

CHM1011

CHEMISTRY I

Laboratory Manual

CLAYTON CAMPUS
SEMESTER 1, 2021

monash.edu/science

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(i) Introduction

Welcome to the CHM1011 laboratory experience. Chemistry is fundamentally an experimental science, and it is the joy of designing and testing ideas with experiments that both explore and impact on the world around us that makes chemistry so fascinating and innovative. This practical course will introduce you to some of the most important laboratory skills and techniques used in chemistry, skills that should be useful to you even if chemistry doesn't become your major. As practicing chemists, not only will this laboratory course expose you to practical techniques, you will also see how some of the theoretical principles of chemistry were uncovered, and how chemistry is applied on a day-to-day basis all over the world. Above all, we hope that you will enjoy the course, make new friends, and acquire some of the interest and fascination for chemistry that your lecturers and teaching associates (TAs) have.

Dr. Sara Kyne (First Year Coordinator, 2021)

Dr. Toby Bell (Unit Coordinator, 2021)

(ii) CHM1011 Laboratory Timetable 2021

On-campus (dual mode) delivery¹

Groups		A / C	B / D	Online report due ²
Week	Date ³	Laboratory exercise		
1	1/3	Ex 1 - Geometric Models: VSEPR Method (dry laboratory – online)		7 days (one week)
2	8/3	Ex 2 - The Chemistry Circuit		7 days (one week)
3	15/3		Ex 2 - The Chemistry Circuit	7 days (one week)
4	22/3	Ex 3 - Transformations of Copper		7 days (one week)
5	29/3		Ex 3 - Transformations of Copper	7 days (one week)
	5/4	Mid-semester break		
6	12/4	Ex 4 - Calorimetric Analysis of Baking Powder		7 days (one week)
7	19/4		Ex 4 - Calorimetric Analysis of Baking Powder	7 days (one week)
8	26/4	Ex 5 - Acid-base Titrations		7 days (one week)
9	3/5		Ex 5 - Acid-base Titrations	7 days (one week)
10	10/5	Ex 6 - Timing the Iodine Clock		7 days (one week)
11	17/5		Ex 6 - Timing the Iodine Clock	7 days (one week)
12	24/5			

¹ All dry laboratory exercises are held online via Zoom, and wet laboratory exercises are held on-campus.

² Complete your laboratory report online, via Moodle. Your online report must be submitted within seven days (one week) of the completion of your class. Laboratory reports are due at the start time of your timetabled laboratory session.

³ Week commencing. Check your timetable to confirm the date of your laboratory sessions.

Online only delivery¹

Group		C	Online report due²
Week	Date³	Laboratory exercise	
1	1/3	Ex 1 - Geometric Models: VSEPR Method	7 days (one week)
2	8/3	Ex 2 - Quantitative Analysis of Vitamin C in Ribena® & Fruit Juice	7 days (one week)
3	15/3		
4	22/3	Ex 3 - Transformations of Copper	7 days (one week)
5	29/3		
	5/4	Mid-semester break	
6	12/4	Ex 4 - Calorimetric Analysis of Baking Powder	7 days (one week)
7	19/4		
8	26/4	Ex 5 - Acid-base Titrations	7 days (one week)
9	3/5		
10	10/5	Ex 6 - Timing the Iodine Clock	7 days (one week)
11	17/5		
12	24/5		

¹ All laboratory exercises (dry and wet) are held online via Zoom.

² Complete your laboratory report online, via Moodle. Your online report must be submitted within seven days (one week) of the completion of your class. Laboratory reports are due at the start time of your timetabled laboratory session.

³ Week commencing. Check your timetable to confirm the date of your laboratory sessions.

(iii) Aims of the Laboratory Course

The laboratory course has four main aims:

- *Understanding scientific method.*

Scientific method is important across all the sciences. It develops critical thinking and analysis. You will be shown the basis of scientific inquiry, how to pose scientific questions, find evidence and formulate answers.

- *Illustrating theoretical principles.*

Laboratory work serves to illustrate many of the theoretical principles covered in the unit content and demonstrates practical applications.

- *Developing experimental skills in the practice of chemistry.*

There are many skills involved in handling laboratory instruments and equipment, in carrying out chemical transformations, and in making chemical measurements.

- *Enhancing teamwork, time management skills and broader communication skills – both to peers and the general public.*

These skills are important for you to be able to use and make the most of the scientific skills and knowledge that you accrue doing your academic study.

(iv) Structure of the Laboratory Course

To prepare for each laboratory session, read the exercise in the laboratory manual and perform the pre-laboratory activities (see section (ix) for more details).

The class is divided into groups, and during any laboratory session each group may be carrying out a different exercise. Carefully check which exercise you will be doing prior to your session.

(v) Icons Used in this Manual

	Work on your own		Good scientific practice		Something to think about
	Work in a pair		Use the Standard Operating Procedure		Hazard exists
	Work in a group of three		Hot. Take care		Corrosive
	Work in a group of four		Harmful		Clean and put away all glassware used

(vi) General Information**Materials Required**

During your laboratory session, you will have access to all glassware, equipment and chemicals that you will need. You will need the following, otherwise you will be unable to complete the laboratory session:

- Laboratory notebook (stapled A4), ruler, calculator and pen
- Laboratory coat
- Laboratory safety glasses (students who wear contact lenses should purchase specific wrap-around safety goggles)
- Fully enclosed shoes with firm soles
- Face mask (for use in the laboratory only)

Note: You will be asked to leave the laboratory if you wear open-toe or other inappropriate footwear (including thongs, sandals, ballet shoes and slippers) to your laboratory session.

GSP**(vii) Laboratory Hours and Contact Details**

Laboratory classes	Tuesday to Friday mornings 10:00 AM – 12:30 PM	Doors close 10:15 AM
	Monday to Friday afternoons 2:00 PM – 4:30 PM	Doors close 2:15 PM

The enquiries counter is open	10:45 AM – 12:00 noon
	2:45 PM – 4:30 PM

The contact e-mail for First Year Chemistry is sci-chem-firstyear-enquiries@monash.edu.

The contact number for the First Year Laboratories is 990 59625.

(viii) Laboratory Rules**Attendance**

The laboratory component is a hurdle requirement for this unit. If you arrive late and miss the safety briefing, you will not be permitted to commence the class. The pre-laboratory exercise must be completed and passed in order to undertake the laboratory exercise.

It is your responsibility to ensure that the workspaces used are thoroughly cleaned and dried, and that all equipment is put away before you leave.

Note: The School of Chemistry takes your well-being and safety, and that of everyone in the laboratory, very seriously. As such, you can be asked to leave the laboratory for the following reasons (see Safety Rules and Safety Precautions in the Student Handbook):

- Aggressive, offensive, or intimidating behaviour
- Behaviour which is deemed to be hazardous to yourself and/or class mates
- Wearing inappropriate footwear
- Not wearing appropriate health and safety personal protective equipment
- Using mobile phones in the laboratory

GSP**GSP**

You may be allowed to continue the laboratory course but only after consultation with the First Year/Unit Coordinator.

Absence

If you are absent from any laboratory session due to exceptional circumstances beyond your control, you may apply for special consideration. For additional details please refer to the School of Chemistry Undergraduate Handbook available on Moodle.

If you have serious difficulties of any kind throughout the year, we recommend that you notify the Faculty of Science Student Services and contact the First Year Coordinator or Unit Coordinator.

(ix) Pre-Laboratory Activities and Laboratory Reports

In this unit, your pre-laboratory activities must be completed online, via Moodle, 30 minutes prior to the beginning of the laboratory session. The pre-laboratory activities must be passed in order to be able to complete the laboratory exercise. Please note that no exemptions are given for the pre-laboratory activities.

Laboratory reports will be completed on Moodle, as online laboratory reports; these may also include your laboratory notebook pages, and things like graphs or instrument printouts.

Your laboratory reports must be completed on Moodle by the due date (found on Moodle).

For all exercises, the report is due one week (seven days) following the completion of the exercise. These include weekends, university holidays, and public holidays. Reports are due at the start time of your normal laboratory session (10:00 AM for morning sessions and 2:00 PM for afternoon sessions). Note that for submissions made after this time, a late penalty will be applied (10% per day as determined by University Policy). No late submissions can be accepted after seven days from the original due date.

GSP

Please note that all results must be written in pen (not pencil), as per industry standards. This standard protects you by showing your work is original and has not been altered. It also ensures that your documents will scan properly for Moodle. Laboratory notes written in pencil will not be accepted. Graphs can be plotted in pencil.

(x) Assessment

The laboratory assessment component constitutes 30% of the total mark for CHM1011.

The laboratory component is a **hurdle requirement** to pass the unit (overall 45%).

Your assessment will be based on:

- Your pre-laboratory activities
- The way in which you work in the laboratory (participation, experimental technique) and the results that you achieve (where relevant)
- Your observations, analysis, and online reports

Each laboratory report is marked out of **45**. Each pre-laboratory activity is marked out of **5** and passing this is required to complete that practical exercise. Marking schemes are given on Moodle for each exercise. You can check your laboratory marks on Moodle.

Laboratory Notebook: All experimental records and observations must be recorded in a laboratory notebook (bound, stapled, A4 size) in pen.

A typical entry (see example to the right) should include:

- Name, student ID (and partner/group members if applicable)
- Experiment title
- Dates
- Method recorded in brief, including any changes and/or details of any method that you designed
- Experimental results, data and observations
- Important notes
- TA signature (validated with date stamp)

Name: Jane Doe ID: 98765432 Partner: John Smith Date: 05.03.20

Experiment X: Analysis of Soil Samples via UV-Visible Spectrophotometry

Values from Pre-lab Quiz: Concentrations in the soil samples were calculated to be:

Copper: between 50 ppm and 210 ppm; Cobalt: between 25 ppm and 70 ppm.

Method: As per CHM1011 2020 Laboratory Manual, Exercise X, p. 92. Calibration curves (Cu^{2+} , Co^{3+}) will be prepared using 4 standard solutions and a blank. Soil samples (10 g) will be extracted into water, filtered, made up to 25 mL, and analysed via UV-Vis.

Changes to method: Unknown soil sample 3 required a longer mixing time than the 10 minutes stated in the method; time was increased to 20 mins.

Once prepared, sample 2 required a 1/5 dilution, as the Abs. was over 1.

Observations:

Sample 1: Solution was pale purple in colour; possible mix of Cu and Co?

Sample 2: Solution was bright blue in colour, suggesting a high [Cu]

Sample 3: Solution was a light blue colour, suggesting the presence of Cu

Data:

Copper analysis at 810 nm

Solution	Concentration (ppm)	Absorbance
Blank	0.00	0.000
Standard 1	40.0	0.200
Standard 2	80.0	0.400
Standard 3	120.0	0.600
Standard 4	160.0	0.800
Sample 1	~~~~~	0.700
Sample 2	~~~~~	1.250
Sample 3	~~~~~	0.350
Sample 2 (diluted 1/5)	~~~~~	0.250

MRC

The typical mark breakdown for your laboratory notes may be:

Name and student ID (& partner/group members if applicable).	1	All results, data and observations recorded.	1
Experiment title and date recorded.	1	Punctuality to class.	1
Notes written in pen.	1	Good attitude and participation.	1
Neat and legible entry.	1	Good housekeeping (all glassware, reagents and apparatus cleaned up).	1
Method recorded in brief, including changes to experimental variables.	1	Laboratory notes signed by TA (validated with date stamp).	1

(xii) Useful Equations and Information for Laboratory Work**Equations**

- To calculate mass or moles: $m = n \times M$

where: m = mass in grams (g)

n = number of moles (mol)

M = molecular mass in g mol^{-1}

- To calculate moles, concentration or volume: $n = c \times V$

where: n = number of moles (mol)

c = concentration in M (molarity, mol L^{-1} or mol dm^{-3})

V = volume in litres (L or dm^3)

- To calculate concentration or volume when performing a dilution: $c_1 \times V_1 = c_2 \times V_2$

where: c_1 = concentration of initial solution V_1 = volume of initial solution

c_2 = concentration of final solution V_2 = volume of final solution

Note: Units for concentration and volume should be the same on both sides of the equation

Conversions**Scientific Notation:**

$100 = 1 \times 10^2$	$1000 = 1 \times 10^3$	$10000 = 1 \times 10^4$	etc.
$0.1 = 1 \times 10^{-1}$	$0.01 = 1 \times 10^{-2}$	$0.001 = 1 \times 10^{-3}$	etc.

Moles:

1 mole (mol) = 1000 millimoles (mmol)

Volume:

1 litre (L) = 1000 millilitres (mL)

1 millilitre (mL) = 1000 microlitres (μL)

Mass:

1 gram (g) = 1000 milligrams (mg)

1 milligram (mg) = 1000 micrograms (μg)

Units of Concentration:

M = molarity = $\text{mol/L} = \text{mol L}^{-1} = \text{mol dm}^{-3}$

ppm = parts per million = mg L^{-1} ; $\mu\text{g mL}^{-1}$; $\mu\text{L L}^{-1}$; $\mu\text{g g}^{-1} = \text{mg/L}$; $\mu\text{g/mL}$; $\mu\text{L/L}$; $\mu\text{g/g}$

Advice:

Refer to the Workbook Week 0 on Moodle.

Always include appropriate units in your workings and your final answer.

Give your final answer to the correct number of significant figures. See the following examples and Moodle if you need further advice.

Moles and Masses Calculation Examples

1. How many moles of sulfuric acid (H₂SO₄) are there in 250 mL of a 0.200 M solution?

A. $n = c \times V$
 $= 0.200 \text{ mol L}^{-1} \times 0.250 \text{ L}$
 $= 0.0500 \text{ mol}$
 $= 5.00 \times 10^{-2} \text{ mol}$

2. What is the molecular mass of H₂SO₄?

A. $M(\text{H}_2\text{SO}_4) = 2 \times M(\text{H}) + M(\text{S}) + 4 \times M(\text{O})$
 $= 2 \times 1.008 + 32.066 + 4 \times 15.999$
 $= 98.078 \text{ g mol}^{-1}$

3. What is the mass (in grams) of H₂SO₄ in Q1 above?

A. $m = n \times M$
 $m = 5.00 \times 10^{-2} \text{ mol} \times 98.077 \text{ g mol}^{-1}$
 $= 4.90 \text{ g}$ (3 significant figures, consistent with the data given)

4. 50.0 mL of a hydrochloric acid (HCl) solution reacts exactly with 20.5 mL of 0.212 M sodium hydroxide (NaOH) solution. Calculate the molarity (M) of the hydrochloric acid solution.

A. We need to determine the amount (in mole) of HCl per litre of solution.

We know the reaction is $\text{NaOH (aq)} + \text{HCl(aq)} \rightarrow \text{NaCl (aq)} + \text{H}_2\text{O (l)}$

Molar ratio = 1 : 1

Based on the information provided, 50 mL of hydrochloric acid solution contains the same number of moles of HCl as 20.5 mL of 0.212 M sodium hydroxide solution contains moles of NaOH

$$n = c \times V$$

$$n_{\text{NaOH}} = 0.212 \text{ mol L}^{-1} \times 0.0205 \text{ L}$$

$$= 0.004346 \text{ mol}$$

$$= 4.35 \times 10^{-3} \text{ mol}$$

$$= n_{\text{HCl}}$$

Therefore, the concentration of HCl is:

$$c_{\text{HCl}} = n_{\text{HCl}} / V$$

$$= 4.346 \times 10^{-3} \text{ mol} / 0.0500 \text{ L}$$

$$= 0.08692 \text{ mol L}^{-1}$$

$$= 8.69 \times 10^{-2} \text{ M (mol L}^{-1}\text{, 3 significant figures)}$$

Exercise 1: Geometric Models - Valence-Shell Electron-Pair Repulsion (VSEPR) Method

Learning Outcomes:



1. Construct models to aid visualisation of chemical structures.
2. Manipulate common diagram types and their conventions to represent a range of organic and inorganic molecules.
3. Visualise molecules in three dimensions to enhance understanding of shapes and stereochemistry.
4. Generate molecular geometries, based on bond numbers and angles.

Note: This laboratory exercise is an online dry laboratory.

This exercise covers examinable material that will not be covered explicitly elsewhere in the unit.

Background: *Chemistry: Atoms First* (2019), Chapter 4; Blackman *et al.*, *Chemistry* (2019), Chapter 5.

1. Introduction

Lewis structures are schematic drawings that show how the atoms in a molecule are connected. Unpaired electrons are shared with neighbouring atoms to form single, double or triple bonds. However, although Lewis structures can show the valence electron distribution, they do not accurately depict three-dimensional structures.

Historically, a number of models have been proposed to describe molecular shapes and structure. One of these is the Valence-Shell Electron-Pair Repulsion (VSEPR) model. The basis of the VSEPR model is “each group of valence electrons around a central atom is located as far away from the others as possible in order to minimise repulsions”.

Consider the two systems in Figure 1.1. The figure on the left represents the centre atom in a small molecule with two regions of negative charge (electron domains) surrounding it. VSEPR theory predicts these two regions of negative charge will adopt a position that maximises the distance between them. The figure on the right represents a similar case with three electron domains. These configurations determine the geometry of the molecule.

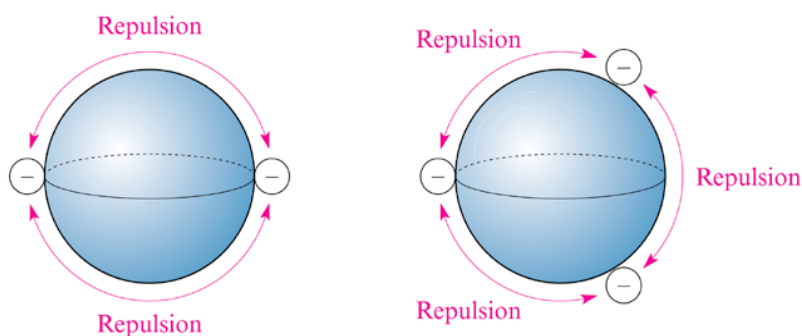
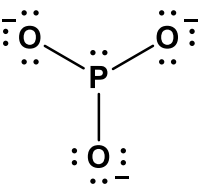
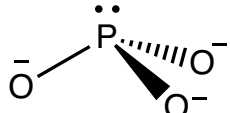


Figure 1.1. VSEPR models with two and three electron domains about a spherical centre.

2. Exercise

You will predict and analyse the three dimensional shapes adopted by different small molecules. Your task is to construct a number of different molecules and complete a description of their structural properties. An example of the table needed to complete this exercise in your laboratory notebook is shown in Table 1.1 using the phosphite ion (PO_3^{3-}).

Table 1.1: Phosphite ion structural properties

Molecule: Phosphite ion, PO₃³⁻	2.1. Lewis structures (including resonance forms) 	Formal charge (FC) on each atom: $FC_O = 6 - (6 + 2/2) = -1$ $FC_P = 5 - (2 + 6/2) = 0$
No. of valence electrons: 1x5 + 3x6 + 3 = 26		
2.2. Number of atoms bonded to central atom 3	2.3. Number of non-bonding electron pairs on the central atom 1	∴ Number of electron domains 3 + 1 = 4
2.4. Parent geometry TETRAHEDRAL	2.5. Molecular geometry with ideal bond angles TRIGONAL PYRAMIDAL	2.6. Perspective drawing 

Multimedia simulations can be used to help visualise the three dimensional (3D) structure of the molecule or ion of interest. To do this, access the 3D modelling simulation here:

https://phet.colorado.edu/sims/html/molecule-shapes/latest/molecule-shapes_en.html

On the landing page of the simulation, choose "Model". This section allows you to see the 3D molecular shape of a molecule and the expected bond angles according to a prediction made based on the number of bonding pairs and non-bonding (lone) pairs. Use the 3D modelling simulation tool to assist you completing this exercise.

You may also want to construct physical models yourself using for example, toothpicks and plasticine/putty.

2.1. Lewis Structures (*Chemistry: Atoms First* (2019), Chapter 4.4; Blackman *et al.*, *Chemistry* (2019), Chapter 5.3)

Follow the five steps below to construct a Lewis structure for a molecule or ion.

- 1) Determine the total number of valence electrons in the molecule or ion.
- 2) Arrange the atoms to show how they are connected. All of the atoms are grouped around a central atom, which is usually the least electronegative in the molecule or ion. Place a bonding pair of electrons between each pair of adjacent atoms to give a single bond.
- 3) Begin with the terminal atoms and add enough electrons to each atom to give all of the terminal atoms an octet (except for hydrogen, consider why this is).
- 4) Place any electrons left over on the central atom.
- 5) Minimise the formal charges. If the central atom has fewer electrons than an octet, use lone pairs from terminal atoms to form multiple bonds to the central atom in order to achieve an octet (Note there are exceptions to this rule; e.g. Be and B).

The formal charge for each atom:

- Depends on the difference between the number of valence electrons in the free atom and the number assigned to it in the Lewis structure;
- Is a way of computing the charge distribution within a Lewis structure (the sum of the formal charges on the atoms within a molecule or ion must equal the overall charge);
- Does not represent a true charge on an atom, it is used to predict the most likely structure when a compound has more than one possible Lewis structure.

$$\text{Formal Charge (FC)} = \# \text{ Valence Electrons} - (\# \text{ Lone Pair Electrons} + \frac{1}{2} \# \text{ Bonding Electrons})$$

Using the phosphite ion as an example:

- a) Total number of valence electrons is $(1 \times 5) + (3 \times 6) + 3 = 26$.

(i.e. # of P and O valence electrons, plus the negative charge.)

- b) Position the 3 oxygen atoms symmetrically around the phosphorus atom.

Three sigma bonds (representing 2 electrons each) are introduced to connect the atoms.

(Note: We still need to incorporate $26 - 6 = 20$ electrons into the structure.)

- c) Introduce lone pairs to the terminal atoms (oxygen) so that each one possesses an octet of electrons.

(Note: We still need to incorporate $20 - 18 = 2$ electrons into the structure.)

- d) Place the two remaining electrons on the centre atom (phosphorus).

- e) Calculate the formal charge (FC) on each atom, and minimise those formal charges where possible². The sum of the formal charges should equal the overall charge on the molecule.

To calculate the FC for oxygen and phosphorous in the phosphite ion:

$$\text{FC}_\text{O} = \# \text{ O valence electrons} - (\# \text{ lone pair electrons on the O atom} + \frac{1}{2} \# \text{ bonding electrons})$$

$$= 6 - (6 + 2/2) = -1$$

$$\text{FC}_\text{P} = 5 - (2 + 6/2) = 0$$

$$\text{Sum of FC} = (-1 \times 3) + 0 = -3 = \text{charge on the phosphite ion}$$

In this example, the oxygen atoms are identical, and the formal charge on each oxygen atom is the same. Note that sometimes atoms in a molecule will have different formal charges even if they are the same element.

In some cases, two or more resonance structures may exist. For example, the nitrate ion (NO_3^-) has three equivalent resonance forms (Figure 1.2).

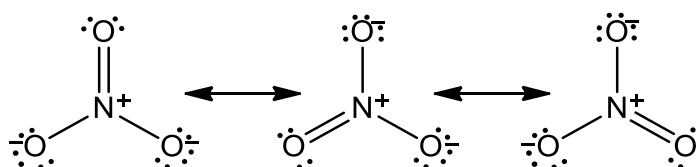


Figure 1.2: Resonance structures of the nitrate ion

² Generally octets surround atoms from the second period of the periodic table, especially C, N, O and F, as such atoms can accommodate a maximum of eight electrons in the available orbitals. However, atoms from the third and subsequent periods can often accommodate more than eight electrons. This is a phenomenon known as octet expansion.

In this example, formal charges are minimised by forming a double bond between one of the oxygen atoms and the central nitrogen atom. However, this double bond can be formed between any of the three oxygen atoms. Experiments show that all of the N–O bonds are identical (somewhere between a single and a double bond). To represent this, we depict all of the resonance structures, connected by double-headed arrows, to show that the true structure is a blend of all three resonance structures.

2.2. Number of Atoms Bonded to the Central Atom

The phosphite ion has three atoms bonded to the central atom.

2.3. Number of Non-bonding Electron Pairs on the Central Atom

The phosphite ion has one electron pair on the central atom.

2.4. Parent Geometry (*Chemistry: Atoms First* (2019), Chapter 4.6; Blackman *et al.*, *Chemistry* (2019), Chapter 5.4)

The parent geometry corresponds to the number of 'electron domains' about the central atom. An electron domain may be a single, double or triple bond, or a lone pair. The fundamental principle governing geometries in VSEPR theory is that electron domains render positions such that they are as far away from each other as possible due to the electrostatic repulsion. Names for the different parent geometries are given Table 1.2.

The phosphite ion has 4 electron domains (3 single bonds and one electron pair). Therefore, phosphite will possess a tetrahedral parent geometry.

2.5. Molecular geometry with ideal bond angles

The molecular geometry depends only on the number of atoms bonded to the central atom and the parent geometry. The molecular geometry (the 3D shape) may be different to the parent geometry.

The phosphite ion has 4 electron domains, with 3 atoms bonded to the central atom, plus one lone pair on the central atom. Therefore, although the parent geometry is tetrahedral, the molecular geometry is trigonal pyramidal (Figure 1.3).

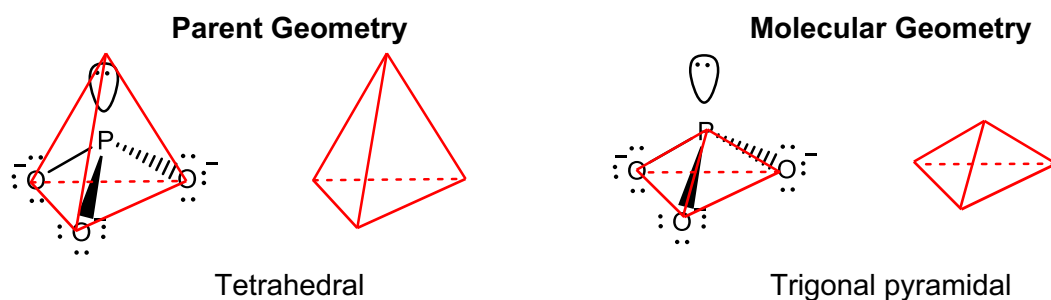


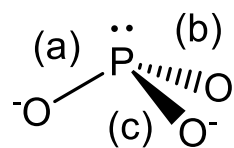
Figure 1.3: Difference in parent geometry and molecular geometry for the phosphite nitrate ion

2.6. Perspective Drawing

3D models can be used to help draw perspective structures. Also, use the following guidelines to draw the perspective structures:

Consider that the centre atom and at least one terminal atom are in the plane of a piece of paper.

- Bonds lying in the plane of the paper are represented by a solid line.
- Bonds projecting 'into' the paper away from you are represented with a hatched wedge-shaped line.
- Bonds projecting 'out of' the paper towards you are represented with a solid wedge-shaped line.



3. Report

Complete your laboratory report online, via Moodle. Ensure you complete all sections.

Your online report must be submitted within seven days of the completion of your class.

Table 1.2: Different parent and molecular geometries as described by VSEPR theory.

# of Electron Domains	Parent Geometry	# of Atoms Bonded	# of Lone Pairs e ⁻	Molecular Geometry	Bond Angles	Perspective Drawing
2	Linear	2	0	Linear	180°	
3	Trigonal Planar	3	0	Trigonal Planar	120°	
		2	1	Bent	<120°	
4	Tetrahedral	4	0	Tetrahedral	109.5°	
		3	1	Trigonal Pyramidal	<109.5°	
		2	2	Bent	<109.5°	
5	Trigonal Bipyramidal	5	0	Trigonal Bipyramidal	120° (equatorial) 90° (axial)	
		4	1	Disphenoidal (see-saw)	<120° (equatorial) <90° (axial)	
		3	2	T-shaped	<90°	
		2	3	Linear	180°	
6	Octahedral	6	0	Octahedral	90°	
		5	1	Square Pyramidal	<90°	
		4	2	Square Planar	90°	
		3	3	T-shaped	<90°	
		2	4	Linear	180°	

Exercise 2: The Chemistry Circuit

Learning Outcomes:



1. Determine the error of an auto pipette.
2. Accurately prepare a primary standard solution using a volumetric flask.
3. Prepare a saturated salt solution via incremental addition of a solute to a solvent.
4. Determine the accuracy of a dilution using UV-visible spectrophotometry.

Hazard Identification and Risk Assessment			
Identify the Hazard (the potential to do harm)	Risk (the probability that harm may result)	Control the Risk (preventing an incident)	Disposal of waste
Citric Acid May cause eye irritation. Harmful if swallowed. Irritating to skin.	Moderate	PPE (gloves, lab coat and safety glasses). Wash skin or eyes immediately under water if spill or splash occurs.	Down the sink with plenty of water
Sodium Hydrogen Carbonate Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
Sodium Chloride Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
Food colour (Yellow, colour components; 102 & 122) Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
Food colour (Green, colour components; 102 & 133) Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
Food colour (Blue, colour component; 133) Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
Food colour (Red, colour component; 124) Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
General Glassware Cuts	Medium	Handle with care, dispose of broken glass using dustpan and brush. If cut occurs, see your TA and seek first aid.	Labelled broken glass bin.

1. Introduction

There are many skills involved in handling laboratory instruments and equipment, particularly when working in a new laboratory environment. To exercise will assist you to become familiar with the First

Year Chemistry Laboratories, and the instruments, techniques and glassware to be used throughout this unit. This exercise will consist of individually performing four separate laboratory techniques.

1.1 Volumetric Flask

A volumetric flask is a precision piece of glassware calibrates to an accurate volume of solution (at a certain temperature). These flasks are used to prepare standard solutions with a known concentration, and to carry out precise dilutions.

1.2 Auto-Pipette

An auto-pipette is an excellent way to deliver a very precise amount of water or an aqueous solution. Auto-pipettes often do not work well with solvents other than water due to the low surface tension of solvent (the liquid will drip out of the tip before you can deliberately release it).

Use the standard operating procedure (SOP) “fixed volume auto-pipette” and practice using the pipette a few times before you perform your experiment.



2. Experimental Procedure

Work individually and record all experimental data immediately in your laboratory notebook.

Your TA will assign you to a starting experiment (2.1, 2.2, 2.3 or 2.4). You will have 30 minutes to complete that experiment, and you will then move on to the next experiment in the list. By the end of the session, you will have completed all four of the experimental procedures.

Practical skills tests: Some laboratory techniques will be checked by your TA so that you can receive feedback to help you improve. Copy Table 2.1 into your laboratory notebook, and have your TA observe you complete each task.

Figure 2.1: Fixed volume auto-pipette

Table 2.1: Practical skills test

Practical Skill	Satisfactory?	TA comments
Weigh a powder on a top-loading balance		
Correctly fold fluted filter paper		
Accurately make up a solution in a volumetric flask		

2.1 Accurately Making a Primary Standard Solution

A primary standard solution has an exact concentration of a reagent when it is prepared. They are made using a volumetric flask. Your TA will assign you a 25.00 mL solution to prepare: 0.0250 M citric acid or 0.0500 M sodium bicarbonate.

Use the values you calculated in your pre-laboratory quiz to determine the mass of solid required. All measurements, observations and calculations should be recorded into your laboratory notebook.

- Weigh out the mass of solid required in a weighing vial (use a top-loading mini laboratory balance, such as the one in Figure 2.2).
- Quantitatively transfer the solid into a 25.00 mL volumetric flask using a short-stemmed funnel. Make sure all of the solid is transferred into the flask (see Figure 2.3).
- Add deionised water until the flask is approximately $\frac{3}{4}$ full. Add the stopper, hold it on, and invert several times to mix the solution.
- Make the solution up to the mark with water use a dropping (Pasteur) pipette, and thoroughly mix the solution again.
- Test the pH of the solution using universal pH paper.



Figure 2.2: Top-loading mini balance and spatula

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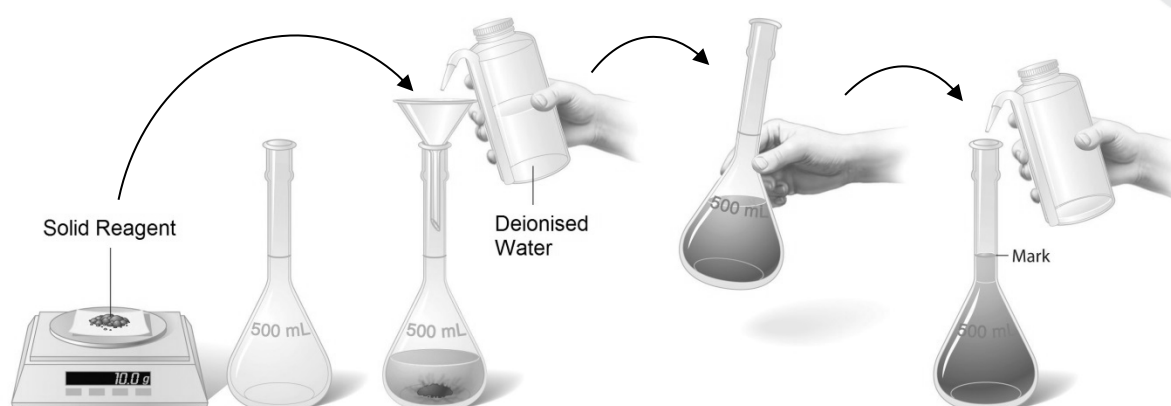


Figure 2.3: Making up a standard solution (Averill and Elderidge, 2007, General Chemistry, Ch 4.2).

2.2 Making a Saturated Sodium Chloride Solution

The skill of making a saturated solution is routinely used in chemistry; for example, when purifying a chemical via recrystallisation (a technique covered in semester 2). A saturated solution contains the maximum possible amount of a solute (e.g. NaCl) that dissolves in a solvent (e.g. water). The saturation point is dependent on a range of factors, including the temperature of the solvent, the solvent, and the solute.

What is the solubility of sodium chloride in water under standard laboratory conditions?

To make up a saturated solution, a volumetric flask need not be used; however, it is good scientific practice to record: volume and temperature of the solvent, and mass of solute used to make up the solution, so that you can replicate the experiment in the future.

- Using a measuring cylinder, add 10 mL of deionised water to a (labelled) 100 mL beaker. Record the temperature of the water.
- In a weighing vial, weigh out ~2.5 g of sodium chloride on a top-loading mini balance. Record the exact mass weighed.
- Add the sodium chloride to the beaker of water, and stir the solution with a glass stirring rod until all of the solid has dissolved.
- Continue adding ~0.5 g of sodium chloride at a time, stirring after each addition. Record the mass of sodium chloride added at each addition.

- e) Once you have reached the saturation point for the solution (when no more solid will dissolve), calculate the mass of sodium chloride required to produce the saturated solution.
- f) Filter the saturated sodium chloride solution through fluted filter paper (see Figure 2.4) into a 100 mL conical flask using a long stem funnel, and discard the filter paper and precipitate into the solid-waste bucket. The solution in your beaker is a saturated sodium chloride solution.
- g) Clean all equipment and glassware used.

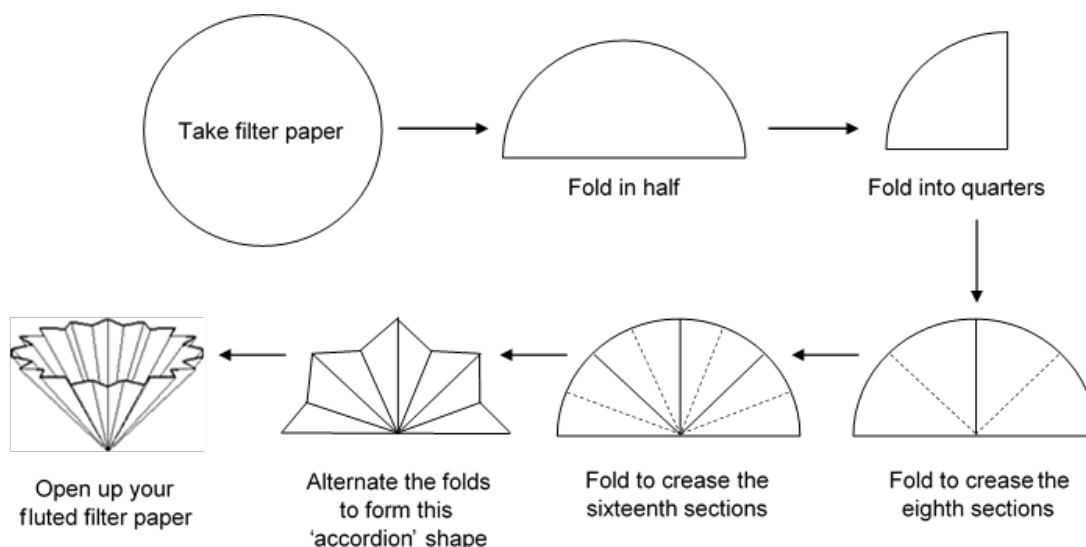


Figure 2.4: How to fold fluted filter paper

2.3 Accurate Dilution of a Solution

To perform a dilution with accuracy is a fundamental chemistry skill. Analytical glassware is used to perform a dilution, which provides measurements to a high degree of accuracy and precision.

Today, you will perform a dilution of a food dye, then check your accuracy using UV-visible spectrophotometry. You will make up your diluted solution using a burette and a volumetric flask.

There are four stock solutions of food dye to choose from (see Table 2.2). Please perform the dilution and measurements for one only. Clearly record in your laboratory notebook which food dye used.

Table 2.2: Food dye stock and dilute solution concentrations and wavelengths for absorbance

Food dye	Stock Solution Concentration	Dilute Solution Concentration	λ_{max}
Red	33.0 ppm	6.60 ppm	510 nm
Yellow	23.0 ppm	4.60 ppm	420 nm
Green	22.0 ppm	4.40 ppm	630 nm
Blue	7.20 ppm	1.44 ppm	630 nm

You will make up 25.00 mL of the diluted solution. Use the values you calculated in your pre-laboratory quiz to determine the volume of stock solution you will need to measure out. All measurements and observations should be recorded into your laboratory notebook.

- Prepare a 25.00 mL volumetric flask by rinsing it with deionised water, and label the flask.
- Label a clean 100 mL beaker; in the beaker, collect approximately 15 mL of the food dye stock solution.
- The burette is set up on a retort stand (see Figure 2.5). Rinse the burette with 2 x 5 mL water, then 1 x 3 mL stock solution.
- Using a funnel, add the remainder of the stock solution to the burette.
- Remove the funnel from your burette. Remove any air bubbles from the tip of the burette by running a small volume of solution from the burette into a waste beaker.
- Record the volume of the solution (bottom of the meniscus) to within 0.05 mL.
- Into the volumetric flask, deliver the required volume of stock solution to be diluted, record the volume of the solution.
- To make up the solution, follow the procedure as set out in Section 2.1, steps c and d.

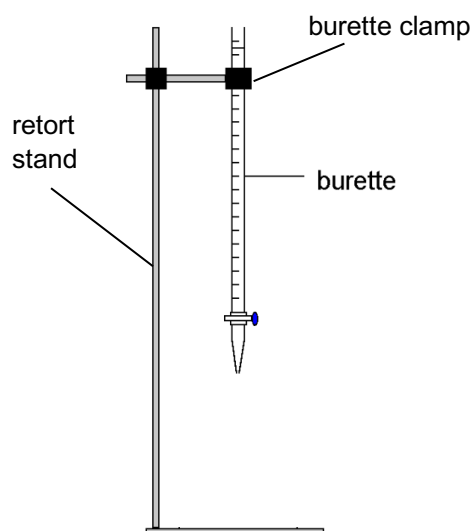


Figure 2.5: Burette set-up

- Using a Pasteur pipette, transfer some of the diluted solution into a cuvette (about $\frac{3}{4}$ full). Wipe the outside of the cuvette with a Kimwipe. Also prepare cuvettes of the stock solution, and water (blank) for analysis.
- Measure the absorbance (A) of your diluted solution, and the stock solution, at the wavelength listed in Table 2.2, using water as the blank. Follow the SOP for the Pasco Visible Spectrophotometer. Record the absorbances in your laboratory notebook, and calculate the percentage error³ of your dilution.
- Clean up all equipment and glassware used.



2.4 Determination of the Error in an Auto-Pipette

It is important to practice your skills with the auto-pipette to ensure accurate delivery of solution volume. Practice using the auto-pipette a few times. Copy Table 2.3 into your laboratory notebook to fill out with your measurements.

- In a vial, weigh the mass of an aliquot of water from the auto-pipette, using the top-loading mini balance. Tare the balance and vial before weighing the aliquot.
- Repeat this measurement so that you have a total of three replicates.
- The density of water at 22 °C is 997.77 kg m⁻³. Calculate the mean aliquot volume (μL) and standard deviation⁴ of the measurements.
- Tidy up all equipment used.



$$^3 \%Error = \frac{|Abs_{stock\ solution} - (Abs_{dilute\ solution}) \times 5|}{Abs_{stock\ solution}} \times 100$$

$$^4 \text{ To calculate the standard deviation: } SD = \sqrt{\frac{\sum (x_1 - \bar{x})^2}{n-1}}$$

- Convert the masses of water (g) to volumes (in μL)
- Calculate the mean volume
- For each volume: subtract the mean and square the result
- Calculate the mean of those squared differences (divide by $n-1$)
- Standard deviation is the square root of the value obtained in iv).

Table 2.3: Determining the error in an auto-pipette

Pipette Make and Volume:		Pipette ID Number:
Aliquot Number:	Weight (g)	Volume (μL)
1		
2		
3		
Mean		Mean $\pm$ sd

3. Report

Complete your laboratory report online, via Moodle. Ensure you perform all necessary calculations and complete all sections.

Additional guidance for writing the reflective discussion.

You are probably used to writing about science in an objective way. However, it is often useful to reflect on your experiences in the laboratory or on your course in order to evaluate how you are developing, especially in terms of transferable and personal skills and qualities. Being able to reflect on what you've learned and communicate it (for example, to an employer) is an important skill.

This type of writing is very different from the usual writing you may have been doing and as scientists, we often find it quite challenging. You will be asked to write reflective pieces throughout your chemistry course. Reflective writing should:

- Capture our response to experiences, opinions, events, or new information.
- Look at how experiences might be useful in the future.
- Describe your response to thoughts and feelings.
- Be a way of thinking.
- Explore your learning.
- Be an opportunity to gain self-knowledge.
- Move beyond subject knowledge and skills to focus on personal development.
- Be personal.

A reflective discussion will help remind you what you have learned during an activity. Reflection helps you bring ideas together and helps you learn more out of an activity. Discuss what you learned during this practical exercise, in terms of transferable skills and personal qualities. How did this build on previous experiences and how might it be useful in the future? (Use full sentences and discuss at least three points). For example: I found it hard to make sure I wrote all my notes down but with so many observations I could see how important it was to do that. (Do not use this example!)

We are looking for you to reflect on soft/transferable skills, and personal growth, rather than focusing on chemistry skills/techniques.

We don't want you to discuss the chemistry (for example, although you may have learned how a spectrometer works, or how to make up a standard solution, we don't want you to discuss these, as they are chemistry techniques). We do want you to reflect on what you have learned personally which could then be applied to future situations (which could include labs, although we'd love for you to think beyond that as well! Think about things like time management, organisational skills, being prepared, etc.). You could mention these in the context of chemistry labs (in the sense that that is where you have learned them), but they are not specifically chemistry skills.

Your online report must be submitted within seven days of the completion of your class.

Exercise 3: Transformations of Copper

Learning Outcomes:



1. Understand the difference between a physical and chemical change.
2. Appreciate that solubility rules can simplify the task of converting one compound to another.
3. Perform a separation by filtration.
4. Perform calculations involving molarities and stoichiometry.

Hazard Identification and Risk Assessment			
Identify the Hazard (the potential to do harm)	Risk (the probability that harm may result)	Control the Risk (preventing an incident)	Disposal of waste
Sodium Hydroxide (4 M) Corrosive, causes burns, damaging to eyes	Low risk	Avoid contact with skin and wash immediately with water if spill occurs. In the case of contact with eyes, seek medical advice.	Corrosive waste carboy in fumehood
Sulfuric Acid (6 M) Corrosive, causes burns, damaging to eyes.	Medium risk	Use in fumehood. Handle with care. Wash skin immediately under water if spill occurs. In the case of contact with eyes, seek medical advice.	Corrosive waste carboy in fumehood
Hydrochloric Acid (5.5 M) Corrosive, causes burns, damaging to eyes.	Medium risk	Use in fumehood. Handle with care. Wash skin immediately under water if spill occurs. In the case of contact with eyes, seek medical advice.	Corrosive waste carboy in fumehood
Copper(II) Chloride Damaging to eyes, can irritate skin. Harmful if ingested or inhaled	Low risk	Avoid contact with skin and wash with water immediately if spill occurs.	Corrosive waste carboy in fumehood
Copper Metal	Low risk	Avoid contact with skin and wash hands after handling.	White bucket
Zinc Powder Irritating to eyes and respiratory system	Low risk	Avoid inhalation of solid and spilling onto skin. Wash skin with water if it comes into contact.	Put filter paper with zinc residue in white bucket
Hotplate Burns	Low risk	Do not place hands directly over hotplate or directly on surface. Use wooden peg to remove hot objects.	N/A
General glassware Cuts	Medium risk	Handle with care, dispose of broken glass using dustpan and brush. If cut occurs, see your TA and seek first aid.	Labelled broken glass bin.

Background: Workbook Week 0; Chemistry: Atoms First (2019), Chapter 7; Blackman et al., Chemistry (2019), Chapter 3.

1. Introduction

In chemistry, it is very important to be able to manipulate the state of a metal salt or metal. This is especially important in mining where the ore that is mined is always in a different form from what we need for use in manufacturing. For example, when Cu_2S ore is heated with air, it reacts with oxygen to form copper metal and sulfur dioxide ($\text{Cu}_2\text{S} (\text{l}) + \text{O}_2 (\text{g}) \rightarrow 2 \text{Cu} (\text{l}) + \text{SO}_2 (\text{g})$).

In this laboratory exercise you will transform a copper salt into three different forms to produce copper metal (Figure 3.1). As you do this, you will gain experience observing and recording your scientific observations. These are very important scientific skills.

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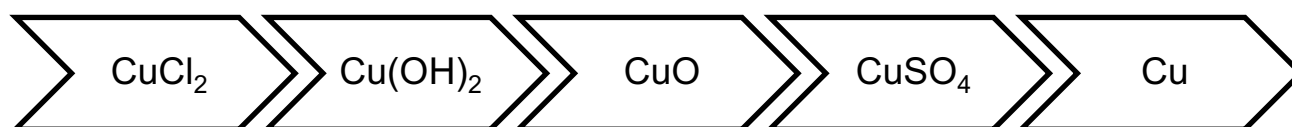
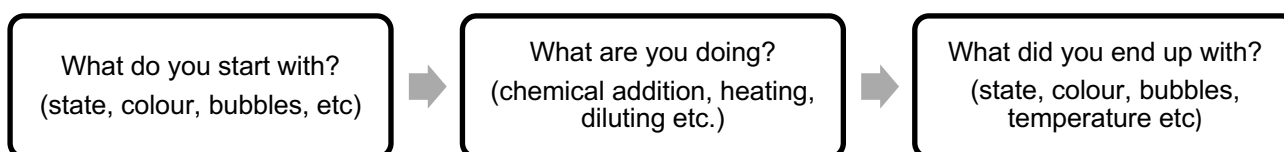


Figure 3.1: Copper transformations

1.1 Recording Results and Observations

When recording observations for experiments, it is important to use unambiguous wording, and be as specific as possible. Recording accurate observations makes it much easier to work out what (if any) reaction has occurred. The properties of reactants and products (i.e. colour, state, physical appearance etc.) inform us when writing chemical equations. For example, 'clear' can be an ambiguous word – is it colourless or transparent?

Observations for reactions should cover at least three key points:



Example: The aqueous copper chloride solution started out as transparent and bright blue. Upon addition of 4 M sodium hydroxide a precipitate formed. The solution became opaque (due to the suspended precipitate) and the colour changed to a very light blue.

1.2 Writing Chemical Equations

Chemical equations are written to assist with explaining observations, these include full equations, ionic equations and net ionic equations. It is important to remember that the chemical equations should match the observations. Always include states: solid (s), liquid (l), gas (g) and aqueous (aq) meaning dissolved in water and balance the equations.

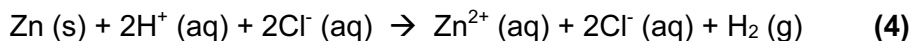
For example, start by writing out the reactants on the left-hand side (LHS) of the reaction arrow, and the products on the right-hand side (RHS) **(1)**. Next, balance the equation (there should be exactly the same number of each atom on both sides of the reaction **(2)**). Finally, add the states based on your observations to give the full equation **(3)**.

$\text{Zn} + \text{HCl} \rightarrow \text{ZnCl}_2 + \text{H}_2$ **(1)** LHS & RHS are not balanced

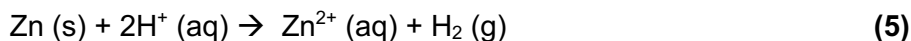
$\text{Zn} + 2 \text{HCl} \rightarrow \text{ZnCl}_2 + \text{H}_2$ **(2)** 2 times HCl on LHS to balance the Cl & H atoms on RHS

$\text{Zn} (\text{s}) + 2 \text{HCl} (\text{aq}) \rightarrow \text{ZnCl}_2 (\text{aq}) + \text{H}_2 (\text{g})$ **(3)** Add the states

Notice that the HCl and ZnCl₂ are in the aqueous state because the reaction was performed in water. Acids, bases and salts usually dissolve in the water, and will thus be present in their ionic forms. These ions are dissociated and this can be represented by an ionic equation (4).



From the ionic equation it is possible to determine which atoms/ions have undergone a physical change, and which are spectator ions. When writing the net ionic equation we omit the spectator ions, as they do not play a part in the actual chemical reaction (5).



2. Experimental Procedure

2.1 Copper(II) chloride to copper(II) hydroxide

- Weigh 0.10 – 0.12 g of CuCl₂·2H₂O in a weighing vial (use a top-loading laboratory balance).
- Weigh the capped vial and contents accurately on an analytical balance.
- Transfer the solid into a 100 mL beaker.
- Re-weigh the capped vial on the analytical balance.
- Calculate the accurate mass of CuCl₂·2H₂O added.
- Add 10 mL of deionised water to the beaker and stir to dissolve.

GSP

Record your observations. Write an ionic equation for the change that occurred.

Add about 0.5 mL of NaOH (4 M) dropwise with stirring. Note: a full Pasteur pipette contains approximately 1.5 mL. Any solid which forms is copper(II) hydroxide, Cu(OH)₂. Record your observations. Write a net ionic equation for the change that occurred.



To ensure the reaction is complete, there must be excess NaOH in the solution. The presence of excess OH⁻ can be tested by checking the pH with pH paper. Test your solution by immersing a glass rod in the solution, and touch a small square of pH paper on a watch glass. If the solution is not basic, add more NaOH solution until it is basic.

2.2 Copper(II) hydroxide to copper(II) oxide

Heat the contents of the beaker on a hotplate for about 5 minutes, stirring occasionally. This will remove a water molecule to give copper(II) oxide, CuO. Record your observations. Write an equation for the reaction.

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2.3 Copper(II) oxide to copper(II) sulfate

After 5 minutes, carefully remove the beaker from the hotplate using a wooden peg and let it cool down to room temperature in the fumehood. Filter the copper(II) oxide precipitate through a fluted filter paper (see Exercise 2, Figure 2.5) into a 100 mL conical flask using a glass funnel. Transfer all of the solid to the filter paper; rinse the beaker with deionised water into the filter paper/funnel. Discard the filtrate.

Place a 100 mL beaker under the filter funnel. Pierce a hole in the filter paper using a spatula and wash the precipitate through the hole with approximately 2 mL of 6 M sulfuric acid, H₂SO₄ (using a Pasteur pipette). Wash through the remaining solid on the filter paper with the acid filtrate in the beaker. When all the solid has dissolved, wash



the filter paper with approximately 4 mL water. Record your observations. Write the net ionic equation for the reaction.

2.4 Copper(II) sulfate solution to copper

Use a small spatula to measure out approximately a pin tac's worth (●) of zinc powder, Zn, onto a watch glass. In the fumehood carefully add the zinc powder in very small portions to the coloured solution. Allow the beaker to stand, stirring occasionally with a glass stirring rod until the solution becomes colourless. If the solution is not completely colourless, add a little more Zn. Record your observations. Write an equation and the net ionic equation for the reaction.



Wash the precipitate twice. To do this, allow the precipitate to settle and decant (pour off) most of the supernatant (solution above the precipitate). Add approximately 10 mL of water, swirl the contents, allow the precipitate to settle and decant the supernatant. Repeat with a further 10 mL water. Add approximately 0.5 mL 5.5 M hydrochloric acid (HCl) to the solid and stir. Record your observations.

You may or may not observe a reaction here. If you do, write the equation and the net ionic equation. If you do not, explain why HCl was added to the solid. Solid zinc reacts with HCl to evolve hydrogen gas, H₂, but copper does not.

Wash the precipitate with deionised water twice by decantation, leaving it almost dry. Place the beaker containing the precipitate on the hotplate and heat it until it appears dry. Remove the beaker from the hotplate using a wooden peg and let it cool down to room temperature in the fumehood.

2.5 Calculation of Percentage Yield

Use the analytical balance to weigh a clean, dry weighing vial and cap. Record the mass.

Quantitatively transfer the precipitate into the vial. Record the mass.

Use your results to calculate the percentage yield of copper.

Show your copper sample to your TA for inspection. Clean all laboratory equipment used.



3. Report

Complete your laboratory report online, via Moodle. Ensure you perform all necessary calculations, complete all sections, and upload your laboratory notes to Moodle.

Additional guidance for writing the technical discussion.

This is where you discuss and give meaning to the results. You should also detail any experimental errors and make any other relevant comments about the experiment. Include possible improvements. Ensure you are not re-stating the method.

Discussion points to consider: (Note: The points below are not intended as questions! They are here to give guidance on what to include in your discussion. Do not simply list answers - incorporate them into a full discussion.)

- Relate results back to the aim and introduction of the experiment – what was trying to be done, did the results support the aim of the experiment?
- Discuss the yield. Are there alternative reaction pathways which could improve the expected yields? What are they?

- Discuss the chemistry behind the colour changes, and relate this to the actual transformations of copper throughout the experiment.
- What factors may have affected the final result? What are some sources of error that may have been encountered, and how would they have impacted on the results?
- If you actually wanted to achieve copper at the end (for example, if you had an ore that you were extracting), would this be a suitable method? Why/why not?

Your online report must be submitted within seven days of the completion of your class.

Exercise 4: Calorimetric Analysis of Baking Powder

Learning Outcomes:

1. Distinguish between exothermic and endothermic reactions.
2. Use calorimetry to determine the enthalpy of a chemical reaction.
3. Devise a method using calorimetry to analyse a powder with an unknown composition.
4. Manipulate data and determine unknown compositions using a calibration curve.



The IDEA Experiments you will come across in units such as CHM1011, BIO1011 and PHS1011 are a little different to some of the laboratory exercises you will have already done in your Science degree. These exercises will not come with a procedure provided in the laboratory manual. For some parts of this exercise, you will have to design and perform your own experiment.

You will be working in a group of 3-4 students. You need to assign roles within your group and delegate the work that has to be done.

At the end of the session, you need to have finalised all experimental work, shared the data and discussed your results with your group.

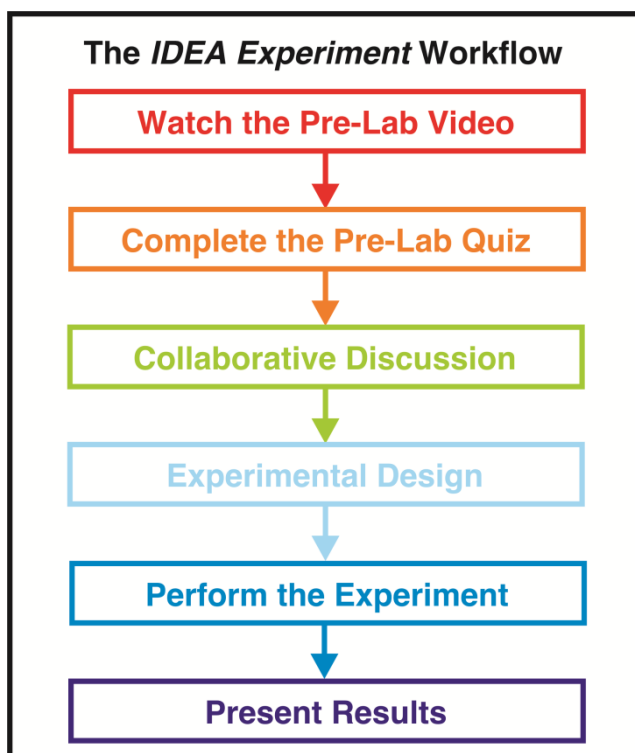
"IDEA" stands for Inquiry-Design-Explore-Answer, and that's exactly what working scientists do in their professional life.

Inquiry: A sound scientific approach to answering questions and solving problems always commences with the process of asking deep questions. What is the quickest way to perform this experiment? The most ethical? The least expensive? The 'greenest'? Are you able to foresee roadblocks along the way?

Design: Once you have considered the limitations, challenges and practicalities of the experiment, you can start to design your experiment. Weigh up what expertise and techniques you have at your disposal. Ask questions of each other, your TA, and the literature, and come up with a flow chart for how the experiment will proceed.

Explore: When you are ready to commence, delegate different jobs amongst your team, and start to gather data. You may find along the way that your design needs to be changed.

Answer: Once you have gathered the best data you can, see if you are able to form some conclusions. Discuss the results of your experiments amongst your group and determine whether the original problem has been solved.



Hazard Identification and Risk Assessment			
Identify the Hazard (the potential to do harm)	Risk (the probability that harm may result)	Control the Risk (preventing an incident)	Disposal of waste
Citric Acid May cause eye irritation. Harmful if swallowed. Irritating to skin.	Moderate	Wash skin or eyes immediately under water if spill or splash occurs.	Down the sink with plenty of water
Sodium Hydrogen Carbonate Non-hazardous	Low	Wash skin immediately under water if spill occurs.	Down the sink with plenty of water
General glassware Cuts	Medium	Handle with care, dispose of broken glass using dustpan and brush. If cut occurs, see your TA and seek first aid.	Labelled broken glass bin.

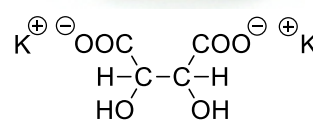
Background: Workbooks Weeks 7 and 8; Chemistry: Atoms First (2019), Chapter 9; Blackman et al., Chemistry (2019), Chapter 8.

1. Introduction

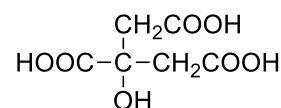
Baking powder and baking soda are common ingredients used in cakes and other baked goods. Baking soda is pure sodium bicarbonate (NaHCO_3) whereas baking powder consists of baking soda and one or more acid salts. These salts are usually classified into two categories: low-temperature acid salts (see Figure 4.1) such as potassium tartrate or calcium dihydrogen phosphate, and high-temperature acid salts such as sodium aluminium sulfate or sodium aluminium phosphate. If baking soda is used, then an acidic ingredient, such as lemon juice (which contains citric acid) or vinegar (which contains acetic acid) must be added to the batter so that a chemical reaction occurs.



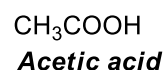
In this practical a mixture of citric acid (CA) and sodium bicarbonate (SB) will be used. Citric acid is a triacid ($\text{pK}_{\text{a}1} = 3.1$, $\text{pK}_{\text{a}2} = 4.7$ and $\text{pK}_{\text{a}3} = 6.4$). Sodium bicarbonate (NaHCO_3) is an amphoteric species: it can act as an acid and as a base ($\text{pK}_{\text{a}}(\text{HCO}_3^-/\text{CO}_3^{2-}) = 10.4$ and $\text{pK}_{\text{a}}(\text{H}_2\text{CO}_3/\text{HCO}_3^-) = 6.4$).



Potassium tartrate



Citric acid



Your goal is to determine the exact composition of Monash University's new baking powder.

2. Calorimetry

Calorimetry is used to quantify the heat released or absorbed by a chemical reaction or a physical process. Chemical reactions that raise the temperature (release thermal energy) are called

Figure 4.1: Chemical structures.

exothermic reactions. Chemical reactions that lower the temperature (absorb thermal energy) are called endothermic reactions. Change in temperature due to chemical reactions is measured as enthalpy (H). The enthalpy of a chemical species describes its internal energy.

If a reaction is exothermic, then the chemical species loses internal energy to its surroundings (raising the temperature), and therefore ΔH is negative. Conversely, if a reaction is endothermic, the chemical species absorb energy from its surroundings (lowering the temperature), and therefore ΔH is positive. We will be investigating the thermodynamics of the chemical reaction between citric acid and sodium bicarbonate.

The change in enthalpy (ΔH) of a chemical reaction is related to the change in temperature (ΔT):

$$\Delta H = -mC\Delta T$$

where m is the total mass of solution (g) and C is the heat capacity ($\text{J}\cdot\text{K}^{-1}\cdot\text{g}^{-1}$).

Recording the change in the temperature of the system gives information about the change in enthalpy of the system.

3. The Experiment

After instructions from your TA, record all observations and determine the chemical reaction that occurs in your laboratory notebook. As you conduct your experiments, some useful things to consider might be:

- What is the balanced chemical equation?
- What type of reaction is it?
- Can you explain your observations based on the chemical reaction?
- Is the reaction exothermic or endothermic?
- What would the energy diagram (to illustrate the relative ΔH) for this reaction look like?
- How will you measure ΔT and why will you measure it like that?
- What mixture compositions will you use, and why?
- If you were to perform an experiment using 2 g of only one solid (either citric acid or sodium bicarbonate), what would you expect for the change in temperature? Why? Would it be worth trying it out?
- What calculations will you need to perform?
- What method will you use to determine the composition of Monash University's baking powder? What glassware and equipment will you use?
- What composition of baking powder would you suggest that Monash University produces to give the best cake (if we were to assume that the more gas produced, the more the cake will rise)? Can you explain your answer using experimental results and the chemical equation?
- Using your experimental results, can you calculate (or approximate) the change in enthalpy of the chemical reaction?

4. Calibration Curve

In order to determine the composition of a mixture, scientists determine calibration curves using mixtures of known compositions. They then measure and plot the change in the 'significant parameter' (y axis) versus the composition (x axis) as shown in Figure 4.2. The choice of experimental data is very important to cover the whole range of expected compositions. Finally, the scientists perform the same experiment with the mixture of unknown composition and use the calibration curve to deduce the composition of the mixture.

In the following example, the change in temperature (ΔT) is plotted against the mass percentage of solid A. Six mixtures are prepared within the range 0-50%. The unknown mixture has a ΔT of 5.0 °C. Using the line of best fit (line that matches most of the experimental data – see Figure 4.2), the mass percentage of solid A can be deduced and found as 25%.

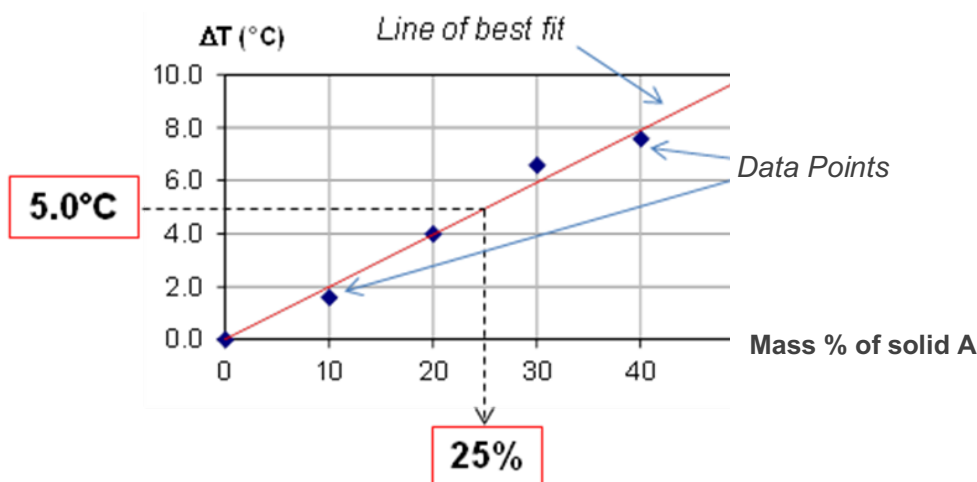


Figure 4.2: Example calibration curve.

In the experiment you will perform, the significant parameter will be ΔT which will be plotted against the mass fraction of citric acid x_{CA}^{mass} or the molar fraction of citric acid x_{CA}^{molar} .

$$x_{CA}^{mass} = \frac{m_{CA}}{m_{TOT}}$$

m_{CA} = Mass of citric acid

m_{TOT} = Total mass of powder to be analysed

$$x_{CA}^{molar} = \frac{n_{CA}}{n_{TOT}}$$

n_{CA} = Moles of citric acid

n_{TOT} = Total moles of powder to be analysed

$$\text{molar ratio} = \frac{n_{SB}}{n_{CA}}$$

n_{SB} = Moles of sodium bicarbonate

Choose the experimental data with care to cover the whole range of expected compositions.

For each mixture, the total mass must be 2.00 g. The first composition you will investigate is given below. Then you are free to choose your own composition of mixtures and to investigate as many mixtures as you wish (see Table 4.1).

$$m_{TOT} = 2.0 \text{ g}$$

$$m_{CA} = 0.87 \text{ g}$$

$$m_{SB} = 1.13 \text{ g}$$

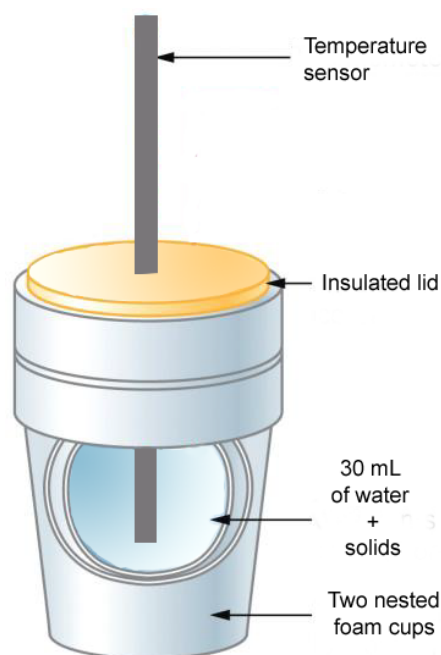
Table 4.1: Calorimetry data.

Citric acid (CA)		Sodium bicarbonate (SB)		Total mass	Total moles	x_{CA}^{mass}	x_{CA}^{molar}	Molar ratio	ΔT (°C)
m (g)	n (mol)	m (g)	n (mol)	m_{TOT} (g)	n_{TOT} (mol)				
0.87		1.13							

4.1 Experimental Procedure

Remembering the pre-laboratory video:

- Nest two foam cups inside each other and place in 400 mL beaker.
- Add 30 mL of water into the cup and place a lid on it. The water should be brought to room temperature prior to beginning the experiment. Why?
- Clean, dry and label 3 weighing containers (citric acid, NaHCO_3 , mixture).
- Weigh out the desired amount of citric acid into the labelled container. Record the exact mass.
- Weigh out the desired amount of sodium bicarbonate into the labelled container. Record the exact mass.
- Transfer both powders into the container labelled mixture. Reweigh the empty containers.
- Insert the Pasco temperature sensor through a small hole in the lid of the cup. Do not touch this when running an experiment. Why?
- Click the "Record" button on the program to begin data recording. The temperature should be constant. If not, click the "Stop" button, wait for a minute before starting again.
- After about 15 seconds have elapsed, lift off the cup lid and quickly add the mixture to the water. Replace the cup lid (avoid gaps). Swirl the beaker for 10-15 seconds. Make sure the temperature sensor is on the edge of the cup while stirring so the temperature can be recorded at any time.
- After overall 30 seconds have elapsed, leave the cup on the bench with the temperature sensor in it. Record the temperature for 5 minutes for the first experiment (for your subsequent experiments you can stop the experiment when you choose).
- Click the "Stop" button to end data recording.
- Dispose of the reaction products in the sink. Rinse the cup with distilled water for use in the next experiment.
- Repeat from step b with different compositions of citric acid and sodium bicarbonate.



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Figure 4.3: Experimental setup

When graphing your data, plot:

- ΔT (y-axis) versus the mass fraction of citric acid x_{CA}^{mass} (x-axis)
- ΔT (y-axis) versus the molar fraction of citric acid x_{CA}^{molar} (x-axis)
- Determine the line of best fit for the graphs

5. Determination of the Composition of the Baking Powder

In your laboratory book, provide the method you plan to perform to determine the composition of the Monash Baking Powder. Your TA must check your method before you can go on. They will provide you with feedback to help improve your experiment design if needed.

Perform the experiment and determine the composition of the Monash Baking Powder. If necessary, you can perform further tests in order to determine the composition. Explain these tests in your report.

Once you have recorded all the data needed in your laboratory book, go to 'Experiment' on the program and select 'Delete All Data Runs' from the list.

Clean all laboratory equipment used



6. Report

Complete your laboratory report online, via Moodle. Ensure you perform all necessary calculations, complete all sections, and upload your graphs and laboratory notes to Moodle.

Your online report must be submitted within seven days of the completion of your class.

Exercise 5: Acid-base Titrations

Learning Outcomes:

1. Construct and interpret an acid-base titration curve.
2. Experiment with a standard pH meter.
3. Distinguish weak and strong acids/bases.
4. Distinguish mono- and polyprotic acids/bases.



Note: Despite only dilute solutions of strong acids and strong bases being used, both are still quite corrosive and particular caution should be taken during handling.



Hazard Identification and Risk Assessment			
Identify the Hazard (the potential to do harm)	Risk (the probability that harm may result)	Control the Risk (preventing an incident)	Disposal of waste
Hydrochloric Acid (0.1 M) Corrosive Causes burns, damaging to eyes.	Solution is dilute. Low risk.	Handle with care. Wash skin immediately under water if spill occurs.	Neutralise and dispose of down the sink with copious amounts of water.
Sodium Hydroxide (0.1 M) Corrosive Causes burns, damaging to eyes.	Solution is dilute. Low risk.	Handle with care. Wash skin immediately under water if spill occurs.	Neutralise and dispose of down the sink with copious amounts of water.
Sodium Carbonate (Na_2CO_3) (solid powder and 0.1 M) Harmful if swallowed Irritating to eyes and skin	Mass of powder used is small. Solution is dilute. Low risk.	Wash skin immediately under water if spill occurs.	Neutralise and dispose of down the sink with copious amounts of water.
General glassware Cuts	Medium	Handle with care, dispose of broken glass using dustpan and brush. If cut occurs, see your TA and seek first aid.	Labelled broken glass bin.

Background: Workbooks Weeks 9 and 10; *Chemistry: Atoms First* (2019), Chapter 14; Blackman *et al.*, *Chemistry* (2019), Chapters 9 and 11.

1. Introduction

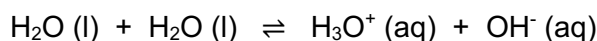
Acids and bases are central to the science and chemistry of water. They are also used extensively throughout industry and are important in nature and food science. The Brønsted-Lowry definition of acids and bases considers an acid-base reaction as a proton transfer from an acid to a base. Thus, within the Brønsted-Lowry definition, an acid is a proton donor and a base is a proton acceptor. In this practical exercise we investigate some of the properties of acids and bases using acid-base titrations.

1.1 The definition of pH

The acidity of an aqueous solution is related to the activity of hydronium ions in solution. However in the laboratory, concentration⁵, $[H_3O^+]$, can be easily measured and is a good approximation for activity. Because of the wide range of $[H_3O^+]$ values encountered, (1 to 10^{-14} M), the acidity is usually measured using a log scale; this is the pH scale. The pH of a solution is calculated by:

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

Water always participates in a self-dissociation equilibrium:



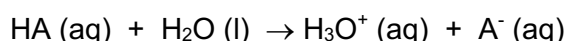
Described by the self-dissociation equilibrium constant:

$$[H_3O^+][OH^-] \equiv K_w = 1 \times 10^{-14} \text{ at } 25^\circ\text{C}.$$

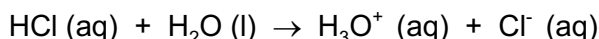
The pH of a neutral solution is 7.00 at 25°C , and $[H_3O^+] = [OH^-] = 1 \times 10^{-7}$ M. Acidic solutions have lower pH values (high $[H_3O^+]$), whereas basic solutions have higher pH values (low $[H_3O^+]$).

1.2 Strong Acids and Bases

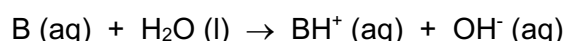
A strong acid is one that completely dissociate in water. The generalised equation for a strong acid is:



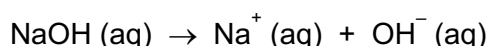
Note the use of the one-way arrow implies that the reaction goes all the way and is not reversible. Hydrochloric acid is an example of a strong acid:



A strong base is one that completely dissociates in water. The generalised equation for a strong base is:

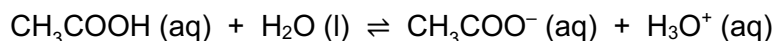


Sodium hydroxide is an example of a strong base:



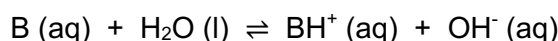
1.3 Weak Acids and Bases

A weak acid is one that coexists at equilibrium with its conjugate (corresponding) base. Acetic acid is an example of a weak acid:



Note the use a reversible arrow for the reaction of weak acids and bases.

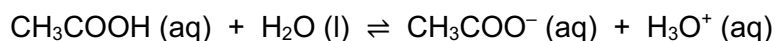
The generalised equation for a weak base is:



⁵ Note: $[X]$ = Concentration of X, in mol L⁻¹

1.4 Acidity Constants

A weak acid exists at equilibrium with its conjugate base. For example, with acetic acid (CH_3COOH):



A reversible arrow is used to depict the equilibrium reaction.

The acidity constant, K_a (an equilibrium constant for an acid), can be determined from the concentrations of acid and base in solution as follows:

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

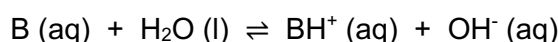
The “p” notation (as used in pH) is based on the definition, $\text{pX} = -\log [\text{X}]$, where p is an abbreviation for power. Using this definition, we can write:

$$\text{p}K_a = -\log_{10} K_a$$

Combining these equations, we find that for any weak acid (HA) with its conjugate base (A^-):

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

A weak base also exists at equilibrium with its conjugate acid. Based on this equation, the basicity constants, K_b , can be defined.



1.5 Measuring pH with a pH Meter

pH can be measured using a glass electrode and a suitable reference electrode. Frequently, the glass electrode and the reference electrode are housed in the same casing as a combination electrode (Figure 5.1). The electrical potential of the glass electrode is sensitive to changes in pH, whilst the reference electrode remains at a fixed potential. The two electrodes, together with the solution between them form an electrical cell, and a voltmeter measures the difference between the two electrodes and can be calibrated to display a reading in pH units (Figure 5.2).

Note: The combination pH electrode is fragile, so raise and lower it with care and do not use it to stir the solution. Keep the electrode immersed in distilled water between readings as it must not be allowed to dry out, so.

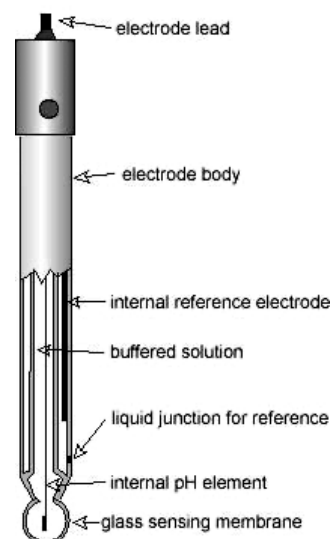


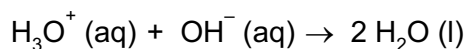
Figure 5.1: Combination pH electrode

Figure 5.2: The pH meter

The readings of a pH meter are only valid if the meter has been calibrated with a buffer of known pH appropriate to the range of measurement. Additionally, temperature has a significant effect on pH measurements, so temperature should always be reported along with pH.

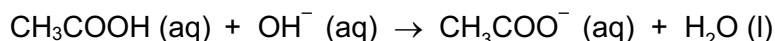
1.6 Acid-base titrations

In a titration, you react a solution with an unknown concentration, with a solution of known concentration in a quantitative manner to determine the unknown concentration. Acid-base titrations are a very common type of titration, and the following reaction occurs:



At this point, you have exact equivalents of the acid and base and have found the equivalence point.

Acid-base titrations can also be carried out using a weak acid and strong base. For example, acetic acid only partially reacts with water, but reacts readily with hydroxide ions when reacted with sodium hydroxide (NaOH), and the following reaction occurs:



In this exercise we will use the pH meter to follow an acid-base titration. We will add an acid standard to a base solution with unknown concentration containing the pH meter. As acid is titrated into the base, the pH decreases, slowly at first and then more quickly until after the equivalence point when the rate of decrease slows down again. The equivalence point is where the number of moles of the standard is equivalent (in stoichiometric ratio) to the number of moles of the unknown. The equivalence point can be determined from a graph of pH against the volume of acid added by finding the point of maximum slope, which is the point at which the curve changes from convex to concave (see Figure 5.3 for an example).

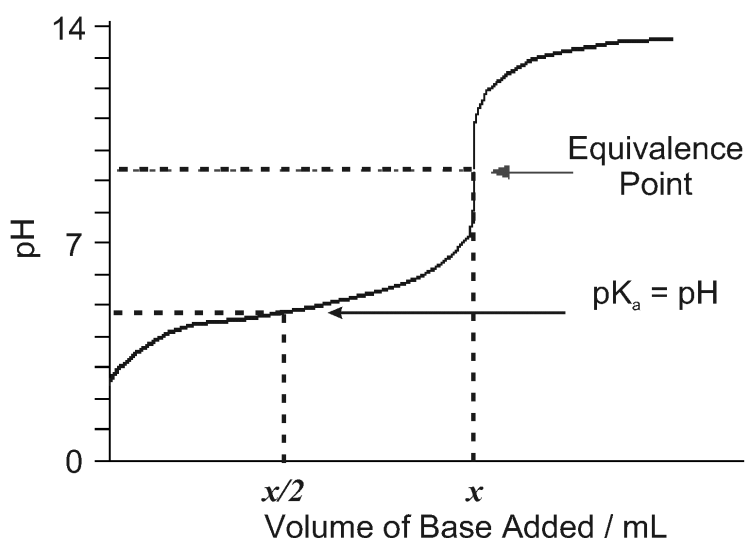


Figure 5.3: Titration of a weak acid with a strong base.

Note in Figure 5.3 the following points: (a) initial reading, pH ~ 3, (b) equivalence point, where the slope of the curve is greatest, (c) $\text{pK}_a = \text{pH}$, the volume corresponding to half of the volume added at the equivalence.

1.7 Bulb pipette usage

The pipette is a precision instrument to deliver an accurately determined volume of liquid (an aliquot). Never hold a pipette by the glass bulb because body heat can cause a significant change in the volume delivered. Always hold it by the upper stem.

Rinse the pipette twice with the solution to be used in the measurements, and then draw the solution by suction to just above the mark and adjust until the bottom of the meniscus just touches the mark. Using the first finger to seal the top allows greater flexibility than using the thumb and is especially helpful when using graduated pipettes. Slowly rotate the pipette to allow accurate control of the level.

The standard procedure for draining a pipette is to hold it vertical with its tip in contact with the side of the vessel for 15 seconds after the continuous discharge

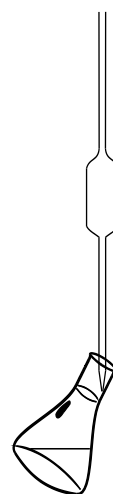


Figure 5.4: Draining a pipette into a conical flask.

has ceased (see Figure 5.4). At the end of 15 seconds, remove the pipette from the vessel. Disregard any liquid remaining in the pipette tip. Always use a pipette filler.

2. Experimental Procedure

When performing each experiment, record and graph at least 20 data points.

When graphing your data, remember to include:

- Appropriate title
- Axes labels (including units)
- Temperature at which the titration was performed
- Equivalence point/s and pK_a s (where appropriate)



GSP

Practical skills tests: Some laboratory techniques will be checked by your TA so that you can receive feedback to help you improve. Copy Table 5.1 into your laboratory notebook, and have your TA observe you complete each task.

Table 5.1: Practical skills test

Practical Skill	Satisfactory?	TA comments
Correctly fill a burette		
Accurately read a burette		
Use bulb pipette correctly		

2.1 Calibration of pH meter

Calibrate your pH meter according to the SOP provided.



SOP

2.2 Titration of ~0.1 M NaOH with ~0.1 M HCl

- a) Pipette 10.00 mL of NaOH into a 100 mL beaker and add 20 mL (measuring cylinder) of distilled water so that the electrode is immersed. Measure the pH and the temperature.
- b) Fill the burette with 0.1 M HCl (record the exact concentration of the acid) and take an initial reading to ± 0.05 mL (there is no need to start exactly at 0.00 mL).
- c) Arrange the burette, beaker, electrode and pH meter so that titration can be conducted while the electrode remains in place (see Figure 5.5).

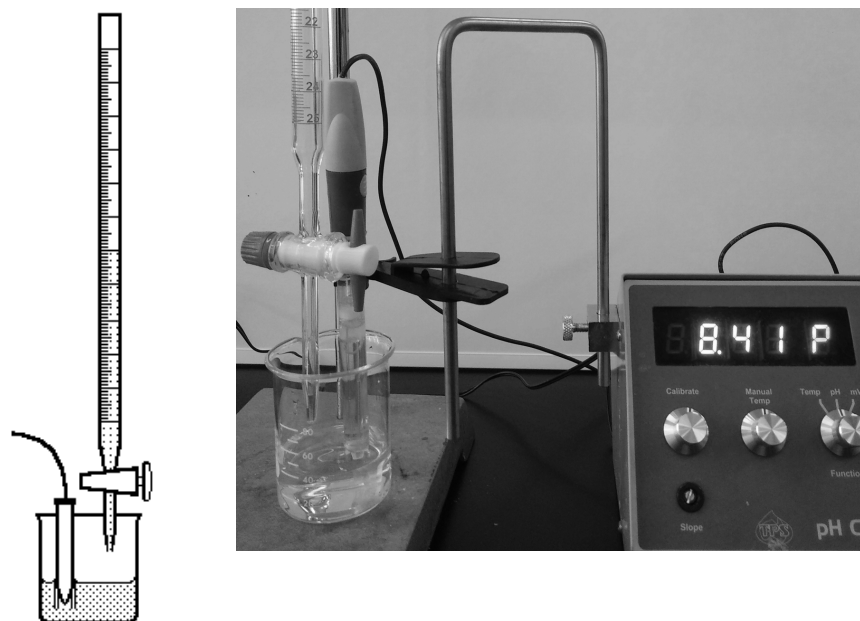


Figure 5.5: Burette set up with pH meter.

- d) Add approximately 1 mL of acid. Record the burette readings to ± 0.05 mL. Swirl the solution thoroughly and measure the pH. Do not remove the electrode or stir with it.
- e) Plot the amount of acid added against the resulting pH. The reading should be plotted as soon as it is measured. Do not record each value separately; plot the values on the graph as you go. The graph scales should be pH 0 - 14 (y-axis), volume of acid 0 - 20 mL (x-axis).
- f) Repeat steps d and e. As the pH begins to fall more steeply, the volume of acid per addition should be reduced to about 0.1 mL. If you overshoot at this stage, repeat the titration.
- g) Once you are well past the equivalence point (steepest point on the graph), use 1 mL additions again.
- h) Mark the equivalence point on your graph. Read the pH of the solution at the equivalence point, and record the corresponding volume of HCl required.
- i) Using the accurate concentration of HCl listed on the bottle, calculate the concentration of NaOH used in the experiment.

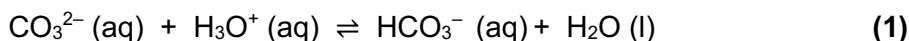
2.3 Titration of ~ 0.1 M Na_2CO_3 with ~ 0.1 M HCl

The procedure is similar to that of the titration of NaOH with HCl. However, there are two equivalence points, one after addition of approximately 10 mL of HCl, the other after addition of approximately 20 mL of HCl. A total of 25 mL of HCl should be added altogether.

- a) Make up a solution of ~ 0.1 M sodium carbonate. To do this, weigh out 0.26 g of Na_2CO_3 , and make up to 25.00 mL (in a volumetric flask) with water. Refer to Exercise 2 for notes on making up a standard solution using a solid reagent and a volumetric flask.
- b) Pipette 10.00 mL of sodium carbonate solution into a clean beaker. Add 20 mL (measuring cylinder) of distilled water so that the electrode is immersed. Measure the pH and the temperature.
- c) Titrate by addition of HCl from the burette (in a similar manner to section 2.2). Remember there are two equivalence points in this titration. Plot the titration curve as you proceed. The graph scales should be pH 0 - 14 (y-axis), volume of acid 0-30 mL (x-axis).
- d) Mark the two equivalence points on your graph.

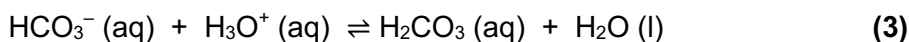
- Use the graph to determine the initial pH (solution of Na_2CO_3) and the pH of a solution of sodium hydrogen carbonate (NaHCO_3) at the first equivalence point.
- Use the graph to determine the pH of a saturated solution of carbon dioxide (the second equivalence point).

The first part of the titration can be described approximately by equation (1) and the graph follows equation (2) where $\text{p}K_{a2} = -\log_{10}$ (acid dissociation constant of HCO_3^-).



$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{CO}_3^{2-}]}{[\text{HCO}_3^-]} \quad (2)$$

- Find the value of pH at the point where $[\text{HCO}_3^-] = [\text{CO}_3^{2-}]$, and hence determine an approximate value of $\text{p}K_a$ for the hydrogen carbonate ion ($\text{p}K_{a1}$ for carbonic acid.).
- Between the first and second equivalence points the reaction can be described approximately by equation (3) and the graph follows equation (4) where $\text{p}K_{a1} = -\log_{10} K_{a1}$ (acid dissociation constant of H_2CO_3).



$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{HCO}_3^-]}{[\text{H}_2\text{CO}_3]} \quad (4)$$

- Find the value of pH at the second halfway point (where $[\text{H}_2\text{CO}_3] = [\text{HCO}_3^-]$) and hence determine an approximate value of $\text{p}K_a$ for carbonic acid ($\text{p}K_{a1}$).
- Compare the calculated mean of $\text{p}K_{a1}$ and $\text{p}K_{a2}$ with the observed pH at the first equivalence point.

Clean-up

Thoroughly clean all your glassware with warm water. Make sure the pH electrode is thoroughly rinsed and stored in a beaker of fresh deionised water.



3. Report

Complete your laboratory report online, via Moodle. Ensure you perform all necessary calculations, complete all sections, and upload your graphs and laboratory notes to Moodle.

Your online report must be submitted within seven days of the completion of your class.

Exercise 6: Timing the Iodine Clock

Learning Outcomes:



1. Explore the concept of 'rate order'.
2. Design a simple experiment to determine the overall rate order of a chemical reaction.
3. Understand the effects of experimental conditions on rate, such as concentration and temperature.
4. Work in teams, delegate tasks, and optimise your time management and efficiency.

The IDEA Experiments you will come across in units such as CHM1011, BIO1011 and PHS1011 are a little different to some of the laboratory exercises you will have already done in your Science degree. These exercises will not come with a procedure provided in the laboratory manual. For some parts of this exercise, you will have to design and perform your own experiment.

You will be working in a group of 3-4 students. You need to assign roles within your group and delegate the work that has to be done.

At the end of the session, you need to have finalised all experimental work, shared the data and discussed your results with your group.

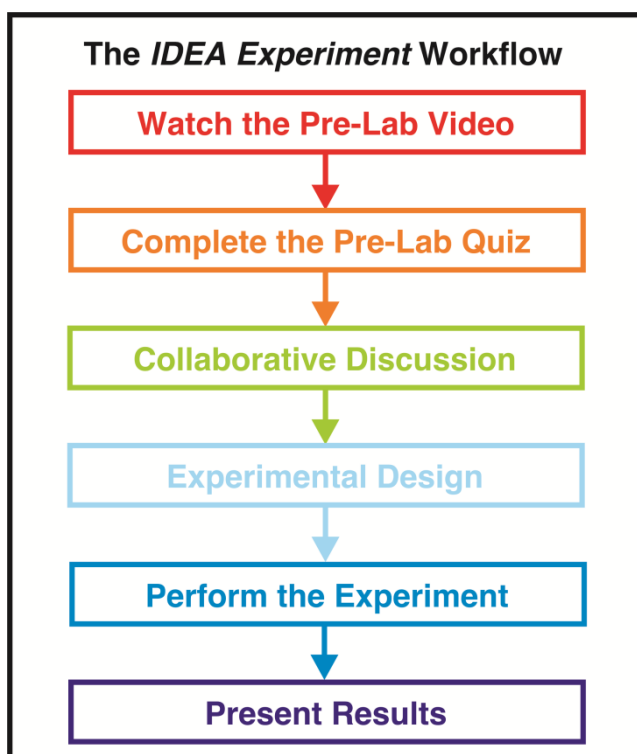
"IDEA" stands for Inquiry-Design-Explore-Answer, and that's exactly what working scientists do in their professional life.

Inquiry: A sound scientific approach to answering questions and solving problems always commences with the process of asking deep questions. What is the quickest way to perform this experiment? The most ethical? The least expensive? The 'greenest'? Are you able to foresee roadblocks along the way?

Design: Once you have considered the limitations, challenges and practicalities of the experiment, you can start to design your experiment. Weigh up what expertise and techniques you have at your disposal. Ask questions of each other, your TA, and the literature, and come up with a flow chart for how the experiment will proceed.

Explore: When you are ready to commence, delegate different jobs amongst your team, and start to gather data. You may find along the way that your design needs to be changed.

Answer: Once you have gathered the best data you can, see if you are able to form some conclusions. Discuss the results of your experiments amongst your group and determine whether the original problem has been solved.



Hazard Identification and Risk Assessment			
Identify the Hazard (the potential to do harm)	Risk (the probability that harm may result)	Control the Risk (preventing an incident)	Disposal of waste
Sulfuric acid (0.36 M) Corrosive Causes burns, damaging to eyes	Solution is dilute. Low risk.	Handle with care. Wash skin immediately under water if spill occurs.	Corrosive waste carboy in fumehood
Potassium Iodide (0.025 M) Irritating to eyes Can stain skin	Solution is dilute. Low risk Will still stain skin at this concentration	Handle with care. Rinse quickly with water if spilt on skin to avoid staining.	Corrosive waste carboy in fumehood
Thiosulfate (0.0025 M) Irritating to eyes, & skin	Low	Avoid contact with skin, wash immediately with water if spill occurs.	Corrosive waste carboy in fumehood
Hydrogen Peroxide (0.8 M) Harmful if swallowed Irritating to eyes and skin	Low	Avoid contact with skin as it can have a bleaching effect, wash immediately with water if spill occurs.	Corrosive waste carboy in fumehood
General glassware Cuts	Medium	Handle with care, dispose of broken glass using dustpan and brush. If cut occurs, see your TA and seek first aid.	Labelled broken glass bin.

Background: Workbooks, Weeks 11 and 12; Chemistry: Atoms First (2019), Chapter 17; Blackman et al., Chemistry (2019), Chapter 15.

1. Introduction

Kinetics is the study of how quickly chemical reactions occur. Some chemical reactions occur very quickly (for example explosions), while others proceed very slowly. Understanding the kinetics of a reaction gives information about the mechanism of that reaction.

1.1 Determining the Rate Order

The rate of a reaction is the increase in concentration of products over time, at a particular instant in time. Alternatively, it can be defined as the decrease in concentration of reactants over a period of time, at a particular instant in time.

During today's experiment, the aim is to determine the rate order of the chemical reaction. That is, how the rate of the reaction changes as the concentration of reactants changes.

- Zero Order kinetics occurs when the concentration of a reactant $[A]$ is increased, and the rate of the reaction doesn't change.

$$\text{Rate} \sim [A]^0$$

- First Order kinetics occurs when the rate of the reaction increases proportionally with concentration. If the concentration of one reactant is doubled, the rate of the reaction doubles.

$$\text{Rate} \sim [A]^1$$

- Second Order kinetics occurs when the rate of the reaction increases proportionally with concentration. If the concentration of one reactant is doubled, the rate of the reaction quadruples (2^2).

$$\text{Rate} \sim [A]^2$$

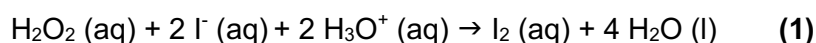
A general expression for the rate of reaction is written as:

$$\text{Rate} = k[A]^x[B]^y$$

In this general rate equation, A and B might be reactants (for example), while x and y are the order of the reaction with respect to reactant A and B respectively. The rate constant is k and the overall reaction order is x + y.

1.2 The Iodine Clock Reaction

The following chemical equation describes the “Iodine Clock Reaction” (1).



Hydrogen peroxide (H_2O_2) is added to a mixture of potassium iodide and sulfuric acid. This process occurs faster or slower depending on the concentration of each reactant. Using the rate of formation of iodine (I_2) you can determine the rate law of the reaction.

1.3 Measuring the Rate

This is done by means of the reaction between iodine (I_2) and a (relatively) small amount of thiosulfate ion ($\text{S}_2\text{O}_3^{2-}$) to form the tetrathionate ion ($\text{S}_4\text{O}_6^{2-}$) as shown in reaction (2).



When hydrogen peroxide is added to the mixture, the iodine formed by reaction (1) reacts instantly with thiosulfate by reaction (2), until all the thiosulfate is used up. The excess iodine begins to accumulate in solution and in the presence of starch forms a complex, which turns the colourless solution turns blue (Figure 6.1).

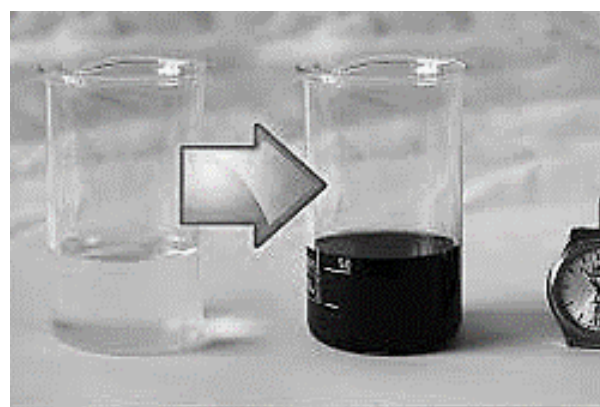


Figure 6.1: Colour change from the iodine clock reaction.

2. Experimental Procedure

2.1 The Aim

The aim of today's experiment is to determine the rate law for the iodine clock experiment. Based on the four reagents in this process, the rate law has the general form:

$$\text{Rate} = [\text{S}_2\text{O}_3^{2-}]^w [\text{H}_2\text{O}_2]^x [\text{I}^-]^y [\text{H}_3\text{O}^+]^z$$

You need to determine w, x, y and z.

Note that there needs to be a large excess of the reactants H_2O_2 , I^- and H_3O^+ for reaction (1), relative to the thiosulfate used for reaction (2).

Some questions you might consider:

- How does the rate change as $[\text{H}_2\text{O}_2]$, $[\text{I}^-]$, and $[\text{H}_3\text{O}^+]$ are increased? Can you test these simultaneously, or do you have to change only one concentration at a time?
- How does the rate change as $[\text{S}_2\text{O}_3^{2-}]$ is increased?
- How does the rate change as temperature changes?

2.2 Demonstration

Watch your TA demonstrate how to carry out this experiment. Here is a basic description of the procedure:

The following materials are added to a 100 mL conical flask, in the order shown:

- 21 mL distilled water (measuring cylinder)
- 18 mL 0.36 M H_2SO_4 (measuring cylinder)
- 5 mL 0.025 M KI (pipette)
- 5 mL 0.0025 M thiosulfate (pipette)
- 0.5 mL starch solution (measuring cylinder)

At this point, a stop-watch or timer should be ready to measure how long it takes for the colour to change. Finally, add:

- 0.5 mL of 0.8 M hydrogen peroxide (pipette, fumehood).

Start the timers as soon as the peroxide is added, and the mixture is swirled gently 3 or 4 times. The solution is left to stand until the blue colour appears. Determine the time elapsed (in seconds).

There are a few important points you should consider.

- Is temperature important? If so, can you measure it?
- Repeat the first experiment (initially performed by your TA) three times to verify the timing.
- When following the instructions above, what is the total volume of the mixture? Do you think this is important?

2.3 Inquire → Design → Explore → Answer

Once your TA has completed the demonstration, you will complete the experiment in your group. It is time to consider how you might design an experiment to determine the overall rate order for the experiment.

In your laboratory book, provide the method you plan to perform in order to determine the rate law for the iodine clock experiment. Your TA must check this before you can go on. They will provide you with feedback to help improve your experimental design if needed.

Complete this experiment, recording all data in your laboratory notebook. Use Section 3 Hints and Tips to guide you.

There is a lot to do, so delegate your jobs accordingly.

3. Hints and Tips

3.1 Results

Copy Table 6.1 into your laboratory notebook to tabulate your data:

- Complete the first row using the values determined in the pre-laboratory questions.
- Calculate the number of moles used/produced, based on the total volume.

- Note the time taken for the colour change to appear.
- Divide the number of moles of iodine produced by the time, to determine the reaction rate.
- Ensure you reproduce the same clock reaction as your TA for comparison.

Graphing your data:

- For each of the variables investigated, plot a graph of reaction rate vs concentration in Excel.
- Don't assume there will be a straight line, or even a gradient. This will depend on the reaction order kinetics.
- Ensure you use the appropriate scale for each graph.

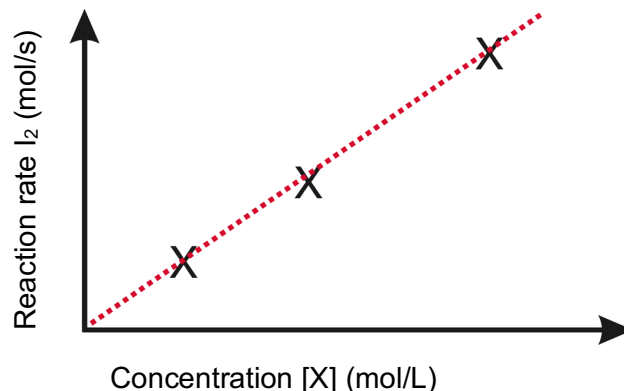


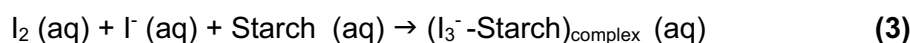
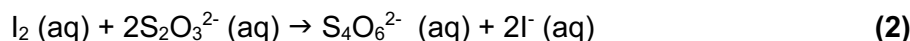
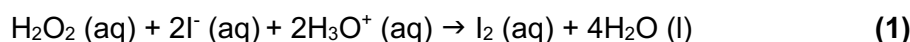
Figure 6.2: Example reaction rate vs concentration graph.

Interpreting your results:

- Use the appearance of your graphs to determine the rate order with respect to $[\text{H}_2\text{O}_2]$, $[\text{I}^-]$, $[\text{H}_3\text{O}^+]$ and $[\text{S}_2\text{O}_3^{2-}]$.
- Therefore determine the rate law for the reaction

3.2 Understanding the Reaction

Below are the reactions occurring in the experiment:



During your practical today you are determining the rate law for reaction (1). In your group, discuss the purpose of reactions (2) and (3). With respect to hypothetical scenarios 1 and 2, discuss what you would observe at the macro and sub-micro level.

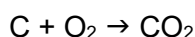
- Scenario 1: No thiosulfate is added to the flask
- Scenario 2: Neither thiosulfate nor starch is added to the flask

Table 6.1: Iodine clock data.

Volume H ₂ O added (cm ³)	[H ₃ O ⁺] _{final} & Vol _{initial} cm ³	[I ⁻] _{final} & Vol _{initial} cm ³	[H ₂ O ₂] _{final} & Vol _{initial} cm ³	[S ₂ O ₃ ²⁻] _{final} & Vol _{initial} cm ³	# mol S ₂ O ₃ ²⁻ reacted in (2)	# mol I ₂ reacted with S ₂ O ₃ ²⁻ in (2)	Time Measured (s) (Colour Change)	Reaction Rate (I ₂) (mol/s)

Explaining the terms 'limiting reagent', and 'in excess':

- Consider the reaction between carbon, C, and oxygen, O₂, to form carbon dioxide:



The stoichiometry is a 1:1 ratio for C and O₂.

Now consider what would occur if 3 moles of C was allowed to react with 4 moles of O₂:

- 3 moles of C would react with 3 moles of O₂ to form 3 moles of CO₂.
- There would be 1 mole of unreacted O₂ remaining.

In this case, carbon was the limiting reagent, and oxygen was in excess.

Calculate the initial concentrations of reagents present in the flask (ignoring the starch).

Determine the empirical ratios for each reagent, based on reaction (1).

- Based on your calculations, is hydrogen peroxide (H₂O₂) the limiting reagent, or in excess? Why?

Now consider the moment when the blue colour change is about to occur (i.e. when there is no more thiosulfate remaining in solution).

Based on the initial concentrations of reagents, calculate the concentrations of the reagents present in the flask at the moment just before the blue colour change begins (ignore the starch, and the S₄O₆²⁻ which is formed as the other byproduct in this reaction).

- Does reaction (1) continue beyond this point? Why or why not?
- Based on your calculations, which reagent in reaction (1) is the limiting reagent?

4. Report

Complete your laboratory report online, via Moodle. Ensure you perform all necessary calculations, complete all sections, and upload your graphs and laboratory notes to Moodle.

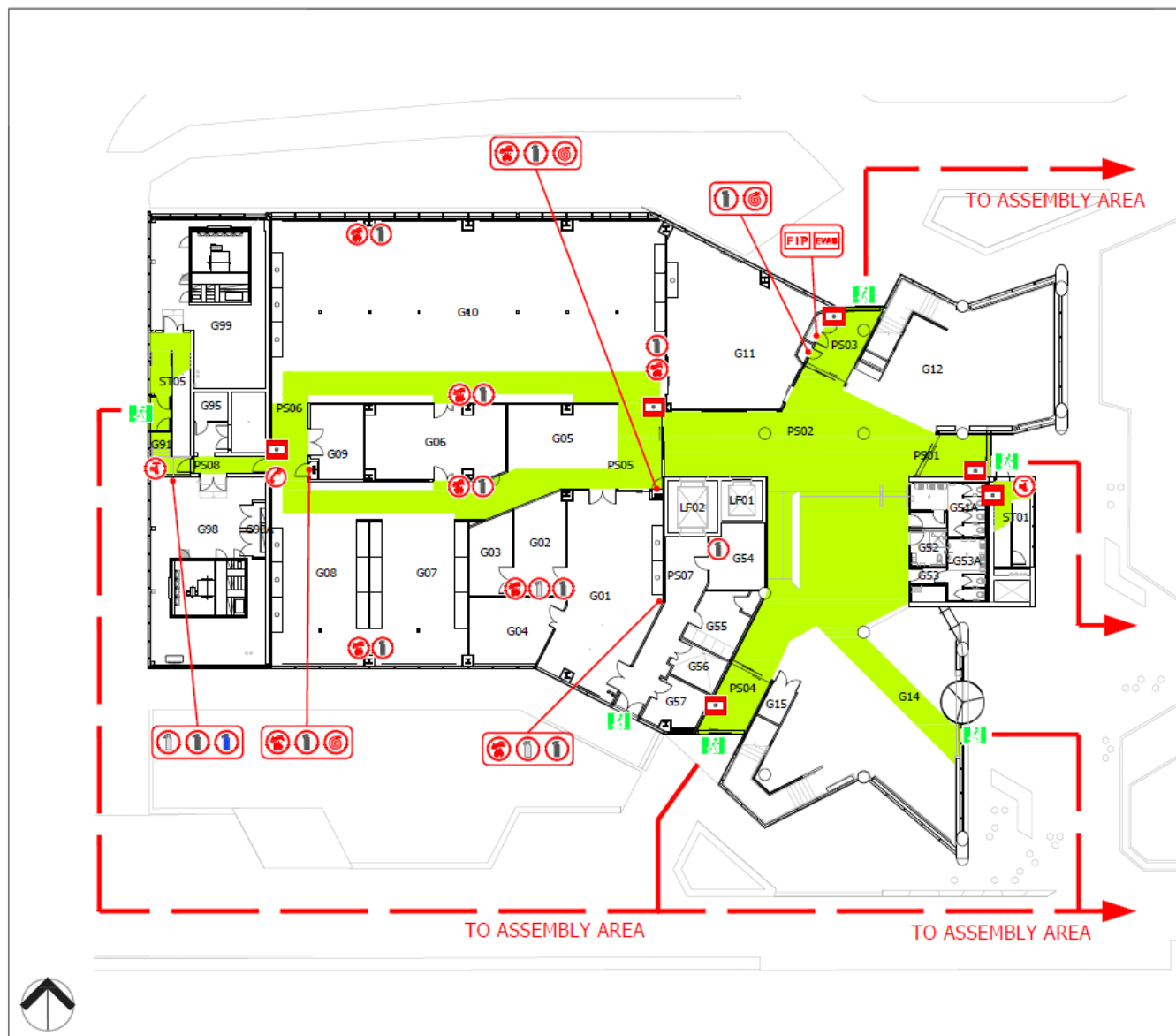
Your online report must be submitted within seven days of the completion of your class

Appendices

Appendix 1: Data Page

Equations Wave equation: $c = \nu\lambda$ Einstein equation: $E = h\nu$ Rydberg equation: $\frac{1}{\lambda} = R \left(\frac{1}{n_a^2} - \frac{1}{n_b^2} \right)$ Balmer equation: $\nu = 3.29 \times 10^{15} \left(\frac{1}{n_a^2} - \frac{1}{n_b^2} \right)$ Bond order = $\frac{1}{2} (\# \text{ bonding electrons} - \# \text{ anti-bonding electrons})$	Physical constants $c = 2.998 \times 10^8 \text{ m s}^{-1}$ $h = 6.626 \times 10^{-34} \text{ J s}$ $R = 1.097 \times 10^7 \text{ m}^{-1}$ $N_A = 6.022 \times 10^{23} \text{ mol}^{-1}$ $R = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ $= 0.08206 \text{ atm L mol}^{-1} \text{ K}^{-1}$ $1 \text{ atm} = 1.013 \times 10^5 \text{ Pa}$ $K_w \text{ at } 25^\circ\text{C} = 1.0 \times 10^{-14}$ $0^\circ\text{C} = 273.15 \text{ K}$
Gases Ideal Gas Equation: $PV = nRT$ Non-Ideal Gas Equation: $\left(p + \frac{n^2 a}{V^2} \right) (V - nb) = nRT$ Total Pressure = Σ Partial Pressures of Component Gases	
Thermodynamics $\Delta U = q + w$ $w = -p\Delta V$ $q = mc\Delta T$ $\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ$ $\Delta G^\circ = -RT \ln K$ $\Delta G = \Delta G^\circ + RT \ln Q$	
Equilibria Henderson-Hasselbach: $\text{pH} = \text{p}K_a + \log \left(\frac{[\text{base}]}{[\text{acid}]}\right)$ $E^\circ_{\text{cell}} = E^\circ_{\text{red}} - E^\circ_{\text{ox}}$	
Kinetics Zero-order reaction: $[A]_t = [A]_0 - kt$ First-order reaction: $[A]_t = [A]_0 \exp(-kt)$ Second-order reaction (only one reactant A): $\frac{1}{[A]_t} - \frac{1}{[A]_0} = kt$ Half-life: $t_{1/2} = 0.693/k$ Arrhenius equation: $k = Ae^{-E_a/RT}$ $\ln \left(\frac{k_2}{k_1} \right) = \frac{-E_a}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$	

Appendix 2: Evacuation Diagram – First Year Chemistry Labs



EMERGENCY PROCEDURES

FOR FIRE

- Activate a **Red** manual break glass alarm

FOR ALL EMERGENCIES

- Call Security on **ext 333**
- Or call **99053333**
- Or use a **Red** emergency telephone
- Assist anyone who needs help *if it is safe to do so*

EVACUATION

Be ready to leave the building:

- If danger threatens
- If instructed by staff, wardens or emergency services
- On hearing the alarms

When evacuating the building:

- Walk. Do not run. Do not use a lift
- Evacuate to the assembly area & stay there until the all clear is given by the authorised person

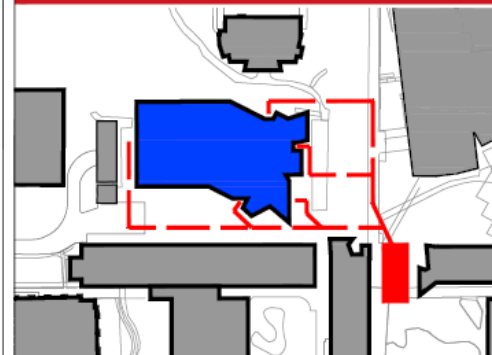
ALARMS

- **Beep-Beep** Prepare to evacuate, turn off equipment & listen for announcements over the PA system
- **Whoop - Whoop** Evacuate to assembly area

SYMBOL LEGEND

Main exit pathway	Emergency warning & intercom system	Fire hydrant
Emergency exit	Manual call point	Fire hose reel
Refuge area	Emergency call point	Fire extinguisher
Fire indicator panel	Emergency phone	Fire blanket

ASSEMBLY AREA



Appendix 3: Periodic Table of the Elements

1 IA 1A																	18 VIIIA 8A						
1 H Hydrogen 1.008	2 IIA 2A																	2 He Helium 4.003					
3 Li Lithium 6.941	4 Be Beryllium 9.012																						
11 Na Sodium 22.990	12 Mg Magnesium 24.305																	5 B Boron 10.811	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998	10 Ne Neon 20.180
		3 IIIB 3B	4 IVB 4B	5 VB 5B	6 VIB 6B	7 VIIB 7B	8 VIII 8	9 VIII 8	10 VIII 8	11 IB 1B	12 IIB 2B	13 IIIA 3A	14 IVA 4A	15 VA 5A	16 VIA 6A	17 VIIA 7A							
19 K Potassium 39.098	20 Ca Calcium 40.078	21 Sc Scandium 44.956	22 Ti Titanium 47.867	23 V Vanadium 50.942	24 Cr Chromium 51.996	25 Mn Manganese 54.938	26 Fe Iron 55.845	27 Co Cobalt 58.933	28 Ni Nickel 58.693	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.631	33 As Arsenic 74.922	34 Se Selenium 78.971	35 Br Bromine 79.904	36 Kr Krypton 83.798						
37 Rb Rubidium 85.468	38 Sr Strontium 87.62	39 Y Yttrium 88.906	40 Zr Zirconium 91.224	41 Nb Niobium 92.906	42 Mo Molybdenum 95.95	43 Tc Technetium 98.907	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.906	46 Pd Palladium 106.42	47 Ag Silver 107.868	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.711	51 Sb Antimony 121.760	52 Te Tellurium 127.6	53 I Iodine 126.904	54 Xe Xenon 131.294						
55 Cs Cesium 132.905	56 Ba Barium 137.328	57-71		72 Hf Hafnium 178.49	73 Ta Tantalum 180.948	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.085	79 Au Gold 196.967	80 Hg Mercury 200.592	81 Tl Thallium 204.383	82 Pb Lead 207.2	83 Bi Bismuth 208.980	84 Po Polonium [208.982]	85 At Astatine 209.987	86 Rn Radon 222.018					
87 Fr Francium 223.020	88 Ra Radium 226.025	89-103		104 Rf Rutherfordium [261]	105 Db Dubnium [262]	106 Sg Seaborgium [266]	107 Bh Bohrium [264]	108 Hs Hassium [269]	109 Mt Meitnerium [278]	110 Ds Darmstadtium [281]	111 Rg Roentgenium [280]	112 Cn Copernicium [285]	113 Nh Nihonium [286]	114 Fl Flerovium [289]	115 Mc Moscovium [289]	116 Lv Livermorium [293]	117 Ts Tennessine [294]	118 Og Oganesson [294]					

Lanthanide Series	57 La Lanthanum 138.905	58 Ce Cerium 140.116	59 Pr Praseodymium 140.908	60 Nd Neodymium 144.243	61 Pm Promethium 144.913	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.925	66 Dy Dysprosium 162.500	67 Ho Holmium 164.930	68 Er Erbium 167.259	69 Tm Thulium 168.934	70 Yb Ytterbium 173.055	71 Lu Lutetium 174.967
	89 Ac Actinium 227.028	90 Th Thorium 232.038	91 Pa Protactinium 231.036	92 U Uranium 238.029	93 Np Neptunium 237.048	94 Pu Plutonium 244.064	95 Am Americium 243.061	96 Cm Curium 247.070	97 Bk Berkelium 247.070	98 Cf Californium 251.080	99 Es Einsteinium [254]	100 Fm Fermium 257.095	101 Md Mendelevium 258.1	102 No Nobelium 259.101	103 Lr Lawrencium [262]