stock solution and the two diluted solutions at the band maximum and at 510 nm, using HCl as the reference.
3. Plot a graph of absorbance A (vertical axis) against concentration [HIn] (horizontal axis) for the two wavelengths you are using.



Pipette



Volumetric Flask

- For good graphing technique:*
- landscape format
  - graph occupy >80% of ordinate and coordinate range
  - label axes with correct scales and units
  - include a title
  - draw the line of best fit
  - show data points used for gradient calculation
4. Use the graphs and the Beer-Lambert Law (equation 13.5) to calculate the molar absorption coefficient of HIn at the two wavelengths. (Units of  $\epsilon$  are  $\text{L mol}^{-1} \text{cm}^{-1}$ )

### Question 2:

What is the information does the magnitude of molar absorption coefficient ( $\epsilon$ ) in Beer-Lambert Law provides about your sample indicator?

### Question 3:

Compare and comment on the magnitude of molar absorption coefficients of HIn determined at band maximum wavelength ( $\lambda_{\max}$ ) and at wavelength of 510 nm.

## Part C: Determination of $\text{pK}_a$ and $K_a$ for bromocresol green.

1. Dispense the stock solution of acetate/acetic acid buffer, pH = 4.76, via a zippette into a clean and DRY container. Make a solution of HIn in the buffer solution, by mixing 10.00 mL of indicator stock solution (using a pipette) with 10.00 mL of buffer (use a pipette) in a clean, dry beaker.
2. Measure the absorbance of this solution at the band maximum using the buffer solution as reference.

3. Determine the equilibrium concentration of HIn in the solution from its absorbance A (use the graph from part B). Then from the known initial concentration of HIn in the indicator/buffer solution you can calculate the equilibrium concentration of  $\text{In}^-$  by difference, since

$$[\text{HIn}]_{\text{equilibrium}} + [\text{In}^-]_{\text{equilibrium}} = [\text{HIn}] \quad (13.6)$$

**NOTE:** The initial concentration of the indicator  $[\text{HIn}]$  in the buffer solution is now half that of the stock indicator solution. Why?

4. Using the equilibrium concentrations of HIn and  $\text{In}^-$  and the pH of the indicator/buffer solution to calculate values of  $K_a$  and  $pK_a$  for bromocresol green accordingly to the given equilibrium expression (equation 13.3).

## RISK ASSESSMENT

### Nature of Chemical Hazard (check as appropriate)

- |   |  |  |                                      |
|---|--|--|--------------------------------------|
| <input checked="" type="checkbox"/> Corrosive   | <input checked="" type="checkbox"/> Irritant | <input checked="" type="checkbox"/> Pungent  | <input type="checkbox"/> Stench      |
| <input checked="" type="checkbox"/> Toxic   | <input type="checkbox"/> Carcinogenic        | <input type="checkbox"/> Mutagenic   | <input type="checkbox"/> Teratogenic |
| <input type="checkbox"/> Oxidising  | <input type="checkbox"/> Pyrophoric          | <input type="checkbox"/> Highly flammable  | <input type="checkbox"/> Cytotoxic   |
| <input type="checkbox"/> Non-commercial compounds where high risk is assumed based on personal experience (no data available) |  | <input type="checkbox"/> Non-commercial compounds where low risk is assumed based on personal experience (no data available) |                                      |
| <input type="checkbox"/> Reacts violently with water  |  | <input checked="" type="checkbox"/> Minimal risk   |                                      |

### Procedural hazards

- |   |   |
|---|---|
| <input type="checkbox"/> Large scale reactions, particularly involving solvent distillation | <input type="checkbox"/> High pressure reactions                                  |
| <input type="checkbox"/> Reactions in sealed tubes  | <input type="checkbox"/> Radioactivity above the specified OHS levels?            |
| <input type="checkbox"/> Potentially explosive reactions                                    | <input type="checkbox"/> Reactions in glass or other containers under high vacuum |
| <input type="checkbox"/> Other  |   |

### Special Precautions

- |   |  |                                    |
|---|--|------------------------------------|
| <input checked="" type="checkbox"/> Special eye protection  | <input type="checkbox"/> Safety shield                         | <input type="checkbox"/> Face mask |
| <input checked="" type="checkbox"/> Special clothing/gloves | <input type="checkbox"/> Is help necessary during the process? | <input type="checkbox"/> Any other |

### Special Location

- |  |                                       |   |                                |
|--|---------------------------------------|---|--------------------------------|
| <input type="checkbox"/> Fume Cupboard | <input type="checkbox"/> Schlenk line | <input type="checkbox"/> Biohazard laboratory | <input type="checkbox"/> Other |
|--|---------------------------------------|---|--------------------------------|

### Waste Disposal

- |                                     |                                   |  |  |
|-------------------------------------|-----------------------------------|--|--|
| <input type="checkbox"/> Sharps     | <input type="checkbox"/> Biowaste | <input type="checkbox"/> Cytotoxic waste | <input type="checkbox"/> Filter papers |
| <input type="checkbox"/> Filter aid | <input type="checkbox"/> Silica   | <input type="checkbox"/> Other           |  |

### Category of Risk (tick one)

- 3 Minimal risk
- 2a Low risk (Fume hood recommended)
- 2b Low risk (Fume hood/Schlenk line essential)
- 1a Significant risk (Chemical hazard)
- 1b Significant risk (Special location or facility)

Risk Assessed by: Coordinator: Sonia Horvat

Date: 6th January 2018

## Notes

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# Techniques and Instrumentation

## Balances

The balance, an instrument employed to determine mass, is one of the most important instruments in chemistry: Two types are commonly employed in the 1000-Level Laboratory.

### Denver (or Mettler) Top-loading Balance

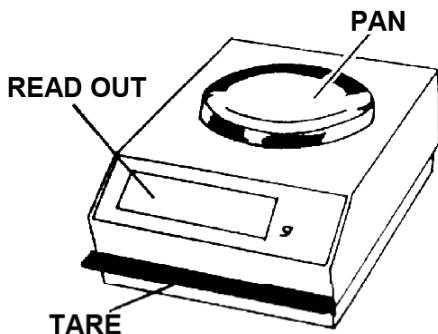


Figure 1: Denver (or Mettler) Top-loading Balance.

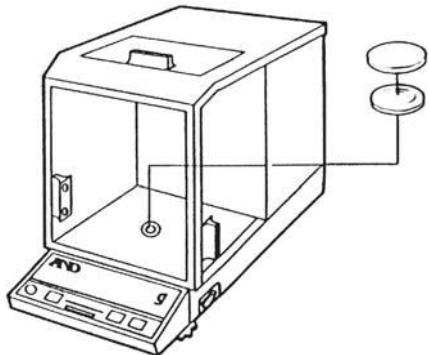
**Sensitivity and Applications:** For rapid weighing of 0 - 300 g to a maximum accuracy of 0.01 g. Used for weighing starting materials and establishing the yields of synthetic procedures.



**Method of Weighing:** Place a receiving vessel or weighing paper on the balance pan and press the tare bar. This will “tare” (zero) the balance. Load the vessel or paper with the material - the mass of material will be digitally displayed on the screen.

Top-loading  
balance

### AND 120 (or Denver A200) Analytical Balance



Analytical balance

Figure 2: AND 120 (or Denver A200) Analytical Balance.

**Sensitivity and Applications:** For weighing of 0 to 120 g to a maximum accuracy of 0.0001 g. To be used in all analytical procedures.

**Method of Weighing:** Using a top-loading balance, weigh into a clean dry glass vial the approximate amount of material required and proceed to the analytical balance. Tare the balance with the pan unloaded and then place your vial of compound on the balance pan. Record the combined mass of the vial and compound. Transfer the compound to the receiving vessel using a glass funnel and wash bottle if necessary (*do not wash the vial!*). Immediately re-weigh, on the same balance, the empty vial and record the mass of the vial. The mass of compound delivered to the receiving vessel is obtained by difference. Be sure the doors of the balance are closed during all weighing.

### Care of Balances

To ensure proper function and long and reliable service, exercise care when using these sensitive and expensive (*ca.* \$5,000) instruments.

- If a substance is accidentally spilled on the balance or balance pan, it must be removed at once. A brush or tissue may be used but in some cases a slightly moistened cloth is necessary. Failure to clean up may result in corrosion or impaired performance of the balance. Keep the balance area clean by removing any tissue or glassware you have used or brought into the area.
- Do not overload the balance by placing large items of glassware on it.

### General Apparatus and Operations

Each student, or student pair, should be allocated a box stocked with the apparatus and glassware required for the scheduled experiment. If a box cannot be found, or any equipment is missing, faulty or broken, consult your demonstrator and/or practical staff in the preparation room.

Some chemicals and ‘specialty’ items, e.g. filter paper and matches, are available from your demonstrator or the technical staff. New apparatus and techniques will be introduced and discussed during the PreLab talk presented by your demonstrator; this section allows you to prepare for first encounters and refresh your memory before subsequent ones.

### Burners

The Bunsen burner (Fig. 3) is widely employed for heating in chemical laboratories. A collar at the base of the burner stem is used to adjust the gas/air mixture of the flame and hence its temperature. To operate the burner, attach it by means of rubber hosing to a gas outlet (colour coded orange).

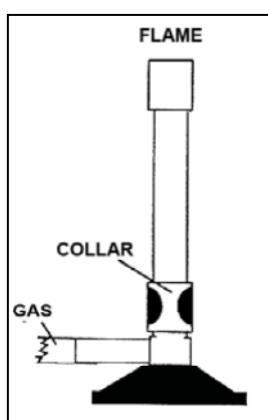


Figure 3: Bunsen burner.

Using the adjustable collar, close off the two holes at the base of the burner, turn on the gas, wait a few seconds and use a match or lighter to light the burner. Adjust the burner to the desired heat; low and high temperature flames are yellow and blue, respectively. When in use, make sure the burner is stable and away from clothing, hair and other flammable objects. When not in use turn it off or adjust to the more visible yellow flame. After use ensure that the gas tap is turned off before disconnecting the rubber hose. Treat laboratory gas supplies with the same care exercised in domestic situations. Never heat flammable solvents with a Bunsen burner or any other naked flame. A burner is normally used in conjunction with a tripod and gauze mat, upon which the vessel to be heated rests.

## Hot Plates and Water Baths

Electrical devices known as hot plates, which function just like their kitchen counterparts, are employed to heat samples in the laboratory. **It is impossible to visually gauge the temperature of a hot plate so be careful not to inadvertently burn yourself on it.** Hot plates should not be employed to heat flammable solvents. Water or steam baths are safe for use with flammable solvents; add or remove the concentric rings upon which the vessel will rest so to maximise the surface area of glass in contact with steam. **Take care, as steam is also capable of causing serious burns.**

## Wash Bottles

Number of solvents are dispensed using flexible plastic wash bottles. If empty, ensure that the bottles are filled with the solvent stated on the side of the bottle. Do not contaminate the bottles with foreign substances and do not be tempted to use them as water pistols - one day the use of a bottle containing a toxic or corrosive solution will cause injury.

## Distilled Water

Distilled water must be used in all synthetic reactions and analytical procedures. Normal tap water should be employed in apparatus such as condensers and when washing up. Distilled water outlets are available above each sink (the taps with green handles).

## Glassware

Most of students are familiar with the function and operation of common items of glassware; if in doubt ask your demonstrator. In syntheses, a measuring cylinder is generally employed to measure volume. When performing analyses, special volumetric glassware must be employed (see below). Ensure that all glassware is clean before and after use. Never heat glassware suddenly or unevenly.

## Titrimetric (Volumetric) Analysis

The fundamental unit employed in measuring the volume of a liquid is the litre. A closely related unit is the millilitre (mL), where:

$$1 \text{ litre} = 1000 \text{ mL}$$

$$1 \text{ cm}^3 = 1 \text{ mL}$$

$$1 \text{ dm}^3 = 1 \text{ litre}$$

The most commonly used apparatus in volumetric or titrimetric analysis are volumetric flasks, burettes and pipettes. These apparatus, are designated Class A or Class B depending upon the stated tolerance or variation from the stated volume. For example, a Class B 250 mL volumetric flask would hold  $250 \pm 0.2 \text{ mL}$  at  $20^\circ\text{C}$  (Class A:  $250 \pm 0.1 \text{ mL}$ ).

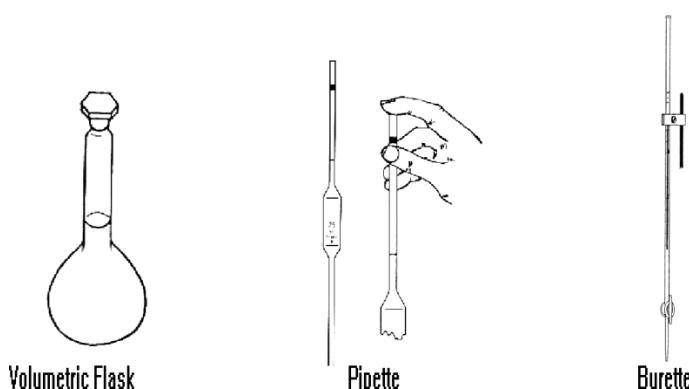


Figure 4: Volumetric apparatus.

## Cleanliness

For good results, the apparatus must be clean and free of grease. As a test for cleanliness fill the apparatus with water and allow it to empty; the item is clean if an unbroken film of water remains. If the water collects in drops the glass is dirty and should be cleaned or exchanged for a clean unit. Specific handling instructions must be followed for best results as given below.

## Volumetric Flasks

A volumetric flask is a flat-bottomed pear shaped flask with a long neck (Figure 4). It is employed to prepare solutions of known concentration by placing a known mass of solid in the flask using a funnel and wash bottle, adding solvent and mixing to dissolve the solid and finally diluting to the prescribed volume. It may also be used for precise dilution by the addition of a known volume, usually by pipette (see later), followed by dilution. A thin line etched around the neck of the flask indicates the level to which the flask must be filled for it to contain the stated volume of solution.



Volumetric Flask

When properly filled, the bottom of the solvent meniscus should be tangential to the graduation mark, the front and back of which should be seen as a single line. At the final stage of filling, solvent should be added drop-wise with a disposable pipette. It is best to stand up straight and hold the etched line at eye level at the final stage of filling. After placing a stopper on the flask, solutions should be thoroughly mixed by inverting the flask about 10 times.

If not completely dry the flask should first be rinsed with the solvent that it will contain. Volumetric flasks should never be heated.

## Pipettes

Pipettes consist of a central cylindrical bulb joined at both ends to narrower tubing - a calibration mark is etched around the suction end and a fine tip is drawn at the delivery end (Figure 4).



Pipette

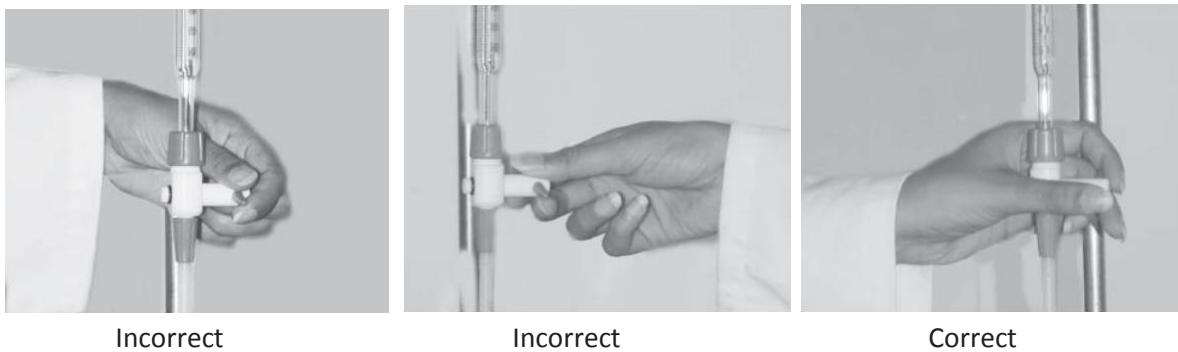
The pipette is designed to deliver a small *constant volume* of liquid known as an *aliquot*. Pipettes are first rinsed with distilled water. This is followed by rinsing twice with the solution it is to deliver and finally filled beyond the graduation line using a pipette-filler (suction device) - for safety reasons *never fill by sucking with your mouth*.

Using your index finger (or the pipette-filler if there are difficulties with the 'index finger' method), allow the liquid to drain from the tip of the pipette until the meniscus of the solution is tangential to the graduation mark when the pipette is held vertically. Any drops adhering to the tip should be removed by stroking the tip across a glass surface. The aliquot is then delivered to the receiving vessel, the tip of the pipette touching the wall during delivery and for three seconds afterwards. Droplets on the glass wall of the vessel need to be washed down into the bottom of the vessel before the titration. A small amount of liquid will remain in the tip of the pipette and no attempt should be made to add it to the receiving flask.

**Do not pipette directly from stock solutions - instead pour the solution into a clean dry beaker before pipetting.**

## Burettes

A burette is a long cylindrical and continually graduated tube of uniform bore. It is closed at the bottom by a glass or teflon stopcock (Figure 4). Burettes are designed to deliver variable volumes of solution; these are generally called *titres*.



**Figure 5:** Correct and Incorrect Handholds for a burette.

The burette is first rinsed with distilled water and followed by the solution it is to deliver. With the assistance of a funnel, the burette is filled with the solution; the funnel is *immediately removed* from the burette to avoid drops from it entering the solution during the experiment. Ensure that the tip of the burette is filled with solution (and no air bubbles are present) when the initial reading is taken. The meniscus should be tangential to the graduation line and this should be recorded with “eyes level” with the top of the solution. The height of burette can be adjusted by moving up or down along the clamp. A piece of white cardboard placed behind the burette may assist reading. **For safety reasons, NEVER climb on stools in making burette readings.**

Following the delivery of the solution a second final reading is taken and the volume delivered determined by difference. During delivery of the solution, control the stopcock by placing the fingers of the left hand around the back of the burette stopcock and the thumb in front. Hold and adjust the tap of the stopcock between the thumb and the fore and middle fingers, while swirling the solution in the receiving flask with the right hand (Figure 5). If you are left-handed, consult your demonstrator about adjusting your burette so that you can use it correctly.

**Important:** Burettes must be read to 2 decimal places at all time.

## Titrations

A titrimetric or volumetric analysis simply determines the volume of a solution of accurately known concentration which is required to react *quantitatively* (completely) with a solution of the substance being determined. The solution of accurately known concentration is called the *standard solution*. This permits, through application of appropriate chemical equations, the molar equivalent and therefore mass, of the unknown substance to be calculated.



Volumetric  
techniques

Typically, an aliquot of the *analyte* (unknown) is pipetted into a clean (not necessarily dry) Erlenmeyer flask. This solution is *titrated* by carefully adding the *titrant* (known standard solution) from a burette until the *endpoint* of the reaction is reached. The endpoint is normally indicated by a change in colour of a suitable *indicator*, added before or during the titration.

Knowledge of the volume of the aliquot (dictated by pipette size), the titre, the concentration of the titrant and the chemical equation for the reaction, permits the calculation of the unknown concentration and therefore the mass of the unknown in the analyte.

## FILTRATION

Filtration is the separation of precipitates from their mother liquor by passage through a porous barrier, typically a filter paper or a porous glass ‘frit’. Crystalline products are generally collected by

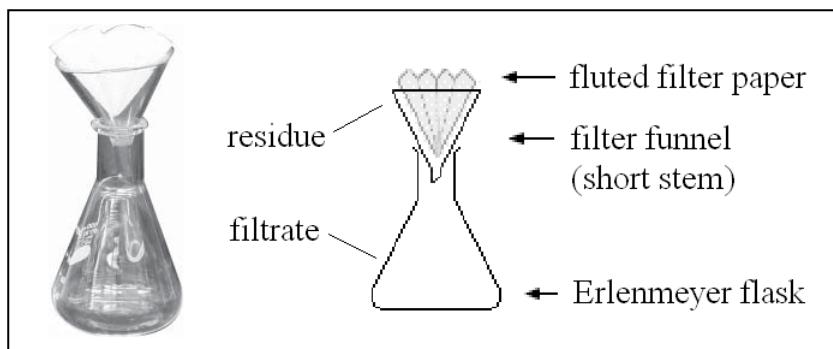


Figure 6: Gravity filtration.

this technique. As well, it may be used to remove solid impurities from suspensions; in this case the *filtrate*, the clear solution that passes through the filter, is the desired ‘fraction’ from the filtration.

### Gravity Filtration

In gravity filtration (Figure 6), a folded or fluted filter paper is placed in a funnel supported above the collecting vessel and the mixture to be filtered is poured into the filter paper such that the solution flows through the paper into the collecting vessel. (Ensure that the mixture does not spill over the top edges of the filter paper.)



Gravity Filtration



Fluted filter paper

### Vacuum Filtration

In vacuum filtration, the passage of the solution through the filter is accelerated by application of a vacuum. A Hirsch or Buchner funnel is first fitted with a suitably sized filter paper (one which just covers the holes in the porcelain funnel). Then the apparatus (shown in Figure 7) is set up. The filter paper is positioned in the funnel so that all holes are covered, then it is wetted with the solvent about to be filtered. Application of a vacuum to the side arm flask draws the paper onto the funnel and seals the edges to the porcelain, thereby preventing solid from being drawn under the paper into the collecting vessel.

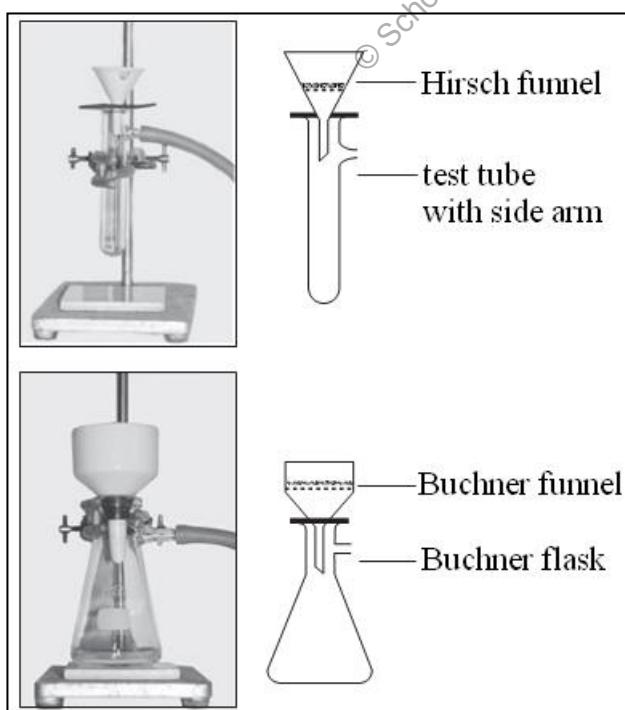


Figure 7: Vacuum filtration.

When rinsing the crystals collected by filtration, ensure that the washing solvent contacts all the solid being washed (swirling solid and solvent on the filter with a spatula before applying full vacuum is helpful here). When disassembling the apparatus, remove the vacuum hose from the side arm flask before turning off the vacuum. This will prevent contamination of the filtrate and is



Hirsch vacuum filtration



Buchner vacuum filtration

especially important if the filtrate is to be collected. Filtration is an essential step in the purification of compounds by recrystallisation.

## PURIFICATION OF COMPOUNDS

Generally, solids are purified by recrystallisation and liquids by distillation.

### Recrystallisation

Recrystallisation of a solid involves:

- i. dissolving the solid in hot solvent;
- ii. filtering the hot solution to remove insoluble impurities, if these are present;
- iii. cooling the filtrate until crystallisation is complete;
- iv. collecting and washing the crystalline solid;
- v. drying the solid.

A solvent is generally chosen in which the impurities are more soluble than the substance being purified. When crystals of the substance form on cooling the hot solution, any impurities remain in the solution. This is because foreign molecules are seldom of the correct shape and charge distribution to fit into the crystal structure of another compound.

The compound is placed in an appropriate size conical flask or test tube, a small amount of the recommended solvent is added, and the mixture is heated to boiling. A boiling water bath is used for solvents with boiling point < 80 °C and electrical hot plates for solvents with boiling points > 80 °C. If the compound has not dissolved after several minutes of boiling and stirring, more solvent is added drop-wise until dissolution occurs. If the solution contains a small amount of undissolved material, do not add more and more solvent in the hope of dissolving this; remove undissolved material by hot filtration.

If the solution is highly coloured, some of the coloured impurities may be removed by adding a small amount of charcoal (remove the flask from the heat before adding charcoal), boiling for a few minutes and then filtering the hot solution. If the solvent is a volatile compound, e.g. ethanol or acetone, the filtration is carried out by gravity through a fluted filter paper as shown in Figure 6. Filtration under suction through a pre-heated Buchner or Hirsch funnel may be used if the solvent is not volatile e.g. hot water (Figure 7). An alternative mode of filtration involves using a Pasteur pipette fitted with a plug of cotton wool as a filter apparatus - this apparatus may be pre-heated with hot solvent if a hot-filtration is required. A demonstrator will show you this technique if it is required.

If a mixed solvent is used for recrystallisation, the compound is dissolved in the minimum volume of hot solvent in which the compound is readily soluble, and then the other warm solvent is added drop- wise until a faint cloudiness appears. The mixture is then heated until it is clear (sometimes a drop of the first solvent is added to clear the solution).

If the use of charcoal is required for the crystallisation from mixed solvents, then charcoal is best added before the addition of the second solvent. The solution of the compound in the first solvent is boiled with charcoal, the charcoal filtered off and then the second solvent is added to the hot filtrate, as above.

In all cases the hot solution is allowed cool slowly, whereupon crystallisation of the compound should occur. Occasionally crystallisation is slow to start and can be aided by adding a small crystal of the un-recrystallised compound to the solution (saved for that purpose) or by scratching the side of

the flask (or tube) with a glass rod. If no crystallisation occurs after these techniques have been tried, then most likely too much solvent was used in dissolving the compound. The excess solvent may be boiled off on a water bath or a hot plate.

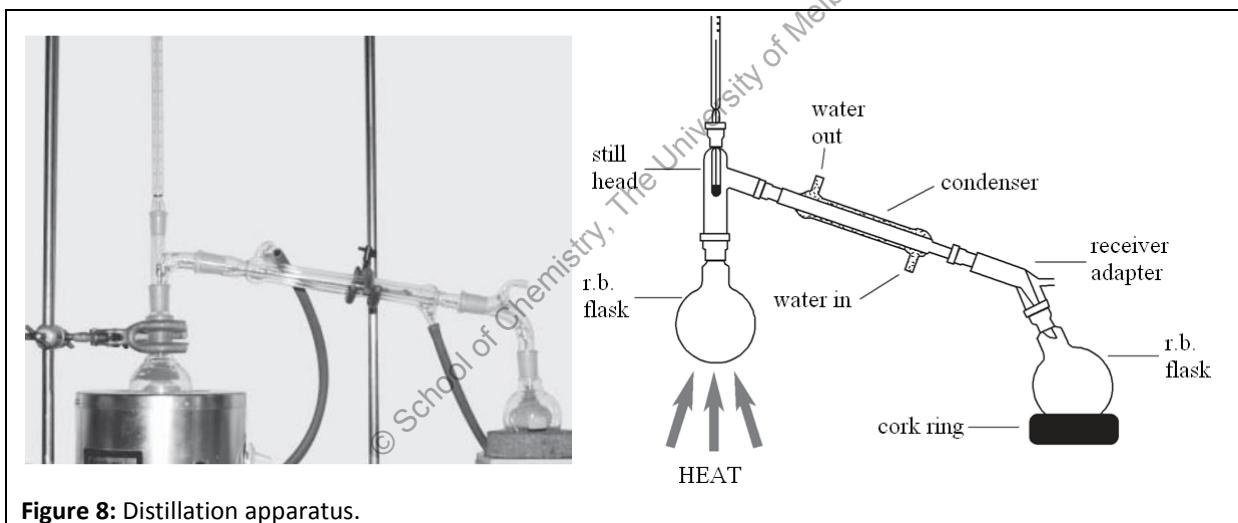
The recrystallised compound is collected by filtration under suction using a Hirsch funnel for small amounts of compound or a Buchner funnel for large amounts (Figure 7). The crystals are pressed with the spatula onto the filter paper in the funnel to drain them thoroughly.

The crystals are washed by first releasing the suction, adding a small amount of solvent and then sucking them dry as quickly as possible. A second crop of crystals can sometimes be obtained by concentrating the mother liquor, but it must be kept separate from the first crop until its purity is checked.

Crystals are dried by leaving them spread on a watch glass until all the solvent has evaporated. For compounds with melting point well above 100 °C, heating the crystals on the watch glass over a boiling water bath may speed up the drying.

### Distillation

Distillation is a method of purifying liquids based on differences in boiling point. A simple distillation set-up is given in Figure 8.



**Figure 8:** Distillation apparatus.

This simple assembly is not very efficient and will only separate liquids of very different boiling points: about 30-40 °C difference in boiling points.

In setting up the apparatus for distillation, the following points must be observed:

- The distillation flask, the condenser and the receiver flask must be clamped so that the whole assembly is secure, but not strained. The receiver- adaptor must have a vent open to the atmosphere. Distillation must not be conducted in a closed system.
- The distillation flask must not be more than three-quarters full of liquid and a boiling chip must be added before the liquid is heated.

- Liquids boiling below about 80 °C are heated on a water bath, and an electrical heating mantle is used for liquids boiling above 80 °C. A simmerstat is used with the heating mantle to allow temperature control.
- The heating must be stopped before the distillation flask becomes completely dry. Many liquids, particularly alkenes and ethers may contain peroxides, which become explosive in highly concentrated residues.
- Make a constant check to ensure that water is running (a gentle flow is sufficient) through any condenser which you are using and that the connections are sound.

## CRITERIA OF PURITY OF COMPOUNDS - PHYSICAL CONSTANTS

Physical properties such as melting point (m.p.), boiling point (b.p.), refractive index and density are used in identification and characterisation of many compounds. In addition, the observed melting point or boiling point may give information about the purity of the substance under consideration.

### Melting point of a substance

The melting point of a substance is defined as the temperature at which the liquid and solid phases exist in equilibrium with one another without change in temperature. Most pure organic solids have sharp melting points, but since very few substances melt instantaneously, a melting range is in fact determined - *e.g.* 120-121 °C. This shows the temperature at which melting was first observed (120 °C) and the temperature at which melting was complete (121 °C).

Pure compounds have a very short melting range (about 1 °C). A broad range of several degrees is usually an indication of impurity. Very generally, impurities, including solvents, will cause broadening and lowering of the melting point.

This effect can be used as an indication of the identity of two crystalline samples having the same melting point; if they are the same compound, the melting point of an intimate mixture of the two will be unchanged. If the compounds are not identical a depression of the 'mixed melting point' will be observed.

### Melting point determination

Introduce the dry sample into the capillary by tapping the open end in a little pile of finely ground compound. Invert the capillary and shake the compound down by tapping the bottom of the tube on the table top or by using the "tube tapper" of the DigiMelt melting point apparatus. A 1-2 mm length of compound in the bottom of the tube is ample. If too large a sample is used the time required for complete melting is longer and the temperature range larger. Determine the melting point using the DigiMelt melting point apparatus.

## Operation of the DigiMelt Melting Point Apparatus

**Please Note:** The required settings in the apparatus have been programmed by the laboratory staff, so please do not touch any of the *YELLOW* or *BLUE* buttons.



1. Load the capillary tube with the sample (1-2 mm) by tapping the open end in a small pile of finely ground compound.
2. Insert the capillary tube into the hole labelled “*tube tapper*” of the melting point apparatus.
3. Press the “*tube tapper*” button to pack your sample down to the bottom of the capillary tube.
4. Push the “*start/stop*” button to preheat the block to the start temperature (the “*preheat*” LED will light).
5. When the green “*ready*” LED becomes lit, the block is on hold at the start temperature.
6. Insert your capillary tube in the centre hole in the block and wait a few seconds for the capillary tube/sample to come to the “*preheat*” temperature.
7. Push the “*start/stop*” button to begin the temperature ramp (it is preset at 2 °C/minute and the “*melt*” LED will light).
8. Observe your sample during the temperature ramp.
9. A few degrees below the melting point the sample will shrink and may appear soggy; the sample is sintering. The onset of sintering can be recorded, but sintering is not considered to be melting. The beginning of the melting point is defined by the appearance of the first distinct droplet of liquid. The end of the melting ‘point’ range is the disappearance of the last crystals (Figure 9) and the sample turns into a clear liquid.
10. These two temperatures are the start and end temperatures for your melting point range.
11. Remove the capillary tube and dispose in the provided container next to the melting point apparatus.
12. The melting point apparatus will automatically reset itself ready for the next student.
13. The temperatures defining the melting point range should only be quoted to the nearest 0.5 °C. Even this claim to precision is rather illusory because the melting point apparatus is not perfectly calibrated and, more significantly, your judgements of the end and (especially) the beginning of melting are subjective.

MP determination

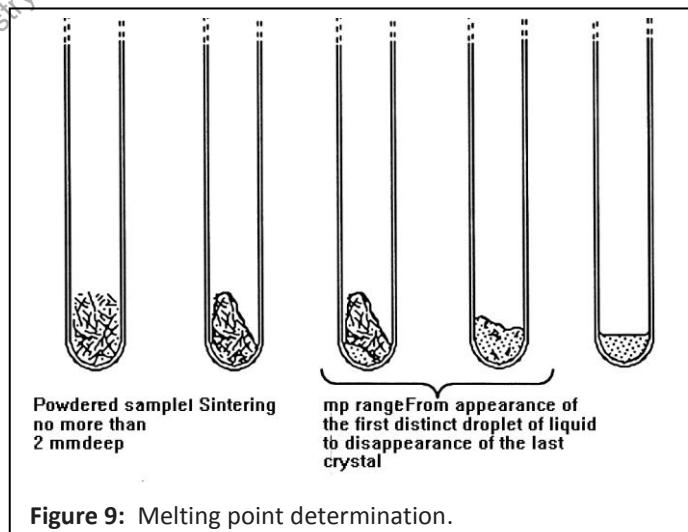


Figure 9: Melting point determination.

## **EXTRACTION**

### **Simple extraction and separation of two immiscible liquids**

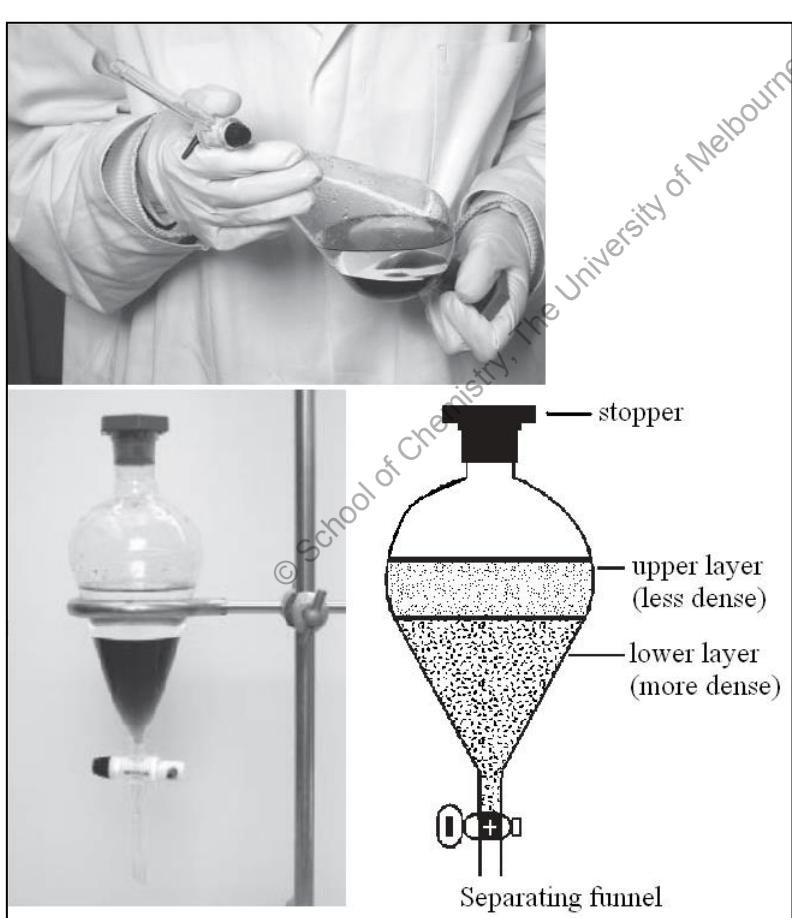
Support the separatory funnel in a ring clamped to a stand.

Pour into it the solution to be extracted and the extracting solvent.

Stopper the separating funnel, remove it from the ring and, holding the stopper and neck firmly with one hand and the stopcock with the other (see Fig. 10), then shake the funnel gently. Turn the funnel upside down and open the stopcock to release the internal pressure. Close the stopcock, shake more vigorously and again release the pressure.

Repeat this procedure four or five times, put the funnel upright into the ring and let it stand undisturbed until the two liquids have completely separated.

Remove the stopper and drain the lower (denser) layer into an Erlenmeyer flask, closing the stopcock, as the meniscus separating the two layers just reaches it. The upper layer can then be drained into another Erlenmeyer flask.

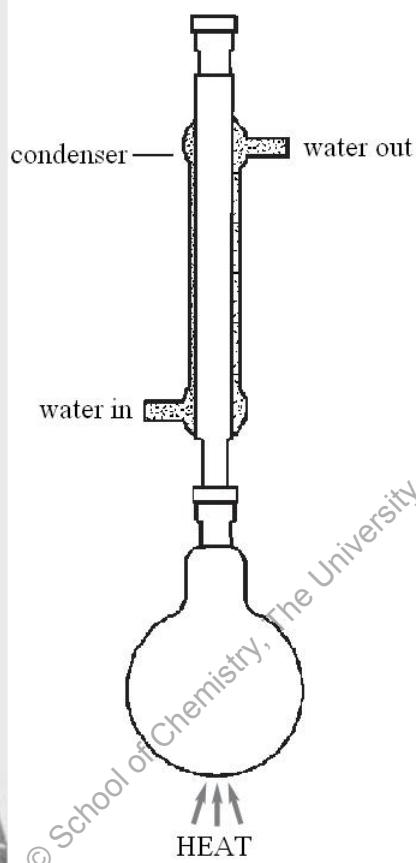


**Figure 10:** Solvent Extraction.

## Refluxing

Refluxing or boiling under reflux is the technique of boiling the mixture and condensing the vapour so that it returns to the mixture. A typical set-up for reflux is given in Figure 11.

Note: Boiling-chips must be added to the mixture before heating is started, otherwise the mixture will overheat and bump. Fresh boiling chips must be added if refluxing is interrupted. Make a constant check to ensure that water is running (a gentle flow is sufficient) through any condenser which you are using and the connections are sound.



Reflux condensor

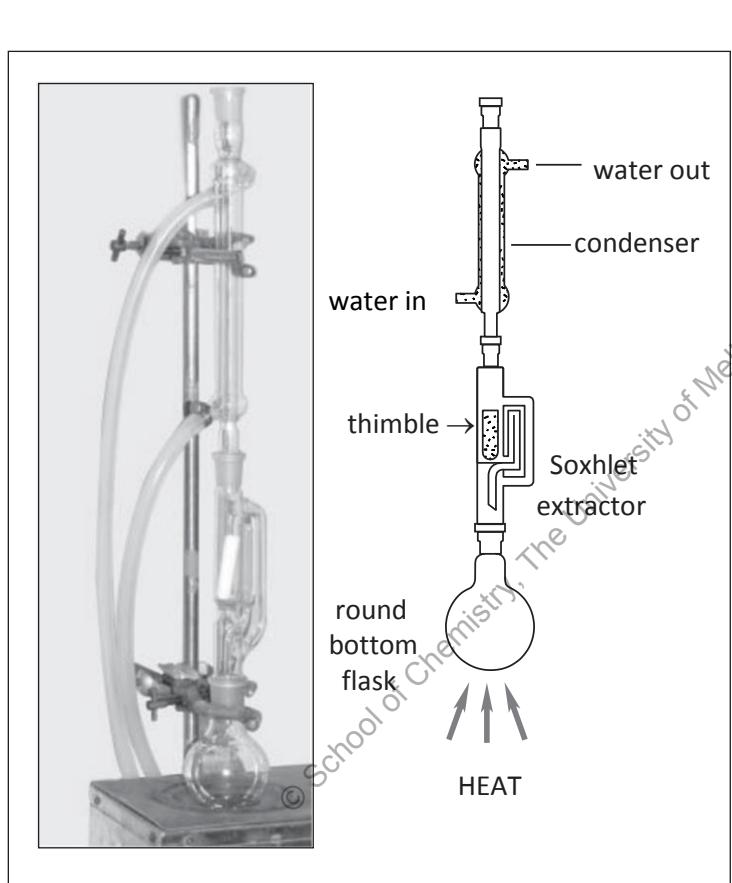
**Figure 11:** Reflux Apparatus.

## Soxhlet extraction

A Soxhlet extraction apparatus (Figure 12) is used for separation of a compound from a solid mixture by continuous solid-liquid extraction.

The solid is placed in a porous thimble in the glass Soxhlet extractor as shown, and the extracting solvent in the boiling flask below. The solvent is heated to reflux and the distillate collects in the Soxhlet and contact with the solid in the thimble and effects the extraction. After the Soxhlet fills to the level of the upper turn in the siphon arm, the solution empties into the boiling flask.

The process is continued for the time it is necessary for effective extraction of the desired compound, which will be collected with the solvent in the boiling flask.



Soxhlet extraction

Figure 12: Soxhlet Extraction.

## TREATMENT OF ERROR IN EXPERIMENTAL DATA

Reference: *Chemistry*<sup>3</sup>, Burrows, Holman, Parsons, Pilling and Price 3<sup>rd</sup> ed. 2017:

Making the measurements: pages 518 – 521;

Significant figures and rounding of decimals: pages 1308 – 1309.

*Chemistry*, Blackman, Bottle, Schmid, Mocerino and Wille 2<sup>nd</sup> ed. 2012:

Uncertainties and significant figures: pages 30 – 34.

### Reliability and precision

When a specified quantity is measured several times by one observer using the same method, it is quite usual for a set of different values to be obtained. Certain questions then arise:

What is the *best* value to take as the result of the measurement?

What *index of reliability* can be placed on the result?

What upper and lower bounds can be placed around the result so that the range of values thus defined can be said almost certainly to enclose the true value of the required quantity?

### Mean

- The best value when n measurements have yielded the values  $x_1, x_2, \dots, x_n$  is the *average* or *arithmetic mean*,  $\bar{x}$  :

$$\bar{x} = \frac{\sum_{i=1}^{i=n} x_i}{n}$$

#### Example:

If volumes 21.74, 21.76, 21.72, and 21.70 mL were obtained by scientist A in four equivalent titrations, the best value to take for the volume would be 21.73 mL.

$$\bar{x} = \frac{21.74 + 21.76 + 21.72 + 21.70}{4} = 21.73 \text{ mL}$$

- How reliable is this figure however? Consider a set of results obtained by scientist B: 21.74, 21.80, 21.61, and 21.77 mL, for which the mean is also 21.73 mL. Clearly these values are more widely scattered than those obtained by scientist A. Perhaps B's method is less reliable or B is not such a careful worker and the result is not so precise as that obtained by A. The actual causes of the greater variability in B's results need not concern us here, but we want a way of specifying it.

### Mean deviation of an individual measurement

B's four values differ from their mean by the amounts: +0.01, +0.07, -0.12, and +0.04 mL, called 'deviations from the mean'. The sum of these deviations (using absolute values irrespective of sign) is 0.24 mL, and the *mean deviation*  $0.24/4 = 0.06$  mL for each value is a measure of the variability of B's work. If A's values are treated similarly the averaged deviation from their mean is  $(0.01 + 0.03 + 0.01 + 0.03)/4 = 0.02$  mL. The greater precision of A's values is shown by their much smaller mean deviation.

### **Confidence range**

It is convenient<sup>‡</sup> to record B's result as  $21.73 \pm 0.06$  mL, giving a range of values as an indication of its reliability. Similar with A's value, with its higher precision, is expressed by writing  $21.73 \pm 0.02$  mL. Writing B's result in this way gives a range of values 21.67 to 21.79 inside which the true value of the volume most probably lies. This is sometimes called the 'confidence range' of the measured value. B's result, based on values showing greater variability, has a much wider confidence range and is correspondingly less reliable. We say that A's result is 'correct (or accurate) to 0.02 mL' or 'to 0.09%' since 0.02 mL is 0.09% of 21.73 mL.

### **Rejection of outlying measurements**

It is important to realise that the concordance of a set of measured values cannot validly be improved by simply rejecting any values that are discordant/not concordant. Hence B must not throw away his second and third values (21.80, 21.61) just because they have large deviations from the mean and seem to be 'in error'. *If we know of no reason why they must be wrong, they should be retained*, as we have no reason to suppose they are any less reliable than the rest of the set. Rejecting them could certainly alter the mean. Conversely, of course, if we know why one value in a set is different from the others it *should* be rejected.

### **Systematic errors**

The scatter of results described above arises from '*random errors*'; the measurements are evidently subjected to irregular fluctuations, which can occur in either sense, causing a particular one to be too high or too low by an unpredictable amount. As we have seen, the problem can be allowed for by using a mean of several values, and by quoting the average deviation from the mean as a measure of the precision of the measurements.

A second type of unreliability of a measurement is attributed to '*systematic errors*' - inaccuracies in the experimental value which are present as constant errors in every measurement obtained by a particular method and which cannot be reduced or allowed for by averaging a set of measurements. They may arise from a fault in the measuring apparatus or from inadequacy on the part of the experimenter.

Systematic errors can be reduced only by careful experimental design. One must use a reliable method and carefully calibrated apparatus, both chosen so that they measure exactly what is required.

### **Precision and accuracy**

We must distinguish between the *precision* of a measurement and its *accuracy*. A result may be precise, *i.e.* show little variation between repeated determinations, but it need not necessarily be accurate. Self-consistent measurements of a quantity may still be subject to systematic errors and hence differ significantly from its true value.

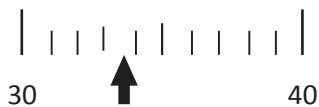
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<sup>‡</sup> This use of the mean deviation to specify a confidence range is not the only way used to record the result of a set of measurements. Indeed, in more advanced work, a quantity known as the 'standard deviation' is used and in many situations, the exact probability that the true value will lie within a prescribed range around the experimental average can be calculated.

### Estimation of precision from single measurements

On many occasions only one measurement of a quantity is made so there is no possibility of estimating precision in the manner just described. In this case, an estimate of the uncertainty is made from the experimenter's ability to read a scale.

The graduations can be imagined further divided by eye:



The pointer is between 33 and 34 and if the interval between could be subdivided into five equal parts, the reading could be given as  $33.2 \pm 0.2$ , meaning that the best value lies between 33.0 and 33.4. In cases where it is felt that the interval could be reliably subdivided into ten equal parts, the reading could be taken as  $33.2 \pm 0.1$ , meaning  $33.1 \leq \text{value} \leq 33.3$ .

Digital instruments display numbers in which the last figure has been rounded off to the nearest digit. We say that this last figure is reliable to  $\pm 0.5$ , or has an error of  $\pm 0.5$ .

### Estimating the error from repeated measurements

In many instances, e.g. chemical analysis, repeated measurements are conducted and the variation of these measurements provides a way of estimating the uncertainty of the reading. While the most common approach to estimating the precision of repeated measurements is to use the standard deviation, a simple approach is used here for relatively small sample sizes to report the mean (or average) deviation.

$$\text{mean deviation of } n \text{ readings} = \frac{\sum_{i=1}^{i=n} |\bar{x} - x_i|}{n}$$

#### Example:

If volumes 21.74, 21.76, 21.72, and 21.70 mL were obtained by scientist A in four equivalent titrations and the average volume is 21.73 mL, the calculation for the mean deviation is:

$$\begin{aligned}\text{Mean Deviation} &= \frac{|21.74 - 21.73| + |21.76 - 21.73| + |21.72 - 21.73| + |21.70 - 21.73|}{4} \\ &= \frac{0.01 + 0.03 + 0.01 + 0.03}{4} \\ &= \pm 0.02\end{aligned}$$

### Error in a quantity calculated from measured values

1. If  $Z$  is derived from the sum or difference of two measured quantities  $X$  and  $Y$ , subject to errors  $x$  and  $y$  respectively, then the largest error possible in  $Z$  will be simply:

$$z = x + y$$

Note that it is the sum of the absolute errors in this case. Thus, if burette readings are reliable to the nearest 0.01 mL, then titres are in fact reliable only to 0.02 mL.

**Example:**

Consider weighing a sample by difference:

$$\text{Mass of weighing bottle + sample initially} = 18.1425 \text{ g}$$

$$\text{Mass of weighing bottle + sample after some sample transferred to flask} = 18.0271 \text{ g}$$

Hence,

$$\text{Mass of sample transferred to flask} = 0.1154 \text{ g.}$$

Taking the weighing error in each weighing as  $\pm 0.2$  mg, the (maximum) error in the sample weight is  $0.0002 + 0.0002 = 0.0004$  g. Hence the sample weight is recorded as  $0.1154 \pm 0.0004$  g, the fractional error being  $4/1154 = 0.35\%$ . Note that although the relative error in each weighing is only about one part in 90 000, the error in the result is about one part in 300.

When difference in two large and comparable quantities are involved, the relative error in the result can be very large compared with the relative errors in the initial quantities.

2. If  $Z$  is a quantity derived from a measured value  $X$  by the calculation:

$$Z = AX,$$

where  $A$  is some factor, a new question arises: If  $X$  is subject to an error  $x$  what is the resulting error  $z$  in  $Z$ ? This is best answered by differentiation, for

$$\frac{\delta Z}{\delta X} = A = \frac{Z}{X}$$

since, for the change  $\delta Z$  in the value of  $Z$  resulting from the small change  $\delta X$  in  $X$ , we have

$$\frac{\delta Z}{\delta X} \approx \frac{Z}{X}$$

It follows that

$$\frac{\delta Z}{Z} \approx \frac{\delta X}{X}$$

$$\text{i.e. } \frac{z}{Z} \approx \frac{x}{X}$$

and the *relative error* (or fractional error) in  $Z$  is the same as that in  $X$ .

If, in another situation,  $Z$  is derived from  $X$  by the calculation represented by:

$$Z = \frac{B}{X} \quad \text{where } B \text{ is some factor,}$$

$$\text{Then ... } \frac{\delta Z}{\delta X} = -\frac{B}{X^2} = -\frac{Z}{X}$$

and now ...

$$\frac{z}{Z} \approx -\frac{x}{X}$$

If we pay no attention to the *sign* of the errors the same result is obtained as before: *viz.* the relative error in  $Z$  is the same as that in  $X$ .

3. If  $Z$  is derived from the product of two measured values  $X$  and  $Y$ :

$$Z = AXY,$$

an error in  $X$  will give rise to an equal relative error in  $Z$  and so also will an error in  $Y$ . When  $X$  and  $Y$  are quite independent of each other the relative error in  $Z$  will, at worst, be the sum of these two relative errors:

$$\frac{z}{Z} \approx \frac{x}{X} + \frac{y}{Y}$$

Note that we have assumed the two errors have acted in the same direction, reinforcing one another and making the resulting error in  $Z$  greatest. This, of course, need not happen - but such a calculation is useful as it enables us to put an upper limit on the uncertainty in  $Z$  arising from errors in  $X$  and  $Y$ .

It follows from the second part of 2 that the same result is true for a calculated  $Z$  derived from the quotient of  $X$  and  $Y$ .<sup>§</sup>

**Example:**

Calculate the concentration (including the percentage error) of a sodium hydroxide solution, if  $27.07 \pm 0.05$  mL of  $0.2000 \pm 0.0005$  M hydrochloric acid are required to neutralise  $25.00$  mL of the sodium hydroxide.

Answer:  $[NaOH] = (27.07/25.00) \times (0.200) = 0.2166$  M

*Error calculation:*

The fractional error in the final NaOH concentration is the sum of the fractional errors in all the quantities used in the calculation.

$$\text{Fractional error in [HC]} = 0.0005/0.2000 = 0.0025.$$

$$\text{Fractional error in vol of HCl} = 0.05/27.07 = 0.0018.$$

The error in the volume delivered by the 25 mL pipette is  $\pm 0.04$  mL, so

$$\text{Fractional error in the vol of NaOH} = 0.04/25.00 = 0.0016.$$

$$\text{Fractional error in [NaOH]} = 0.0025 + 0.0018 + 0.0016 = 0.0059$$

That is 0.6%

$$\text{Error in [NaOH]} = 0.0059 \times 0.2166 \text{ M} = 0.0013 \text{ M}$$

$$i.e. [NaOH] = (0.217 \pm 0.001) \text{ M}$$

The error is rounded off to one significant figure (which underestimates it slightly) and the number of significant figures quoted for the concentration is adjusted to match the error.

<sup>§</sup> In advanced work, when standard deviations are used, more sophisticated considerations of probability and statistical theory enable a somewhat more reliable estimate to be made of the uncertainty of a calculated result. The approximate methods described here will be quite satisfactory in this course, however.

# ChemBytes – Video demonstration of lab techniques

## Using an analytical balance

The analytical balance is a precision instrument. Balances like this can detect mass differences of 0.0001 g.



Analytical balance

<http://www.metacd.com/r/m/ukhcnnyr/OtetGej>

## Using a top-loading balance

A top loading balance like this can detect mass differences of 0.01 g. It is used typically for synthesis, rather than in precise analysis.



Top-loading balance

<http://www.metacd.com/r/m/ukhcnnyr/kFyrkzj>

## Preparing a solution of accurately known concentration

Making up solutions of accurately known concentration is an essential laboratory skill.



Volumetric Flask

<http://www.metacd.com/r/m/ukhcnnyr/O8OH2Hv>

## Transfer of a precise volume of solution using a pipette

Bulb pipettes like this can deliver solution volumes with a reproducibility of better than 0.005 mL.



Pipette

<http://www.metacd.com/r/m/ukhcnnyr/LxyQZxG>

## Carrying out a precise titration

Precise volumetric titration is an important basic procedure in developing your skills for careful and accurate solution and materials handling.



Volumetric  
techniques

<http://www.metacd.com/r/m/ukhcnnyr/kZr7vQs>

## Thin layer chromatography (TLC)

In chromatography, components of a mixture are separated basis on their different interactions with a stationary surface and mobile phase.



TLC setup

## Developing and measuring TLC plates

Separated components in TLC can be observed and measured using different optical and chemical techniques.



Developing TLC  
plates

<http://www.metacd.com/r/m/ukhcnnyr/kdrZu0k>

## Paper chromatography

In chromatography, components of a mixture are separated basis on their different interactions with a stationary surface and mobile phase.



Paper  
chromatography

<http://www.metacd.com/r/m/ukhcnnyr/keYv0dK>

## Solvent reflux - setting up a reflux condenser

Refluxing is a commonly used procedure to carry out a reaction or process in boiling solvent without losing solvent as vapor

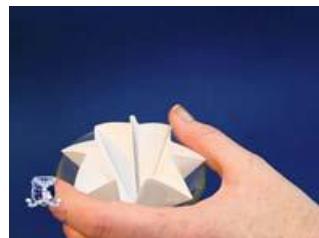


Reflux condensor

<http://www.metacd.com/r/m/ukhcnnyr/kbIQ0iK>

## Preparing a fluted filter paper

Filtration using a fluted paper maximizes the filtration area in a gravity filtration of small amounts of solid from a liquid.



Fluted filter  
paper

<http://www.metacd.com/r/m/ukhcnnyr/NBR7bu1>

## Simple gravity filtration

Gravity filtration is the basic technique to remove small amounts of solid impurities from a liquid.



<http://www.metacd.com/r/m/ukhcnnyr/LIX9EZk>

## Buchner vacuum filtration

Buchner filtration is widely used to collect a solid product from a reaction mixture.



Buchner vacuum  
filtration

## Hirsch vacuum filtration

Hirsch filtration is normally used to collect smaller quantities of solid product from a reaction mixture.

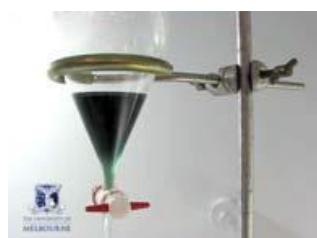


Hirsch vacuum  
filtration

<http://www.metacd.com/r/m/ukhcnnyr/kE3BIIe>

## Using a separating funnel – Menthone

Solvent extraction of menthone from its reaction products in water using a separating funnel



Separating funnel

<http://www.metacd.com/r/m/ukhcnnyr/kcPrOos>

## Using a separating funnel – Lomatiol

Lomatiol is extracted from ether into a basic aqueous solution, using a separating funnel



Separating funnel  
– lomatiol

<http://www.metcdn.com/r/m/ukhcnnyr/k6g38DS>

## Isolating Lomatiol using a Soxhlet extractor

In a Soxhlet extraction, condensed solvent vapour is recycled through a sample to continuously extract soluble components and concentrate them



Soxhlet extraction

<http://www.metcdn.com/r/m/ukhcnnyr/k61Dez9>

## Melting point determination using a DigiMelt

The melting point of a compound is a key property in helping identify it - as well as an indication of its purity.



MP determination

<http://www.metcdn.com/r/m/ukhcnnyr/kaYTQk9>

## Kinetics 1: Preparing the solutions

Good results in this experiment depend on good control of the concentrations, temperature and time for each measurement. This summarises how to do that.

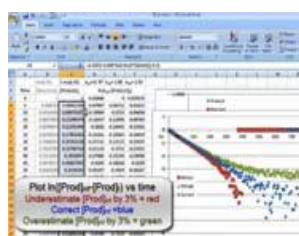


Kinetics –  
Preparing solutions

<http://www.metcdn.com/r/m/ukhcnnyr/EVpekms>

## Kinetics 2: How $[P]_{\text{inf}}$ is measured

Accurate analysis of the kinetic data requires a good estimate of the ‘infinite time’ conductance reading. This video explores that aspect of the experiment.



$K_{\text{inf}}$  impact

<http://www.metcdn.com/r/m/ukhcnnyr/EVpekyv>

# EXPERIMENT 1 – RESULTS SHEET

## ANALYTICAL TECHNIQUES AND METHODS

Name: \_\_\_\_\_

Day: \_\_\_\_\_ AM / Group No.: \_\_\_\_\_

Demonstrator: \_\_\_\_\_

### PART A: WEIGHING AND PIPETTING

Mass of conical flask and aliquot/s of water delivered by pipette

ENTRY	ITEM/S	MASS
1	Empty 100 mL conical flask	g
2	100 mL conical flask + 1 aliquot of water	g
3	100 mL conical flask + 2 aliquots of water	g
4	100 mL conical flask + 3 aliquots of water	g
5	100 mL conical flask + 4 aliquots of water	g
6	100 mL conical flask + 5 aliquots of water	g
7	100 mL conical flask + 6 aliquots of water	g

Mass delivered per aliquot

ALIQUOT 1	ALIQUOT 2	ALIQUOT 3
entry (2) – (1) =	entry (3) – (2) =	entry (4) – (3) =
ALIQUOT 4	ALIQUOT 5	ALIQUOT 6
entry (5) – (4) =	entry (6) – (5) =	entry (7) – (6) =

Mean mass delivered (per aliquot): \_\_\_\_\_

Laboratory temperature (°C): \_\_\_\_\_

Density of water at temperature of laboratory (g/mL): \_\_\_\_\_  
(obtained from Demonstrator)

Volume of water delivered per aliquot: \_\_\_\_\_ ± \_\_\_\_\_ (mean deviation)

*Space for calculations (Part A, add an additional page if necessary)*

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## PART B: DILUTING AND TITRATING

### Titration of hydrochloric acid with sodium hydroxide (using phenolphthalein indicator)

TITRE	INITIAL BURETTE READING (2 decimal places)	FINAL BURETTE READING (2 decimal places)	VOLUME ADDED (2 decimal places)	indicate (✓) the titre used for calculation
1				
2				
3				
4				
5				
6				

Set out carefully your treatment of errors in the following page 136.

Concentration of stock NaOH solution: \_\_\_\_\_ ± \_\_\_\_\_  
(obtained from Demonstrator)

Concentration of diluted NaOH solution: \_\_\_\_\_ ± \_\_\_\_\_

Mean volume of diluted NaOH solution (titre) required to reach endpoint:

\_\_\_\_\_ ± \_\_\_\_\_

Number of moles of NaOH required neutralizing an aliquot of HCl: \_\_\_\_\_

Concentration of hydrochloric acid: \_\_\_\_\_ ± \_\_\_\_\_

Space for calculations © School of Chemistry, The University of Melbourne, 2009

## Treatment of errors:

$$\text{Fractional error} = \frac{\text{error in quantity (e.g. uncertainty in pipette)}}{\text{quantity (e.g. pipette volume)}}$$

- (i) Errors calculation in concentration of diluted NaOH solution:

$$\text{Error} = (\Sigma \text{ fractional errors}) \times \text{final answer}$$

$$= \left( \frac{\text{error}}{\text{conc. NaOH stock sol}} + \frac{\text{error (Part A)}}{\text{pipette volume (Part A)}} + \frac{\text{error}}{\text{volumetric flask volume}} \right)$$

*x concentration of diluted NaOH solution*

=

- (ii) Error calculation in mean volume of diluted NaOH required to reach endpoint (titre):

$$\text{Error} = \text{mean deviation of } n \text{ measurements}$$

$$= \frac{\sum |(\text{volume of diluted NaOH used for each titration} - \text{mean titre})|}{n \text{ number of titrations}}$$

=

- (iii) Error calculation in concentration of hydrochloric acid:

$$\text{Error} = (\Sigma \text{ fractional errors}) \times \text{final answer}$$

$$= \left( \frac{\text{error}}{\text{conc. NaOH dilute sol}} + \frac{\text{error (Part A)}}{\text{pipette volume (Part A)}} + \frac{\text{error}}{\text{mean titre volume}} \right) \times$$

*concentration of HCl*

=

# EXPERIMENT 3 – RESULTS SHEET

## ISOLATION OF A NATURAL PRODUCT AND SOLUBILITIES OF ORGANIC COMPOUNDS

Name: \_\_\_\_\_ Day: \_\_\_\_\_ AM / PM Group No.: \_\_\_\_\_ Demonstrator: \_\_\_\_\_

PART C	COMPOUND	STRUCTURAL FORMULA	FUNCTIONAL GROUP	SOLVENT	OBSERVATION (e.g. soluble/insoluble)	INTERPRETATION <i>explain solubility behaviour of compound in relation to its properties and functional group (e.g. hydrophilic, hydrophobic, polar, non-polar, acidic, basic, molecular mass and chain length)</i>
1	Methanol			<ul style="list-style-type: none"> <li>• 1 mL Water</li> <li>• 1 mL 2 M HCl</li> <li>• 1 mL 2 M NaOH</li> <li>• 1 mL Heptane</li> <li>• Litmus Paper Test</li> </ul>		
2	2-Octanol			<ul style="list-style-type: none"> <li>• 1 mL Water</li> <li>• 1 mL 2 M HCl</li> <li>• 1 mL 2 M NaOH</li> <li>• 1 mL Heptane</li> <li>• Litmus Paper Test</li> </ul>		
3	1-Bromobutane			<ul style="list-style-type: none"> <li>• 1 mL Water</li> <li>• 1 mL 2 M HCl</li> <li>• 1 mL 2 M NaOH</li> <li>• 1 mL Heptane</li> <li>• Litmus Paper Test</li> </ul>		

PART C	COMPOUND	STRUCTURAL FORMULA	FUNCTIONAL GROUP	SOLVENT	OBSERVATION (e.g. soluble/insoluble)	INTERPRETATION <i>explain solubility behaviour of compound in relation to its properties and functional group (e.g. hydrophilic, hydrophobic, polar, non-polar, acidic, basic, molecular mass and chain length)</i>
4	Benzoic acid			<ul style="list-style-type: none"> <li>• 1 mL Water</li> <li>• 1 mL 2 M HCl</li> <li>• 1 mL 2 M NaOH</li> <li>• 1 mL Heptane</li> <li>• Litmus Paper Test</li> </ul>	<small>© School of Chemistry, The University of Melbourne, 2018</small>	
5	Propylamine			<ul style="list-style-type: none"> <li>• 1 mL Water</li> <li>• 1 mL 2 M HCl</li> <li>• 1 mL 2 M NaOH</li> <li>• 1 mL Heptane</li> <li>• Litmus Paper Test</li> </ul>	<small>© School of Chemistry, The University of Melbourne, 2018</small>	

Space for discussion and conclusion:

## EXPERIMENT 4 – RESULTS SHEET

# VAPOUR PRESSURE OF A VOLATILE LIQUID

Name: \_\_\_\_\_

Day: \_\_\_\_\_ AM / PM      Group No.: \_\_\_\_\_

Demonstrator: \_\_\_\_\_

## PART A: MANOMETER PRESSURE MEASUREMENT

Manometer pressure reading: Before \_\_\_\_\_ After \_\_\_\_\_

## PART B: VAPOUR PRESSURE MEASUREMENTS

**Liquid Sample: Name** \_\_\_\_\_ **Formula** \_\_\_\_\_

## PART C: TREATMENT OF DATA

**Experimental data sets measured at different T (°C):**

TEMP (°C)	EQUILIBRIUM VAPOUR PRESSURE ( $p$ mbar)	TEMP (K)

**Data for graph:**

$1/T$ (in K <sup>-1</sup> )	$\ln p$

Plot  $1/T$  (x-axis) vs.  $\ln p$  (y-axis) on your graph paper (make sure the graph occupies most of the page).

Gradient of line of best fit (include units): \_\_\_\_\_

The mathematical relationship between the gradient,  $\Delta H$  and R is: \_\_\_\_\_

The value of R (include units): \_\_\_\_\_

The value of  $\Delta H$  (include units): \_\_\_\_\_

## PART D: CALCULATIONS

Summary:

Volatile liquid: \_\_\_\_\_

1. Estimated normal boiling point of the volatile liquid: \_\_\_\_\_
2. Estimated vapour pressure of the volatile liquid at  $-116\text{ }^{\circ}\text{C}$ : \_\_\_\_\_
3. Energy required to vaporise 27.5 g of the volatile liquid: \_\_\_\_\_
4. Volume of vapour generated at 1 atmosphere and  $40\text{ }^{\circ}\text{C}$  by the evaporation of 27.5 g of volatile liquid (from (3)): \_\_\_\_\_

*Space for calculations:*

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*Space for calculations:*

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# EXPERIMENT 5 – RESULTS SHEET

# GLASS ELECTRODE: BUFFER SOLUTIONS

Name: \_\_\_\_\_

Day: \_\_\_\_\_ AM / PM      Group No.: \_\_\_\_\_

Demonstrator: \_\_\_\_\_

## PART A: TITRATION OF A WEAK BASE WITH A STRONG ACID: ACTION OF INDICATOR

**Initial burette reading:** \_\_\_\_\_ mL

## PART B: BUFFER SOLUTIONS CONTAINING CARBONATE AND BICARBONATE IONS

### RESULTS

SOLUTION in BEAKER	pH	ADDITION of 0.1 M Na <sub>2</sub> CO <sub>3</sub> SOLUTION	ADDITION of 0.1 M HCl	ADDITION of 0.1 M NaOH
40 mL 0.1 M NaHCO <sub>3</sub>	—	Q7 pH =	—	—
40 mL Na <sub>2</sub> CO <sub>3</sub> /NaHCO <sub>3</sub> buffer solution	pH =	—	pH =	pH =
40 mL water	pH =	—	pH =	pH =

### ANSWER TO QUESTIONS

Q8	
Q9	Q10
Q11	Q12
Q13	Q14
Q15	Q16
Q17	

# EXPERIMENT 6 – RESULTS SHEET

## SYNTHESIS AND ANALYSIS OF A POLYIODIDE SALT

Name: \_\_\_\_\_

Day: \_\_\_\_\_ AM / PM      Group No.: \_\_\_\_\_

Demonstrator: \_\_\_\_\_

### PREPARATION OF POLYIODIDE

Mass of tetramethylammonium iodide used: \_\_\_\_\_

Mass of iodine used: \_\_\_\_\_

Yield of polyiodide (mass of dry solid collected): \_\_\_\_\_

### ANALYSIS

Preparation of an analytical solution: by dissolving a known amount of polyiodide salt in a solvent mixture.

	OBJECT	MASS
1	Mass of beaker and sample (~0.2g)	
2	Mass of beaker after sample has been transferred	
3	Mass of polyiodide added to volumetric flask (1 – 2)	

Concentration of sodium thiosulphate solution: \_\_\_\_\_  
(Obtained from Demonstrator)

Volume delivered in one aliquot of polyiodide solution: \_\_\_\_\_

Titration of polyiodide solution with sodium thiosulphate solution

TITRE	INITIAL BURETTE READING (2 decimal places)	FINAL BURETTE READING (2 decimal places)	VOLUME ADDED (2 decimal places)
1			
2			
3			
4			
5			

The titres which are used in the calculations are: \_\_\_\_\_

---

Average volume of sodium thiosulphate required to reach endpoint: \_\_\_\_\_  $\pm$  \_\_\_\_\_

Number of moles of thiosulphate required to reach endpoint: \_\_\_\_\_

Number of moles of iodine ( $I_2$ ) present in one 5 mL aliquot of solution: \_\_\_\_\_

Number of moles of iodine ( $I_2$ ) present in original 50 mL analytical sample: \_\_\_\_\_

Mass of iodine ( $I_2$ ) present in original analytical sample: \_\_\_\_\_

Percentage of iodine ( $I_2$ ) by mass in your polyiodide: \_\_\_\_\_

Value of "x" in the formula  $N(CH_3)_4I(I_2)_x$ : \_\_\_\_\_

What is the formula of your salt? \_\_\_\_\_

Space for calculations (if not sufficient, use an extra sheet)

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## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 1: Analytical Techniques and Methods</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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Date:.....

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**EXPERIMENT 1: ANALYTICAL TECHNIQUES AND METHODS**

	COMMENTS	MARK
Cal Prelab Module		/2
Report		/1
Part A: <b>Calibrating a Pipette</b> Volume of pipette      3 marks Calculations            2 marks Calculation of Mean Deviation        3 marks		/8
Part B: <b>Volumetric Analysis</b> Concordant titres     3 marks Calculations           3 marks Errors calculation    3 marks		/9
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 2: The Preparation of Paracetamol</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 2: THE PREPARATION OF PARACETAMOL**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Report</b> Correct style of writing Presentation		/4
<b>Product Appearance</b>	Appearance: <i>wet / dry</i>  <i>good colour / poor colour</i>  <i>crystalline / powder</i>	/3
<b>Product Yield</b> Yield 5 marks Calculation of %yield 2 marks	Yield: <i>low / medium / high</i>	/7
<b>Melting Point</b>		/2
<b>Ferric(III) Chloride Test</b>		/2
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	/20

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## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 3: Isolation of a Natural Product – II</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 3: ISOLATION OF A NATURAL PRODUCT – II**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Part A:</b> <b>Isolation of Lomatiol</b> 4 marks Product appearance Product description  Calculation of % yield      1 mark	Appearance: <i>wet / dry</i>  <i>dirty / clean</i>  Yield: <i>good yield / poor yield</i>	
<b>Part B:</b> Hydrosulphite reduction      1 mark		/6
<b>Part C:</b> <b>Organic Compounds</b> 10 marks		/10
<b>Report</b> Correct style of writing      2 marks		/2
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 4: Vapour Pressure of a Volatile Liquid</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 4: VAPOUR PRESSURE OF n-PENTANE**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Part B: Vapour Pressure</b> Data Table                    2 marks Discussions 1 to 2            2 marks		/4
<b>Part C: Treatment of Data</b> Graph                        5 marks Calculations of $\Delta H_{vap}$ 4 marks		/9
<b>Part D: Calculations</b> Questions 1 to 4            1 mark each		/4
<b>Report</b> Presentation		/1
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 5: Glass Electrode: Buffer Solutions</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 5: GLASS ELECTRODE: BUFFER SOLUTIONS**

	<b>COMMENTS</b>	<b>MARK</b>
<b>Cal Prelab Module</b>		<b>/2</b>
<b>Report</b>		<b>/1</b>
<b>Part A: Acid /Base Titration</b> Titration graph                    2 marks Titres                              2 marks Questions                         6 marks		<b>/10</b>
<b>Part B: Buffer Solutions</b> Observations Explanations Questions		<b>/7</b>
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	Experiment 6: A Polyiodide Salt: Synthesis and Analysis
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 6: A POLYIODIDE SALT: SYNTHESIS AND ANALYSIS**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Part A</b> <b>Preparation of Salt:</b> Product Appearance      3 marks  Product Yield            3 marks	Appearance: <i>wet / dry</i>  <i>crystalline / powder / non homogeneous</i>  Yield: <i>low / medium / high</i>	/6
<b>Part B</b> <b>Analysis of Salt:</b> Concordant titres      4 marks Calculations of average titre and MD      3 marks Calculation of %I <sub>2</sub> 3 marks Salt formula            1 mark		/11
<b>Report</b> Presentation Correct units/significant figures		/1
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	/20

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<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 7: The Reduction of Benzoin</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 7: THE REDUCTION OF BENZOIN**

	COMMENTS	MARK
Cal Prelab Module		/2
Report Correct style of writing		/4
Product Appearance	Appearance: <i>wet / dry</i> <i>good colour / poor colour</i> <i>crystalline / powder</i>	/2
Product Yield Calculation of theoretical yield Calculation of % yield	Yield: <i>low / medium / high</i>	/5
Melting Point		/2
Thin Layer Chromatography Calculations of R <sub>f</sub> values Comments on TLC plate		/5
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 8: The Oxidation of Menthol</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 8: THE OXIDATION OF MENTHOL**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Report</b> Presentation Answers of questions		/5
<b>Product Appearance</b>	Appearance: <i>wet</i> / <i>dry</i>  <i>poor colour</i> / <i>good colour</i>	/4
<b>Product Yield</b> Calculation of theoretical yield Calculation of % yield	Yield: <i>low</i> / <i>medium</i> / <i>high</i>	/6
<b>Melting Point</b>		/3
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 9: Rates of Reaction: The Hydrolysis of <i>t</i>-Butyl Chloride</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 9: RATES OF REACTION: THE HYDROLYSIS OF t-BUTYLCHLORIDE**

	COMMENTS	MARK
<b>Cal Prelab Module</b>		/2
<b>Report</b> Presentation Discussions 1 and 2		/4
<b>Results</b> Table of results      1 mark Graphs                  4 marks Calculations of <i>k</i> 's    5 marks		/10
<b>Questions</b> Q1                        3 marks Q2                        1 mark		/4
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

# REPORT COVER SHEET AND FEEDBACK SHEET

School of Chemistry

The University of Melbourne

**PLEASE COMPLETE AND ATTACH AS THE FRONT PAGE OF YOUR REPORT**

## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 10: Practical Evidence of the “Particle in the Box” Concept</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 10: PRACTICAL EVIDENCE OF THE PARTICLE IN THE BOX CONCEPT**

	<b>COMMENTS</b>	<b>MARK</b>
<b>Cal Prelab Module</b>		<b>/2</b>
<b>Graphs:</b> Absorption Spectra      2 marks Extinction Coefficient      2 marks Concentration Effect      2 marks		<b>/6</b>
<b>Data Analysis:</b> Step (2) to Step (6)      each 1 mark Step (7)      2 marks		<b>/7</b>
<b>Questions:</b> Question 1 to Question 9		<b>/5</b>
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 11: Electrochemistry: EMF Measurements</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 11: ELECTROCHEMISTRY: EMF MEASUREMENTS**

	<b>COMMENTS</b>	<b>MARK</b>
<b>Cal Prelab Module</b>		<b>/2</b>
<b>Part A: Half-Cell Potential</b>  Step (1) 1 mark Step (2) 5 marks Steps (3) and (4) 4 marks		<b>/10</b>
<b>Part B: <math>I_3^-/I^-</math> Half -Cell</b>  Graph (Set 1) 2 marks  Graph (Set 2) 2 marks		<b>/4</b>
<b>Ratio of Gradient Calculation</b>		<b>/2</b>
<b>Report</b>  Presentation Nernst equation verification		<b>/2</b>
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

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<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 12: Synthesis of Hexaamminecobalt(III) Chloride</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 12: SYNTHESIS OF HEXAAMMINECOBALT(III) CHLORIDE**

	COMMENTS	MARK
Cal Prelab Module		/2
Report Presentation Correct style of writing		/1
Part A: <b>Product Synthesis</b> Product appearance      2 marks Product yield      5 marks Correct calculations      2 marks	Appearance: <i>wet and dirty / dry and clean</i> <i>poor colour / good colour</i> <i>crystalline / powder</i>  Yield: <i>low / medium / high</i>	/9
Part B: <b>Test Tube Reactions</b> Reaction of $[Co(NH_3)_6]Cl_3$ 2 marks Reactions of $[Co(OH_2)_6]^{2+}$ 6 marks		/8
<b>TOTAL</b>	Demonstrator Signature:	/20

# REPORT COVER SHEET AND FEEDBACK SHEET

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## Laboratory Report Cover Sheet

<b>Student Name:</b>	
<b>Student Number:</b>	
<b>Subject Name &amp; Code:</b>	
<b>Demonstrator:</b>	
<b>Experiment Title:</b>	<b>Experiment 13: Spectroscopy: Determination of the pK<sub>a</sub> of an Acid–Base Indicator</b>
<b>In cases where group work is involved, give details of your group here:</b>	

Due Date: \_\_\_\_\_

Special Consideration application submitted: YES/NO

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**EXPERIMENT 13: SPECTROSCOPY: DETERMINATION OF  $pK_a$  OF AN ACID–BASE INDICATOR**

	<b>COMMENTS</b>	<b>MARK</b>
<b>Cal Prelab Module</b>		<b>/2</b>
<b>Result</b> <b>Part A:</b> Spectrum of indicator      4 marks		
<b>Part B:</b> Graph (Beer-Lambert Law) 4 marks Calculation of $\epsilon$ 4 marks		
<b>Part C:</b> Calculations of $K_a$ and $pK_a$ 2 marks		<b>/14</b>
<b>Report</b> Questions      3 marks Presentation and Graphing techniques      1 mark		<b>/4</b>
<b>TOTAL</b>	<b>Demonstrator Signature:</b>	<b>/20</b>

## CHEMICAL PRELAB RECEIPT – CHEM1000X, X = 3, 4, 6 or 9

Name: \_\_\_\_\_

Student Number: \_\_\_\_\_

Day: M / T / W / T / F Session: AM / PM / EVE

Group Number: \_\_\_\_\_

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