

# First Year Undergraduate Chemistry Laboratory Course Manual 2011-2012

## Core Chemistry 1A and 1B: Skills Block 2

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# Core Chemistry 1A and 1B

## First Year Chemistry Laboratory Course Manual 2011-2012

### SKILLS BLOCK 2

Name .....

Core Chemistry 1A session:

Day/Time: ..... Group name: .....

Core Chemistry 1B session:

Day/Time: ..... Group name: .....

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7. The fire action signs in the laboratory indicate the nearest fire alarm and the emergency exit. There are two carbon dioxide fire extinguishers on either side of the central pedestal and another in the instrument room. There is also a foam spray fire extinguisher on either side of the central pedestal and one at each fire exit. A general fire practice is held twice yearly to check the smooth operation of the procedure so you should ensure that you know where to go in an emergency.
8. Pipetting by mouth is not allowed. Use a bulb or automatic pipette.
9. Do not inhale vapours or make skin contact with any substances. Use gloves where necessary always remembering that they are semi-permeable.
10. Experiments must be conducted on clean working surfaces; any spillage should be cleaned immediately. A high standard of tidiness should be maintained at all times. Contaminated surfaces and equipment must be cleaned as soon as it is practicable after use. The equipment should then be put away. Do not clutter bench-space with unused equipment and bottles of chemicals.
11. Waste should be disposed of in the appropriate containers: solvents should be placed in either C, H, N, O-containing waste solvent bottles (Category C Waste), or halogen, sulphur-containing waste solvent bottles (Category D Waste). Heavy metal waste should be placed in the appropriate bottle. Broken glassware should be washed and placed in the designated glass bin. Solid waste should be dried, placed in a polythene bag and placed in a solid waste bin. A sharps bin is located in CG195. Consult a demonstrator if you are unsure about the correct disposal procedure.
12. The COSHH assessment of any chemical you use or make will be given in the laboratory script. There are further safety warnings at the appropriate parts of the text. Staff and student demonstrators reinforce these. If you are in any doubt, consult a demonstrator.
13. No unauthorised experiments are to be carried out.
14. It is important to ensure that hands are washed and all protective clothing removed **before** leaving the laboratory.

## Introduction

Chemistry is an experimental science and, as well as attending lectures, both the University and the Royal Society of Chemistry, who accredit your degree, require you to complete a designated number of hours of laboratory work. During the first year, 18 weeks of practical work must be completed. The first year practical course is split into four sections:

1. Induction (Week 1)
2. Skills (Weeks 2-7)
3. Discovery (Weeks 8-16)
4. Projects (Weeks 18-19).

During the next three weeks, you will complete Block 2 of the Skills section. This contains activities designed to ensure that you are familiar with, and have practised, some of the key skills required in the chemistry laboratory. Some of these will be familiar to you from your pre-University studies, but this will vary from person to person. This section will also introduce the various resources you can use in your own time, away from the laboratory, to ensure you are confident in your work and make the most of your time in the laboratory.

### 1.1 The pre-lab exercises

Before every laboratory session, one or more pre-lab exercises must be completed. These may involve reading, watching video clips, answering questions or using interactive software to rehearse techniques. The instructions for these exercises will be accessed via DUO, the university Virtual Learning Environment. Becoming familiar and comfortable with the use of DUO is one of the aims of this section.

Pre-lab exercises will often contain summative aspects (i.e. the marks will count towards the overall marks for the Laboratory Course), and they must be completed in the week before you attempt the laboratory activity. All pre-lab work must be finished an hour before the relevant laboratory session so that completion can be checked. For example, a student attending the Thursday laboratory session, which begins at 2.00pm, must have completed the pre-lab exercises by 1.00pm that same day. Anyone arriving at a laboratory session without having completed the pre-lab exercises will be sent away to complete them before being allowed to begin work in the laboratory. Failure to complete the pre-lab exercises on time will incur a marks penalty. Your time in the laboratory will become very pressured if you are sent away to complete the pre-lab exercises. Good time management is the key to success in most areas of university life, but particularly in your laboratory work!

If there are any problems with access to DUO or LabSkills using personal computers, there are open-access machines available for use in the library and at other points around the science site. There may also be provision in college. Ask for help if problems arise when accessing the pre-lab exercises. Failure to access the exercises will not be accepted as a reason for incomplete pre-lab

work unless the laboratory course leader (Dr J. M. Robson) is informed in advance of the deadline so alternative arrangements can be made.

### 1.2 LabSkills

Many pre-lab exercises will involve you using LabSkills. This is an electronic laboratory textbook for you to use to gain confidence in assembling and using apparatus before you begin work in the laboratory. Interactive exercises are designed to allow you to practice key techniques and learn more about apparatus and safety as you progress through the course. During the Skills section of your laboratory course, the pre-lab exercises will be very prescriptive in their use of LabSkills. You can, however, access it at any time during your undergraduate career at Durham to refresh your memory of any basic techniques and skills before any practical class. LabSkills also contains useful glossaries and worked examples of calculations that you will find useful. It will be accessible in the laboratory for additional assistance if you need it.

### 1.3 The laboratory sessions

One laboratory session per week will be assigned to Core Chemistry 1A, and a second session per week for Core Chemistry 1B. Experiments that will count towards Core Chemistry 1A will contain a suffix of 'A' in the title (e.g. Experiment 7A) and will be carried out by everybody. Experiment titles containing a suffix of 'B' (e.g. Experiment 7B) will count towards Core Chemistry 1B and will be carried out only by those studying Core Chemistry 1B.

In this term (Michaelmas), University weeks begin on a Thursday and end on a Wednesday. Those students only studying Core Chemistry 1A will be assigned one laboratory session per week and will carry out all of the 'A' experiments. Those students also studying Core Chemistry 1B will be assigned two sessions per week. The first session of the week is assigned to be the Core Chemistry 1A session, and the second session of the week is the Core Chemistry 1B session. For example, if the two allocated laboratory sessions are on Thursday morning and Monday afternoon, the Core Chemistry 1A session will be Thursday morning, when an experiment with the suffix 'A' will be performed, and the Core Chemistry 1B session will be Monday afternoon, when the 'B' experiment for that week will be completed.

Laboratory sessions will be allocated during one or two of the following times:

Thursday	9.00am - 12.00pm
Friday	9.00am - 12.00pm
Friday	2.00pm - 5.00pm
Monday	9.00am - 12.00pm
Monday	2.00pm - 5.00pm
Tuesday	2.00pm - 5.00pm
Wednesday	10.00am - 1.00pm

You may only attend the laboratory at your allocated time. A risk assessment is provided in this manual for the chemicals used in each experiment. This must be read carefully before attending the laboratory, and the advice followed throughout each laboratory session. All experimental work must be completed in that laboratory session and your lab notebook and work space signed off before you leave.

#### 1.4 Set Allocation

Students in each laboratory session are allocated to one of three named sets of no more than 20 students. Sets are named after chemical elements and students are assigned to sets in no particular order. Lists showing members of each set are available on DUO and details should be written onto the front of the laboratory manual. Each set will tackle a different activity each week, in a three week cycle, until everyone has completed each activity. The three experiments in the laboratory will then change and each set will again work through each experiment according to the rota.

Set names are as follows:

	Set 1	Set 2	Set 3
<b>Thurs am</b>	actinium	bismuth	cobalt
<b>Friday am</b>	dysprosium	erbium	fluorine
<b>Friday pm</b>	gadolinium	hafnium	yttrium
<b>Monday am</b>	potassium	lithium	molybdenum
<b>Monday pm</b>	niobium	phosphorus	osmium
<b>Tuesday am</b>	rhodium	strontium	tantalum
<b>Wednesday am</b>	uranium	vanadium	tungsten

You will already have been allocated one set for the Core Chemistry 1A session and a second set for the Core Chemistry 1B session. Set lists are available on DUO. Sets will perform experiments according to the following rota:

Week	Set 1	Set 2	Set 3
<b>5</b>	Experiment 5	Experiment 6	Experiment 7
<b>6</b>	Experiment 6	Experiment 7	Experiment 5
<b>7</b>	Experiment 7	Experiment 5	Experiment 6

For example, in week 6, everyone in Set 2 will carry out Experiment 7. Those studying only Core Chemistry 1A will only complete Experiment 7A. Those students also studying Core Chemistry 1B will complete Experiment 7B during their second session of the week. You will find your allocated sets on DUO and you should write the details onto the front of this laboratory manual. Contact Dr J. M. Robson ([j.m.robson@durham.ac.uk](mailto:j.m.robson@durham.ac.uk)) with any queries relating to sets or the experiment rota.



### 1.5 Assessment

Pre-lab exercises will contain assessed components. These exercises will differ between experiments, but assessed aspects will be highlighted. Completion of these exercises is compulsory and there will be a marks penalty for non-completion. The pre-laboratory exercise marks will make up 10% of the total marks for the practical course.

During each laboratory session, work and progress will be assessed. Completion of the lab notebook and performance in the practical tasks will be given marks. Occasionally there will be a small amount of post-laboratory work that will need to be completed to finish each experiment. Marks will be awarded during laboratory sessions throughout the year and will make up 10% of the total marks for the practical course.

In the Skills section, the focus is on the development of a good general technical skill base to be used throughout your practical work at University. There will be one small additional assessed written exercise for those students studying Core Chemistry 1B. From Week 8 onwards, in the Discovery and Projects sections, there will be additional assessed exercises for all students that will count towards your total marks for the practical course.

### 1.6 Assessment summary (bold indicates activities to be completed during Skills)

	Core Chemistry 1A	Core Chemistry 1B
<b>Pre-lab exercises (whole year)</b>	<b>10 %</b>	<b>10 %</b>
<b>Laboratory session marks (whole year)</b>	<b>10 %</b>	<b>10 %</b>
<b>SKILLS Experiment 5B: Determination of the enthalpy of vaporisation of ethanol</b>	-	<b>5%</b>
DISCOVERY Experiment 10A	20 %	-
DISCOVERY Experiment 10B	-	15 %
DISCOVERY Experiment 11B	-	20 %
DISCOVERY Experiment 12A	20 %	-
DISCOVERY Experiment 15A	20 %	-
DISCOVERY Experiment 15B	-	20 %
PROJECT	20 %	20 % *
	<b>100 %</b>	<b>100 %</b>

\*50 % of the project mark for students completing both Core Chemistry 1A and Core Chemistry 1B will be allocated towards the Core Chemistry 1A total marks, and 50 % will be allocated towards the Core Chemistry 1B total marks.

## 1.7 Supervising staff and postgraduate demonstrators

*SKILLS Block 2 – week 5 to week 7 (Thursday 3<sup>rd</sup> November to Wednesday 23<sup>rd</sup> November 2011)*

<b>Senior Demonstrators (staff)</b>	<b>Junior Demonstrators (postgraduate students)</b>
Dr Jacque Robson*	Philip Brown
Prof Mark Wilson	Rachel Carr
Dr Lars Palsson	Alex Dudgeon
Dr Andy Beeby	Becky Edwards
Dr David Carty	Ricardo Girling
Dr Pippa Coffey	Santi Marques Gonzales
	Peter Harvey

\* Laboratory course leader - email: [j.m.robson@durham.ac.uk](mailto:j.m.robson@durham.ac.uk)

**SKILLS**  
**BLOCK 2**  
**EXPERIMENT 5A**

**PREPARATION OF AN ANIONIC  
AND A CATIONIC COMPLEX OF  
NICKEL**

## 5A. Preparation of an anionic and a cationic complex of nickel

An introduction to the study of coordination chemistry is provided in the pre-lab exercise. In this experiment, two different 1<sup>st</sup> row transition metal complexes (coordination complexes) of nickel will be prepared.

### 5A.1 Aims

- To develop knowledge of fundamental aspects of coordination chemistry and ligands
- To prepare  $K_4[Ni(NCS)_6] \cdot 4H_2O$ , an anionic nickel complex with thiocyanate ligands and a positive counterion
- To prepare  $[Ni(NH_3)_6][BF_4]_2$ , a contrasting cationic nickel complex with ammonia ligands and negative counterion
- To gain more practice in synthetic chemistry and in working efficiently
- To increase familiarity with purification techniques such as recrystallisation
- To practice equation writing/balancing for systems containing transition metal complexes
- To develop spelling, punctuation and grammar abilities in the context of scientific writing

### 5A.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read your laboratory instructions through carefully and highlight unfamiliar words or apparatus.
2. In DUO, in the Core Chemistry 1A Laboratory Course section, open Foundation Chemistry LabSkills and click to begin. Click on the 'Equipment Glossary' or the 'Reagent Glossary' at the top of the screen to identify any unfamiliar words contained in your laboratory manual script. Look again over any techniques to be used in the experiment (e.g. Buchner filtration).
3. Using DUO, find and open the document entitled 'Experiment 5A: Introduction to Coordination Chemistry pre-laboratory exercise'. This Word document provides background information about coordination chemistry and should be read carefully. Note that the document contains numerous spelling, punctuation and grammar errors (but the chemistry is sound!). Save a copy of the document with an appropriate filename containing first your surname, then your first name, then the experiment number. For example, if you are called Joe Bloggs, a suitable filename would be BloggsJoe5A.doc. After reading the text thoroughly, ensure that the 'Track Changes' option in Word (which can be located in the Review tab in Word 2010) is turned on and begin to make corrections to the document. 'Track Changes'

means that all the editing can be seen clearly. To assist with this exercise and to give hints as to what to look for, read the document entitled 'Experiment 5A: Top 10 English Writing Issues' (available in DUO). There are approximately twenty errors contained within the document. Once all the appropriate changes and corrections have been made (which should all be highlighted via the 'Track Changes' tool), submit the document via DUO, making sure that the number of errors found and changes made are stated in the Comments section at the point of submission.

4. Prepare the lab notebook. Be sure that the methods for both experiments have been read in detail. Good time management and efficient working in the laboratory are essential if both syntheses are to be completed in one laboratory session. Ensure that the risk assessment has been read and notes made in the lab notebook about specific hazards. In this experiment, thiocyanate ligands will be used. Look up and record the difference between 'cyanide' and 'cyanate', and note the meaning of 'thio' in a chemical name.

### 5A.3 Risk Assessment

CHEMICAL	RISKS	SAFETY
Nickel(II) nitrate hexahydrate, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Harmful. May cause cancer. Avoid contact with skin. Contact with combustible material may cause fire	Wear suitable protective clothing and gloves.
Ethanol, $\text{CH}_3\text{CH}_2\text{OH}$	Flammable.	Keep away from sources of ignition.
Potassium thiocyanate, KSCN	Harmful by inhalation, in contact with skin and if swallowed. Irritant. Contact with acids liberates very toxic gas. Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment	Wear gloves. Avoid release to the environment.
$\text{K}_4[\text{Ni}(\text{NCS})_6] \cdot 4\text{H}_2\text{O}$	Harmful. May cause cancer. Avoid contact with skin.	Wear gloves.
Nickel chloride hexahydrate, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	Toxic if swallowed. Carcinogenic. Irritating to eyes and skin. Avoid contact with skin. Toxic in the environment – may cause long term damage.	Avoid exposure. Wear suitable protective clothing and gloves. This material and/or its container must be disposed of as hazardous waste. Avoid release to the environment.

Sodium tetrafluoroborate, $\text{NaBF}_4$	Harmful. Causes burns. Avoid contact with skin.	Wear gloves.
Concentrated ammonia	Flammable. Toxic by inhalation. Causes burns. Very toxic to aquatic organisms. Can cause irreparable damage, particularly to eyes.	Use only in fume cupboard. Wear suitable protective clothing, gloves and eye/face protection. Keep away from sources of ignition.
Dilute ammonia	Irritant. Can cause burns.	Wear gloves. Do not breathe vapour. Use in fume cupboard.
Hexaamminenickel chloride, $\text{Ni}(\text{NH}_3)_6\text{Cl}_2$	May cause cancer. Harmful. May cause sensitisation – avoid contact with skin.	Wear gloves.
$[\text{Ni}(\text{NH}_3)_6][\text{BF}_4]_2$	Harmful. May cause cancer. Avoid contact with skin.	Wear gloves.

#### 5A.4 Laboratory Activity

Work in pairs for these two experiments, and work in a fume cupboard throughout. Work efficiently but work together so that observations can be recorded by both members of the team. Good team work and time management is essential to get these experiments completed in the time available. Choose a work area with a free locker and select a fume cupboard. Write the number of these at the top of the lab notebook page for this experiment. **Show a demonstrator the lab notebook for marking and inform the demonstrator of the locker and fume cupboard number.** It is essential that you work well as a team and that all work is carried out quickly and efficiently to ensure that both syntheses are completed within the laboratory session. There are breaks in each method where time is needed to wait for recrystallization or for products to suck dry. Use this time to continue with the other synthesis. It will be difficult to prepare both products in one session if they are performed one after the other, so plan your work carefully and use all available time. Good team work is the key, and your ability to work well as a team will be assessed.

##### *Preparation of a Thiocyanato-Nickel Complex $K_4[Ni(NCS)_6] \cdot 4H_2O$*

1. **Wear gloves throughout the experiment, and work in a fume cupboard throughout.** Using an appropriate balance for synthetic work, weigh out 3.0 g of nickel(II) nitrate hexahydrate,  $Ni(NO_3)_2 \cdot 6H_2O$ , and record the mass used. Dissolve in the minimum volume of hot ethanol (less than  $5\text{ cm}^3$ ) in a small conical flask on a stirrer hotplate.
2. Weigh out 2.0 g of potassium thiocyanate, KSCN, and record the mass used. In a second small conical flask, dissolve it in the minimum volume (approximately  $5\text{ cm}^3$ ) of hot water and then add approximately  $10\text{ cm}^3$  of ethanol. Think carefully about the apparatus used to measure out these approximate volumes and use no more precision than is necessary.
3. Mix these two solutions together, with stirring and allow to cool slightly. A precipitate should form. Note all observations in the lab notebook and deduce the identity of this precipitate.
4. Remove the precipitate by vacuum filtration. Wash the precipitate twice with small amounts of alcohol, ensuring all washings are combined with the filtrate (the mother liquor). Keep the filtrate.
5. Prepare a 1:1 mixture of ethanol and water and heat it. Weigh out 3.8 g of potassium thiocyanate and record the mass used. Dissolve the solid in the minimum volume of the hot ethanol/water mixture in a conical flask.
6. Add the filtrate to the potassium thiocyanate solution. Transfer the mixture to a flat bottomed evaporating basin. Stir well and heat on a stirrer hotplate until the solvent volume

has reduced to approximately 5 cm<sup>3</sup>. **Supervise this stage carefully to avoid the solution boiling dry. Do not let the volume decrease too much or it will set solid in the next stage.**

7. Remove the solution from the heat and cool it in a cold water bath. Crystals should begin to form. If crystallisation does not begin after a few minutes, carefully re-heat the solution with stirring until the volume has reduced further, and then cool again. Repeat this process until crystals begin to form within a few minutes of cooling. Once crystals begin to form, use an ice bath to cool further.
8. Collect the product by vacuum filtration, wash quickly with two small portions (approximately 5 cm<sup>3</sup>) of cold ethanol. Suck dry. Record the mass of the product. Calculate the percentage yield (based on the mass of the nickel(II) nitrate hexahydrate used) and record it, with working, in the lab notebook. Place the sample in a clean, labelled sample bag (not a weigh bottle!). Wash up all apparatus, thinking carefully about the disposal of any waste. **Nickel salts are toxic and carcinogenic and must not be disposed of down the sink – use the metal waste disposal receptacle.** Return all equipment to the lab lockers. **Hand the sample to the demonstrator for marking.**
9. Write balanced equations for the formation of K<sub>4</sub>[Ni(NCS)<sub>6</sub>].4H<sub>2</sub>O. Draw the structure of the product, thinking carefully about what is acting as a ligand and what is acting as a counter ion. Show the charge on the transition metal complex and its shape, and the shape and charge of any counter ions. **Show the demonstrator the lab notebook for marking.**

*Preparation of an Ammine-Nickel Complex [Ni(NH<sub>3</sub>)<sub>6</sub>][BF<sub>4</sub>]<sub>2</sub>*

1. **Wear gloves throughout the experiment, and work in a fume cupboard throughout.** Using an appropriate balance for synthetic work, weigh out 1.5 g of nickel(II) chloride hexahydrate and record the mass used. Dissolve in the minimum volume of water (approximately 2.5 cm<sup>3</sup>) in a small beaker or conical flask and slowly add concentrated (0.88 density) ammonia solution (3 cm<sup>3</sup>). A large amount of violet hexaamminenickel chloride, [Ni(NH<sub>3</sub>)<sub>6</sub>]Cl<sub>2</sub>, should form. The solution can be cooled to assist the precipitation. Record observations in the lab notebook and deduce and draw the structure of the hexaamminenickel chloride salt. **Show the crystals and the lab notebook to a demonstrator.**
2. Re-dissolve the crystals by adding cold water (4 cm<sup>3</sup>) and stir well.
3. Remove any undissolved material by vacuum filtration. Keep the filtrate – this is the nickel solution required in step 5.



4. Dissolve sodium tetrafluoroborate,  $\text{NaBF}_4$ , (1.3 g) in the minimum volume of dilute ammonia (boil the solution gently for a few minutes and if any silicate impurity precipitates, filter the solution to remove it).
5. Add this  $\text{NaBF}_4$  solution slowly, with stirring, to the nickel solution from step 3, whereupon the product should precipitate. Record observations in the lab notebook.
6. Isolate the product by vacuum filtration; wash it with dilute ammonia solution until the washings are colourless, then wash with acetone and suck dry. Transfer to a pre-weighed watch glass and record the mass of the product. Write a balanced equation for the synthesis and hence calculate the percentage yield (based on the mass of the nickel(II) chloride hexahydrate used) and record it, with working, in the lab notebook. Place the sample in a clean, labelled sample bag (not a weigh bottle!). **Hand the sample to the demonstrator for marking.**
7. Wash up all apparatus, thinking carefully about the disposal of any waste. **Nickel salts are toxic and carcinogenic and must not be disposed of down the sink – use the metal waste disposal receptacle.** Return all equipment to the lab lockers.
8. Draw the structure of the  $[\text{Ni}(\text{NH}_3)_6][\text{BF}_4]_2$  product, thinking carefully about what is acting as a ligand and what is acting as a counter ion. Show the charge on the transition metal complex and its shape, and the shape and charge of any counter ions. Write down how this nickel complex compares and contrasts to  $\text{K}_4[\text{Ni}(\text{NCS})_6] \cdot 4\text{H}_2\text{O}$ . Note down why the product isolated from aqueous solution was washed with acetone. **Show your answers to the demonstrator. Have the tidy work space, fume cupboard and laboratory locker signed off by the demonstrator.**

**SKILLS  
BLOCK 2  
EXPERIMENT 5B**

**DETERMINATION OF THE  
ENTHALPY OF VAPORISATION  
OF ETHANOL**

## 5B. Determination of the enthalpy of vaporisation of ethanol

The vapour pressure ( $p$ ) of a liquid *whose vapour is ideal* varies with temperature according to the following equation (the Clausius-Clapeyron equation):

$$\ln p = \frac{-\Delta_{\text{vap}}H}{RT} + \text{constant}$$

where  $\Delta_{\text{vap}}H$  is the enthalpy of evaporation, and  $T$  the temperature in K. Therefore a plot of  $\ln p$  against  $T^{-1}$  should be linear with a negative gradient. From the gradient,  $\Delta_{\text{vap}}H$  may be calculated using the correct value for the gas constant.

### 5B.1 Aims

- To determine the enthalpy of vaporisation of ethanol and its standard error using the variation of its boiling point with pressure.
- To use a trolley pump and vacuum distillation apparatus to collect a data set to allow determination of the enthalpy of vaporisation of ethanol.
- To become familiar with the use of liquid nitrogen as a cold trap.
- To use Excel to produce a graph and to perform linear regression on the data.
- To produce a brief written report, using Excel and Word, to summarise the analysis and findings of the experiment.

### 5B.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read the instructions in the laboratory manual through carefully and highlight unfamiliar words or apparatus.
2. In DUO, in the Core Chemistry 1A Labs section, open Foundation Chemistry LabSkills and click to begin. Click on the 'Equipment Glossary' or the 'Reagent Glossary' at the top of the screen to identify any unfamiliar words contained in the laboratory manual.
3. In LabSkills, select the Distillation option from the menu and work through the content, including the Technique, the Video and the Safety sections. If this will be the first time a distillation has been performed, ensure this preparation is carried out thoroughly.

- Using the Chemistry Interactive Lab Primer (<http://chem-ilp.net>), click on the Lab Techniques tab and select Distillation from the menu. Look through the advice given for Simple Distillation, then pay extra attention to the Vacuum Distillation section.
- Ensure that that access is available to a version of Excel to enable the completion of the written report. Install the Data Analysis Toolkit (following the instructions given in Appendix 1) if it is not available. Excel is now able to perform linear regression on the data set produced in this experiment. Linear regression is a mathematical approach to determining the best fit line for a data set. Practice performing linear regression on the example data set provided on page 22.
- This experiment will use liquid nitrogen as a cold trap. Research the properties of liquid nitrogen, find out how it is produced industrially, the boiling point and details of at least two industrial uses of it. Find out how and why liquid oxygen can form when using liquid nitrogen in the laboratory, and why it is hazardous. Make a note of your findings in an appropriate place in your lab notebook.
- Prepare the lab notebook for the experiment, including a brief summary of the findings about liquid nitrogen, a link to the method to be used (or an expanded method, if preferred) and construction of the results table. [Hint: The equipment will be adjusted to give at least 12 different pressures, and the temperature at which the liquid boils will be recorded at each of these different pressures.] Look up and make a note of the normal boiling point of ethanol.

### 5B.3 Risk assessment

CHEMICAL	RISKS	SAFETY
Liquid nitrogen	Causes severe burns. Risk of asphyxiation in confined areas.	Avoid contact with skin and eyes. Wear suitable protective clothing and gloves. Use in a well-ventilated area.
Ethanol, CH <sub>3</sub> CH <sub>2</sub> OH	Flammable.	Keep away from sources of ignition.

**This experiment has more than the usual potential hazards. Please follow the instructions carefully and wear your safety glasses at all times. IF IN DOUBT, ASK!**

Liquid nitrogen is used to trap any ethanol before it gets into the vacuum pump. Handle liquid nitrogen carefully, avoid splashing it on to yourself or others - it can cause burns. Do not seal any vessel containing liquid nitrogen, it is boiling continuously and a catastrophic pressure build can ensue. Liquid nitrogen is not dangerous if allowed to evaporate, but can be absorbed into material, and when in contact with skin can cause burns.

**Remove all jewellery from hands and wrists (including watches) for the duration of the experiment.**

#### 5B.4 Laboratory activity

Work in pairs. Make sure that the distillation apparatus is clean and then wash it out with ethanol. Put about 70-80 cm<sup>3</sup> ethanol in the distillation flask and place the apparatus in the isomantle clamping the apparatus at the condenser. Fit the thermometer and connect the lead from the vacuum system. Fill (about three-quarters full) the Dewar vessel with liquid nitrogen. With the safety tap open, turn pump on, close the safety tap (i.e. close the air intake) and place the Dewar around the trap near the vacuum pump. Introduce the Dewar to the trap slowly to minimise thermal stress. **Make sure that the system is evacuated and sealed before the liquid nitrogen is placed around the trap.** Adjust the inflow of air into the system using the screw clips and the fine needle valve until the pressure is about 950 mbar. Switch on the isomantle and record the steady temperature and pressure once the ethanol has been boiling for a few minutes. Then reduce the pressure slowly (by reducing the air inflow) to about 850 mbar and re-determine the steady boiling point. Repeat until you have at least 12 readings approximately in the range 950 to 300 mbar. Each member of the pair should produce a graph of the results, by first calculating and then plotting  $\ln(p)$  against  $T^{-1}$ , on graph paper and this should be stuck into the lab notebook. **Show a demonstrator the results.**

At the end of the experiment, switch off the heater. Remove the Dewar flask carefully, and slowly discard the liquid nitrogen by pouring on to the floor. Allow at least 10 seconds for the trap to warm, let air into the system until it is all at atmospheric pressure and then switch off the pump. Disconnect the apparatus, discard the ethanol into the solvent C waste Winchester in the fume cupboard and put all glassware away. **Have demonstrator sign off the tidy workspace.** Before leaving the laboratory, ensure that all queries about the production of the laboratory report and the analysis have been answered. It is expected that most students will complete the practical work well within the three hours allocated for the experiment and that the post-laboratory exercise can be carried out immediately afterwards.

#### 5B.5 Post-lab exercise - production of a brief laboratory report

Once the practical work is completed, the results should be analysed and a brief report produced. Guidelines for the production of the report are given below. It is expected that the report will be written immediately after the laboratory work is completed, within the time of the usual laboratory session. **The report must be completed and submitted via DUO within 24 hours of the end of the laboratory session.** Reports should be produced independently. A copy of the report should be printed and stuck into the lab notebook. The analysis for this experiment should be completed using Excel on a computer.

Plot  $\ln p$  against  $T^{-1}$  in Excel. Make sure that only your raw data is input into Excel and use the spreadsheet to perform the calculation of  $\ln p$  and  $T^{-1}$ . Note that the temperature must be an

absolute temperature (i.e. in Kelvin). Be sure to use Excel carefully to produce a clear, labelled graph and use linear regression (explained below) to produce a best fit line and determine the associated errors. Calculate  $\Delta_{vap}H$  from the gradient of the line and also determine the normal boiling point of ethanol.

Notes on use of Excel are available in Appendix 1 on page 22. Use Excel to calculate the standard errors on your gradient and intercept. Compare your value of the enthalpy of vaporisation of ethanol with the value from the literature looked up as part of the pre-lab exercises. Be sure to reference the origin of this literature value.

### 5B.6 Report guidance

Reports should be prepared using Microsoft Word and Excel, should be brief (no more than 2 A4 side including graphs and tables) and should be submitted electronically via DUO within 24 hours of the end of the laboratory session. A paper copy of your submission should be stuck into the lab notebook to follow the notes from the experiment.

Be sure to include:

1. Experiment title and reference, your name, laboratory partners' names, experiment date.
2. Titled and fully labelled Excel-produced graphs. Note that tabulated data should be included in an appropriate format, as recorded. Data should also be presented graphically. Give each graph a Figure caption (e.g. 'Figure 1: A graph to show...'). Microsoft Word can insert these automatically. If you need help in producing graphs using Excel, see the University CIS 'Introduction to Excel 2007: Simple Formulae and Charts' document (Chapters 6, 7 and 8). This can be found at <http://www.dur.ac.uk/resources/its/local/er-guides/ER004.pdf>. This link is available via DUO.
3. A brief comment on your final results, giving reference to tables and figures (e.g. Figure 2 shows the dependence of volume on pressure for...). Be sure to include the values for  $\Delta_{vap}H$  and the boiling point of ethanol determined experimentally.
4. A brief narrative statement of the outcome of the linear regression error analysis (i.e. give the gradient and/or intercept and their errors) and the source of errors. Do not include a copy of the full regression analysis output from Excel.

Feedback on your submission will be provided via DUO. Remember that this submission will count 5% towards your total practical mark for Core Chemistry 1B. Failure to submit the work by the deadline will result in a mark of zero being awarded unless the appropriate procedures have been followed (see p39 of the First Year Handbook).

### 5B.7 Appendix 1 - Linear regression analysis with Excel

Excel can be used to determine lines of best fit to data that fits a straight line (after appropriate manipulation). This can be done simply using the 'add trend line' function. However, this does not determine errors associated with the gradient and the intercept. This can be done using the 'data analysis' tools available with Excel. First, ensure that the data analysis tools are available. If they are not, install them by following the instructions below:

#### *Excel 2003*

Click on the 'add-ins' option of the Tools menu, and then select the 'Data Analysis ToolPak' box. The data analysis tools will then be available under the 'Tools' menu option.

#### *Excel 2007*

Click the Microsoft Office Button (top left corner - the four coloured logo), and then click Excel Options.

Click Add-ins, and then in the Manage box, select Excel Add-ins.

Click Go.

In the Add-Ins available box, select the Data Analysis ToolPak check box, and then click OK.

The data analysis tools will then be available under the 'Data' menu option.

#### *Excel 2010*

Click the File tab and click Options.

Click Add-ins on the left hand menu. In the Manage drop down menu at the bottom of the page, select Excel Add-ins.

Click Go.

In the Add-Ins available box, select the Data Analysis ToolPak check box, and then click OK.

The data analysis tools will then be available under the 'Data' menu option.

Steps 1-4 should be sufficient on a university network computer. If Data Analysis ToolPak is not listed in the Add-Ins available box, click Browse to locate it. If you are prompted that the Analysis ToolPak is not currently installed on your computer, click Yes to install it. It is possible that it will ask for your MS installation disk, if you don't have this, you will be unable to install the Analysis Toolpak on your PC/laptop, in which case, use a university network PC. Once installed, use of the linear regression is identical for 2003, 2007 and 2010 versions of Excel.

In order to use the linear regression software, highlight the columns containing the data to be fit (including the headings), open the Data Analysis dialogue box, and select the Regression option. From the box that opens, clicking on OK should produce the required analysis which may be tabulated on a new sheet. A large amount of information is presented, as can be seen with the example below, where analysis has been performed on an example data set. The important information is in the first part of the final table (highlighted), which gives the intercept as  $3.736 \pm 0.190475163$ , and the gradient (X variable 1) as  $0.196073 \pm 0.003069785$ . Of course, you should not

just quote these values as they appear! **Only use the appropriate number of significant figures for the result and the error, and always quote units.**

**Example of output from linear regression using Excel 2010.**

Data:

x	y
10	5.71
20	7.39
30	9.65
40	11.49
50	13.85
60	15.33
70	17.83
80	19.77
90	21.04
100	23.14

**SUMMARY OUTPUT**

<i>Regression Statistics</i>	
Multiple R	0.999021
R Square	0.998043
Adjusted R Square	0.997798
Standard Error	0.278827
Observations	10

**ANOVA**

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	317.1672436	317.1672436	4079.607666	4.01496E-12
Residual	8	0.621956364	0.077744545		
Total	9	317.7892			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>
Intercept	3.736	0.190475163	19.61410576	4.74773E-08
X Variable 1	0.196073	0.003069785	63.8718065	4.01496E-12

	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	3.296763486	4.175236514	3.296763486	4.175236514
X Variable 1	0.18899379	0.203151665	0.18899379	0.203151665



**SKILLS**  
**BLOCK 2**  
**EXPERIMENT 6A**

**DATA ANALYSIS TOOLKIT:  
ERROR PROPAGATION, LINEAR  
REGRESSION, DATA  
TRANSFORMATION AND  
ERRORS**

## 6A. Data analysis toolkit: error propagation, data transformation, linear regression and errors.

In Experiment 2A, an experiment was designed to collect some data about how much water a paper towel soaked up. This generated 10 values which were analysed using simple statistic tools such as mean, standard deviation and standard error (the standard deviation of the mean). This experiment will allow more advanced data analysis to be practiced. The key to this experiment is to take the time to understand the activities and their application to 'real' experimental data, not just to rush through them as quickly as possible! Discuss the main issues with your group as you work through the exercises together. The skills developed in this activity will be expected to be used during later experiments in the course.

### 6A.1 Aims

- To perform an error propagation for previous experimental results
- To develop understanding of the use of Excel in data handling and data analysis
- To practice appropriate graph-drawing techniques
- To use a computer-assisted learning (CAL) package to develop understanding of common ideas about errors in measurement

### 6A.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read through the laboratory activity instructions carefully and highlight any unfamiliar words. Look these up in the following texts to ensure they are understood.
2. Re-read Gott, R. and Duggan, S. "Understanding Scientific Evidence – How to critically evaluate data" 2003, Sage Publications, London, Thousand Oaks, New Delhi. The full text of this book is available to all Durham students via the University Library web pages. Pay particular attention to the chapters 1, 7, 8 and 9. An additional (and more thorough) approach to the topics covered in this experiment can be found in Taylor, J. R. "An Introduction to Error Analysis: The Study of Uncertainties in Physical Measurements", 1997, University Science Books, Sausalito, California. Durham University library shelfmark: 530.8 TAY.
3. Read over Experiment 3A and practice the calculation that was performed to calculate the concentration of the sodium hydroxide solution so that it is familiar at the start of the

laboratory session. Note down in your lab notebook the formulae for calculating standard deviation and standard error (from Experiment 2A).

4. Prepare the lab notebook. This will include noting down the full titration results from Part 1 of experiment 3A, and making a note of the concentration of NaOH that you reported. If you did not complete experiment 3A for some reason, take the results from someone else and make a note of who the results were collected by.
5. If you are studying Core Chemistry 1B, also review your 'Errors' lecture course notes.

### 6A.3 Laboratory Activity

Working together in groups of two or three at a laboratory computer (group sizes of three are also acceptable), take the time to read through the following information, work through any problems individually using data from the lab notebook and then discuss the answers within the group. Tackle the computer-based tasks together and take turns to practice each skill. This is a group-based collaborative learning exercise so it is important that groups work together and discuss the work.

#### *Propagation of Errors*

In science, the word 'error' does not have its usual connotations of 'mistake' or 'blunder'. An error in a reading may be random (an unpredictable fluctuation in a measurement or observation) or systematic (an error introduced by an experimental flaw or miscalibration in equipment). Any recorded measurement has an 'uncertainty' associated with it.

For example, a 25.00 cm<sup>3</sup> accurate pipette may be used to measure out a volume of a liquid. Looking carefully at the glassware, the manufacturer will usually have stamped the 'uncertainty' on the side of the equipment. If the uncertainty is  $\pm 0.05$  cm<sup>3</sup>, the volume measured out will not definitely be exactly 25.00 cm<sup>3</sup>. The uncertainty in the glassware means that the volume of liquid will be somewhere between 24.95 cm<sup>3</sup> and 25.05 cm<sup>3</sup>. Thus this volume would be reported as 25.00  $\pm 0.05$  cm<sup>3</sup>. When reporting or analysing data from experiments, it is important to have an understanding of the uncertainty inherent in any data. Every measurement taken during an experiment has an uncertainty associated with it. Thus, any quantity calculated using any measurement data will have an associated uncertainty that must be reported. The generic format we use to report uncertainty is:

$$x = x_{\text{best}} \pm \delta x \text{ units}$$

where  $x_{\text{best}}$  is the 'best' estimate of  $x$  obtained from experimental data and  $\delta x$  is the uncertainty in the measurement. The units must also be included for every quantity. Uncertainty may also be reported as a percentage:

$$x = x_{\text{best}} \pm z \% \text{ units}$$

where  $x_{\text{best}}$  is the 'best' estimate of  $x$  obtained from experimental data and  $z$  is  $100(\delta x/x)$ .

$\delta x$  is a measure of precision so this should not be reported to an unrealistic precision e.g. reporting  $g = 9.82 \pm 0.0235 \text{ m s}^{-2}$ , the 3 significant figures for the uncertainty are meaningless. It would be much better to say:  $g = 9.82 \pm 0.02 \text{ m s}^{-2}$ . Don't just report the number of significant figures on the calculator. As a 'rule-of-thumb', state the uncertainty to one significant figure, then quote the value of the quantity to the same number of decimal places as the uncertainty (as shown for  $g$ , above).

In Part 1 of experiment 3A, the concentration of sodium hydroxide was calculated from an average (mean) titre value determined experimentally and thus it will have an uncertainty associated with it. The aim of this task is to determine the uncertainty in the final concentration that was reported by considering the uncertainties involved in the experiment.

The raw datum, the average titre value, used in the calculation for Experiment 3A has an uncertainty associated with it. This can be quantified by calculating the standard error that can be calculated from the titration results.

Calculate the uncertainty in the average titre value reported in your lab notebook for Part 1 of Experiment 3A (use your partner's results if you did not complete this experiment). To do this, you will need to calculate the standard error for the average titre, following the procedure used before in Experiment 2A. To recap:

1. calculate the standard deviation:

$$\sigma_x = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^n (d_i)^2} = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^n (x_i - \bar{x})^2}$$

where  $\sigma_x$  = standard deviation

$x_i$  = a single measurement

$\bar{x}$  = mean

$n$  = number of titrations

2. calculate the standard error:

$$\sigma_{\bar{x}} = \frac{\sigma_x}{\sqrt{n}}$$

where  $\sigma_{\bar{x}}$  = standard error

$\sigma_x$  = standard deviation

$n$  = the number of readings

Show the working and note the uncertainty value in the lab notebook. Report the uncertainty in the format

$$x = x_{\text{best}} \pm \delta x \text{ units}$$

where  $x_{\text{best}}$  is your average titre value for Part 1 of Experiment 3A and  $\delta x$  is the calculated standard error. Compare the values within the group and make any corrections to individual working, if required. **Show the values to a demonstrator.**

To determine any quantity,  $q$ , mathematical manipulation of the raw datum,  $x$ , is usually required i.e.  $q = f(x)$ . In Experiment 3A, the raw datum,  $x$ , is the mean titre value for the  $0.1000 \pm 0.0001 \text{ mol dm}^{-3}$  solution of hydrochloric acid). The quantity,  $q$ , that is calculated from  $x$  is the concentration of the sodium hydroxide solution,  $[\text{NaOH}]$ . Other quantities and numbers used to calculate  $[\text{NaOH}]$ , were:

- the concentration of the HCl, ( $0.1000 \pm 0.0001 \text{ mol dm}^{-3}$ ). Remember that the chemical shorthand for 'the concentration of HCl' is simply  $[\text{HCl}]$ .
- the volume of the NaOH ( $25.00 \pm 0.01 \text{ cm}^3$ ). Use  $V_{\text{NaOH}}$  to represent this quantity.
- the volume of HCl used (given by your average titre, with the uncertainty calculated above). Use  $V_{\text{HCl}}$  to represent this quantity.

Thus to calculate the uncertainty in the value reported for  $[\text{NaOH}]$ , the uncertainties in these other measurements must be taken into account, as these uncertainties will all be combined together in the calculation of  $[\text{NaOH}]$ . This procedure is known as **propagation of errors**. Follow the steps shown below to calculate the uncertainty in your result.

1. Considering the calculation that you performed to work out  $[\text{NaOH}]$ , deduce a formula to show how  $[\text{NaOH}]$  is calculated from  $V_{\text{HCl}}$ ,  $V_{\text{NaOH}}$  and  $[\text{HCl}]$ . Be sure to show the units in the formula. Help each other within the group to deduce an answer. **Show the formula to a demonstrator.**

2. The formula for the calculation of  $[\text{NaOH}]$  involves products and quotients (i.e. there are quantities both multiplied and divided by each other). This is probably the most commonly encountered type of calculation in chemistry. To propagate errors to determine the uncertainty in a final number,  $q$ , the following formula should be used:

$$\left| \frac{\delta q}{q} \right| = \sqrt{\left( \frac{\delta x}{x} \right)^2 + \left( \frac{\delta y}{y} \right)^2 + \dots + \left( \frac{\delta u}{u} \right)^2 + \left( \frac{\delta w}{w} \right)^2 + \dots}$$

where  $x$ ,  $y$ ,  $u$  and  $w$  are the quantities used in calculating  $q$  and  $\delta x$ ,  $\delta y$ ,  $\delta u$  and  $\delta w$  are the uncertainties in those values. Thus, for this example, the formula to be used to determine the uncertainty in  $[\text{NaOH}]$ ,  $\delta[\text{NaOH}]$ , is:

$$\left| \frac{\delta[\text{NaOH}]}{[\text{NaOH}]} \right| = \sqrt{\left( \frac{\delta V_{\text{HCl}}}{V_{\text{HCl}}} \right)^2 + \left( \frac{\delta[\text{HCl}]}{[\text{HCl}]} \right)^2 + \left( \frac{\delta V_{\text{NaOH}}}{V_{\text{NaOH}}} \right)^2}$$

Use this formula to calculate the uncertainty in your value for [NaOH], and report in the same way as in Exercise 1. Help each other to reach an answer, then compare your value with your partner. **Show a demonstrator your results when complete.**

*Using Excel to analyse data sets and draw graphs*

Often, when carrying out an experiment, data can be recorded directly into Excel. The spreadsheet is a powerful tool for analysing data sets. In this task, you will practice using Excel to perform calculations on data and to produce graphs. Use the Help function in Excel (press F1) to assist if you cannot work out how to do each step, or ask the demonstrator.

Below is a table of data which would, if plotted, produce a straight line graph of the form  $y = mx + c$ , where  $m$  is the gradient of the line and  $c$  is the intercept. Open Excel and enter the data exactly as it appears below:

x / units of x	y / units of y
0	-3.60
1	-1.00
2	4.30
3	7.98
4	10.64
5	14.40
6	19.05
7	21.67
8	25.27
9	29.30
10	34.06

Use Excel functions to construct columns showing the values of  $x^2$  and  $x.y$ . Determine the sums of the four columns to give  $\sum x$ ,  $\sum y$ ,  $\sum x^2$  and  $\sum x.y$  and display the values as a row at the bottom of the table. Using the formulae given on the formula sheets (available near each computer), use Excel calculate a value for the gradient,  $m$  and a value for the intercept,  $c$ , for this data. This should be done within Excel and not calculated by hand (i.e. do not use a calculator). To do this, select a new cell, and begin by typing =( ) then construct the required formula within the brackets, clicking on the relevant cells where required. **Show a demonstrator the values you have calculated, and demonstrate what happens to the values of  $m$  and  $c$  if the cell  $x=3$  is changed to  $x=3.5$ .** [Hint: if the formulae are set up correctly, changing the value of 3 to 3.5 should automatically alter the values of  $m$  and  $c$ .]

Excel has some built-in functions to determine the gradient,  $m$ , and intercept,  $c$ , called SLOPE and INTERCEPT. To use these, first select an empty cell. Click on 'Insert' and select 'Function'. Choose

SLOPE from the list of statistical functions. Select the  $x$  and  $y$  data by clicking and dragging the mouse, then click OK. The SLOPE value should now be shown on the spreadsheet. Repeat this process but select INTERCEPT from the statistical functions to determine a value for the intercept. Check that the values match the values for  $m$  and  $c$  calculated above.

Using the Chart function in Excel, draw a scatter graph of  $x$  vs  $y$  and display it as a separate sheet. Make sure that axis labels (' $y$  / units of  $y$ ' and ' $x$  / units of  $x$ ') are shown and add a title. This title should have the names of the group members in it e.g. 'Sample scatter graph produced in Excel with trendline added – Joe Bloggs and Jane Doe'. Format the points so that they show as either crosses or triangles. Format the  $y$  axis so that the number labels are displayed with no decimal places. Investigate the effect of adding and removing major and minor gridlines to the plot area, then remove all gridlines to give a plain white plot area surrounded by a border. Select the data points and right click to add a trendline. Display the equation on the trendline so that values for  $m$  and  $c$  can be seen. These should match the values calculated using the SLOPE and INTERCEPT functions above. Name the sheet in which the chart appears as 'Sample Graph'. **Show your graph to a demonstrator, print off a copy each and stick it into your lab notebook.**

#### *Using Excel to perform linear regression*

As we have seen, Excel can be used to determine the line of best fit for data that fits a straight line (after appropriate manipulation). This can be done simply using the 'add trend line' function on a graph, or by using the SLOPE and INTERCEPT functions. However, this does not determine errors associated with the gradient and the intercept. This can be done using the 'data analysis' tools available with Excel. These should be already installed on the laboratory computers. If you wish to use linear regression at a later date, however, you may need to install them on your own or a University network computer so the instructions are given in Appendix 1 (on page 33):

In order to use the linear regression software, highlight the cells containing the data to be fit (including the headings), open the Data Analysis dialogue box (in the Tools menu), and select the Regression option. From the box that opens, clicking on OK should produce the required analysis which may be tabulated on a new sheet.

Take it in turns to use Excel to generate a linear regression output using the data given above. A large amount of information should be presented, as shown below. The important information is in the first part of the final table (highlighted below), which gives the intercept as  $3.736 \pm 0.190475163$ , and the gradient ( $X$  variable 1) as  $0.196073 \pm 0.003069785$ . Of course, you should not just quote these values as they appear! Only use the appropriate number of significant figures for the result and the error, and always quote units. **Show a demonstrator that you can perform linear regression on the data set to generate the same results as are given below.**

Sample data for practising linear regression:

x	y
10	5.71
20	7.39
30	9.65
40	11.49
50	13.85
60	15.33
70	17.83
80	19.77
90	21.04
100	23.14

SUMMARY OUTPUT

<i>Regression Statistics</i>	
Multiple R	0.999021
R Square	0.998043
Adjusted R Square	0.997798
Standard Error	0.278827
Observations	10

ANOVA

	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	317.1672436	317.1672436	4079.607666	4.01496E-12
Residual	8	0.621956364	0.077744545		
Total	9	317.7892			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>
Intercept	3.736	0.190475163	19.61410576	4.74773E-08
X Variable 1	0.196073	0.003069785	63.8718065	4.01496E-12

	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	3.296763486	4.175236514	3.296763486	4.175236514
X Variable 1	0.18899379	0.203151665	0.18899379	0.203151665

Once you are happy you can use the Regression tool to determine the errors in the gradient and intercept and the values match those given in this table, try it again using the data used to produce the graph earlier and check that the values match those calculated using the trendline function, and using SLOPE and INTERCEPT. Note the gradient and intercept value with their uncertainty in the



appropriate way, paying attention to the number of significant figure. **Show a demonstrator the values with uncertainties to an appropriate number of significant figures.**

*Use of the computer-assisted learning package*

In this section, a Computer Assisted Learning Package (CAL) is used to develop and illustrate some of the concepts that are important when considering errors in measurement.

On a laboratory computer, select the '6A Errors' shortcut on the desktop, then open '6A CAL Package'. The program should then start to load. The program may flash up a warning window, warning you that the program contains macros, and ask how to proceed. Click on "Enable Macros" and the program will start to run. If any other error messages are generated, ignore them and proceed to open the package. Be sure that the 'Text' sheet is visible, enter the names of the group at the top of the sheet and work through the exercises in turn.

From time-to-time you may find that some error messages pop-up - often this is because you try to enter data into a protected cell (i.e. one that you're not allowed to change). Don't panic, click OK and carry on. If in doubt, ask a demonstrator.

At the end, print out a copy of the results sheet (with the name of you and your partner on it) and stick it into your lab notebook. **Show this to a demonstrator.**

#### 6A.4 Appendix 1 – Installing the Data Analysis Toolpak in Excel

If the Data Analysis tools are not available in Excel, install them by following the instructions below:

##### *Excel 2003*

- Click on the 'add-ins' option of the Tools menu, and then select the 'Data Analysis ToolPak' box.
- The data analysis tools will then be available under the 'Tools' menu option.

##### *Excel 2007*

- Click the Microsoft Office Button (top left corner - the four coloured logo), and then click Excel Options.
- Click Add-ins, and then in the Manage box, select Excel Add-ins.
- Click Go.
- In the Add-Ins available box, select the Data Analysis ToolPak check box, and then click OK.
- The data analysis tools will then be available under the 'Data' menu option.

##### *Excel 2010*

- Click the File tab and click Options.
- Click Add-ins on the left hand menu. In the Manage drop down menu at the bottom of the page, select Excel Add-ins.
- Click Go.
- In the Add-Ins available box, select the Data Analysis ToolPak check box, and then click OK.
- The data analysis tools will then be available under the 'Data' menu option.

Steps 1-4 should be sufficient on a university network computer. If Data Analysis ToolPak is not listed in the Add-Ins available box, click Browse to locate it. If you are prompted that the Analysis ToolPak is not currently installed on your computer, click Yes to install it. It is possible that it will ask for your MS installation disk, if you don't have this, you will be unable to install the Analysis Toolpak on your PC/laptop, in which case, use a university network PC. Once installed, use of the linear regression is identical for 2003, 2007 and 2010 versions of Excel.

**SKILLS**  
**BLOCK 2**  
**EXPERIMENT 6B**

**USE OF EXCEL TO ANALYSE**  
**LARGE DATA SETS**

## 6B. Use of Excel to analyse large data sets<sup>1</sup>

In the titration exercises in Experiment 3A, each student in the year group standardised a solution of NaOH and then determined the  $M_r$  of an unknown acid. This produced a large amount of data. It is useful to be able to analyse individual results in the context of this collective data, in the same way that a titration result may have been compared to a whole-class result previously during titration exercises. A familiar method for dealing with more than one piece of data is to calculate a simple mean. Treatment of large data sets, however, often involves a number of accepted statistical analyses other than calculating a mean (or sometimes in order to calculate a mean). The large amount of data involved means that modern spreadsheet software provides an efficient way of handling large data sets. This experiment will allow you to learn how Excel can be used for the analysis of large data sets, and to use some of the tools using data from real class results.

### 6A.1 Aims

- To perform data analysis on a large data set using Excel, including removing outliers, plotting frequency histograms and fitting of histograms to normal distributions using least-squares minimisation algorithms.
- To develop understanding of the meaning of accuracy and precision
- To critically evaluate individual experimental results in the context of the collective data

### 6A.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Review the Errors in Measurement lecture course notes, particularly the sections on the Normal or Gaussian distribution and the use of 'Normal Fitting' of data. Use the lecture notes and the two texts suggested below to develop your understanding of key concepts in data analysis, as well as looking up Normal or Gaussian distributions elsewhere if required. Suggested texts are:

(a) Gott, R. and Duggan, S. "Understanding Scientific Evidence – How to critically evaluate data" 2003, Sage Publications, London, Thousand Oaks, New Delhi. (This book is available as an e-book via the University Library web pages or via the link in DUO)

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<sup>1</sup> This experiment is adapted from Ling, C. D. and Bridgeman, A. J., *J. Chem. Educ.*, 2011, **88**, pp979-982.

(b) Taylor, J. R. "An Introduction to Error Analysis: The Study of Uncertainties in Physical Measurements", 1997, University Science Books, Sausalito, California. Durham University library shelfmark: 530.8 TAY.

2. Look up at least four different common diprotic weak organic acids and note down their structures and  $M_r$  values.

### 6A.3 Laboratory activity

#### *Analysis of the data from standardisation of NaOH*

This section will demonstrate how to fit a normal distribution to a large data set as an alternative method for determining the mean and standard deviation.

1. Working in groups of two or three, find a free computer in the laboratory and open the file named '6B Errors Standardisation of NaOH results' in Excel. This file contains an example set of data from a titration exercise similar to the one performed in Experiment 3A.
2. Save a copy of the file using 6B and the surnames of the group in the filename (e.g. 6ABloggsSmithJones.xls) onto the desktop before beginning to work with the data.
3. Sample results from an experiment by a whole class to standardise NaOH (i.e. to determine the concentration of a solution of NaOH) are given in Column B ('Values Reported'). The numbers to extract as results at the end of the analysis (the mean and the standard deviation) are in blue text. The numbers in black have been generated from the 'Values Reported' to be used to calculate the mean ( $\bar{x}$ ), the standard deviation ( $\sigma_x$ ) and to plot a frequency histogram of the results (see Figure 6B.1). Take some time to look at the data. The value of the mean (shown in blue) includes all the data (all the red numbers). Decide if there are any outliers in the data that should be removed when calculating the mean. If so, calculate a mean without including these outliers and note it. This is very simple to do using Excel: highlight the red numbers required and note the 'Average' value that appears at the bottom of the screen. To omit outliers, simply use the control key whilst dragging the mouse to highlight all data to be included and skip over the outliers.

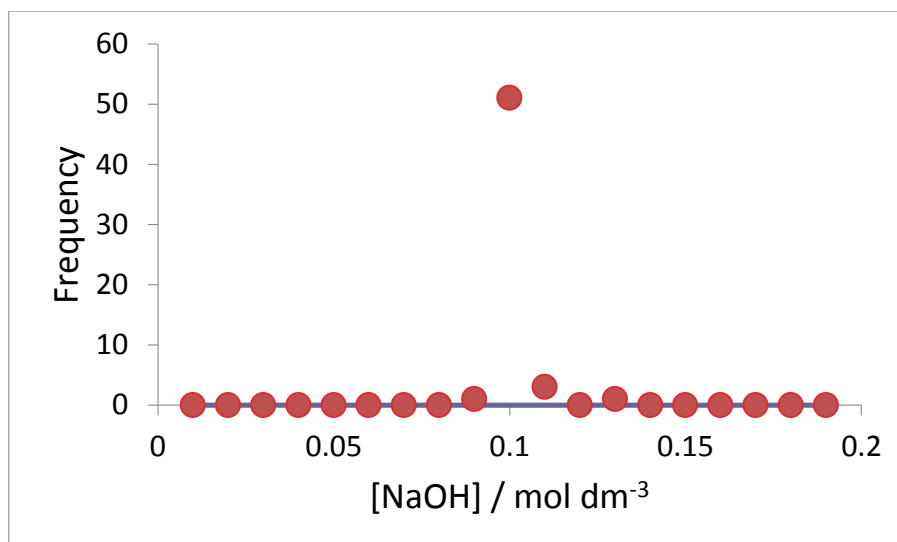


Figure 6B.1: Frequency histogram of the original data. Most data is in one bin so the defined range needs changing

- The range (in the red box) is currently defined to cover all the experimental observations of concentrations of NaOH solution. It can be seen that almost all the data are currently in a single bin in the frequency histogram (shown in Figure 6B.1) apart from extreme outliers. Double-check that the file being worked on is saved with an appropriate filename, then adjust the centre and width of the defined range (the red numbers in cells F4 and F5) to try to obtain meaningful values for  $\bar{x}$  and  $\sigma_x$ , as judged by observing changes in the frequency histogram plot. Changing these values will remove some outliers and alter how the data is presented on the histogram. The aim is to alter the values until a frequency histogram is produced that resembles a Normal (Gaussian) distribution, with the centre peak corresponding to the mean of the data.
- To ensure the centre of the data corresponds well to the mean, a normal (Gaussian) distribution curve can be fitted to the data. A normal distribution has the form  $y = ae^{-[(x-b)^2/2c^2]}$  where  $a$  is the maximum of the peak defined by the normal distribution,  $b$  is the centre and  $c$  is the standard deviation associated with the centre. The full-width at half maximum (fwhm) is  $2(2\ln 2c)^{1/2}$ . The spreadsheet automatically generates reasonable starting guesses for  $a$ ,  $b$  and  $c$  based on  $\bar{x}$  and  $\sigma_x$  for the selected range. These numbers are shown in green in cells F20, F21 and F22. Copy these numbers into the corresponding red cells in the 'Optimised values for Gaussian' box (cells F26, F27 and F28). A normal distribution should appear on the frequency histogram plot. Check that the starting guesses give a typical and sensible normal distribution curve. If not, adjust  $a$ ,  $b$  and  $c$  slightly until the normal curve fits the data. If the normal curve cannot be made to fit sensibly, go back and adjust the histogram centre and bin width and try again. Keep making adjustments until the curve looks like a normal distribution.

- The Solver function in Excel can optimise this fitting of data (by optimising the Gaussian values  $a$ ,  $b$  and  $c$ ) to obtain a good distribution. The optimisation process improves the values of  $a$ ,  $b$  and  $c$  so that the calculated frequencies  $v_{\text{calc}}$  (shown in Column K) match the experimental frequencies  $v_{\text{obs}}$  (shown in Column J) as closely as possible. The Solver function does this by minimising the value of the residual  $(v_{\text{obs}} - v_{\text{calc}})^2$  in cell F14 by changing  $a$ ,  $b$  and  $c$  in cells F26-F28. To use the function, click on Solver. In the 'Set Objective' box select cell F14 and click the 'Min' button. In the 'By changing variable cells' box, select F26-28. Then click Solve. The values in the Optimised Values for Gaussian box will now have changed slightly, as will the normal distribution curve.
- The values in the blue boxes (cells F9 and F10) are the values for the mean  $\bar{x}$  and the standard deviation  $\sigma_x$  after fitting a normal distribution. The  $b$  and  $c$  value in the 'Optimised values for Gaussian' are the mean and standard deviation determined by the Solver function. Note down the value of the mean with the one calculated at the beginning of the exercise using observation alone. **Show the demonstrator your results.**
- Now 'real' data from results obtained earlier this term by the entire class group will be used. Save a copy of the file with a different name (by adding a 2 at the end of the original filename e.g. 6ABloggsSmithJones2.xls). Delete the red numbers in the 'Optimized values for Gaussian' box (cells F26, F27 and F28) to avoid confusion. Open the file '6B NaOH Standardisation Data Autumn 2011'. This file contains the data obtained by the whole year group during experiment 3A. Copy and paste the values for the concentration of NaOH into Column B ('Values reported', the red numbers) of the file.
- Follow steps 3 to 7 to establish a value for the mean and standard deviation for the data by fitting a normal distribution curve and then by using the Solver function. How well does the data fit a normal distribution curve compared to the 'sample' data used first? How does the mean value obtained individually by each student in the group during Experiment 3A compare with the class mean and standard deviation? Discuss this within the group and note the findings.

#### *Analysis of the data from determination of the molar mass of an unknown acid*

This kind of Normal or Gaussian fitting of data can also be used in a more powerful way. In Experiment 3A, the whole class determined values for the  $M_r$  of an unknown acid by using titration. This is a typical undergraduate experiment. Results from a similar laboratory class experiment will be analysed here, but in this case there were three unknown acids available in the laboratory, Acids **A**, **B** and **C**, and each student was randomly assigned one of three acids. The whole class results varied quite significantly and there were three possible 'correct' values of  $M_r$ . There was also less data produced per acid. Trying to analyse this data by hand without knowing which student was

assigned which acid would not be a trivial task. Use of this kind of Excel data analysis and normal fitting, however, allows a simple analysis of this complicated data.

Open the file '6B Errors Mr of unknown acids' and save a copy of the file with a different name (by adding a 3 at the end of the original filename used e.g. 6ABloggsSmithJones3.xls). This file contains the sample  $M_r$  data from a real class using three different unknown acids in Column B ('Values reported', the red numbers). The centre and width of the defined range (in cells F4 and F5) have been set to include all the observed values of  $M_r$ . This frequency histogram should contain three discrete peaks corresponding to the  $M_r$  of the three different acids.

Again, roughly follow steps 3 to 7 above, analyse the results to get the optimum mean  $M_r$  and standard deviation for each of the three acids using the Solver function. This time, however, there should be three discrete ranges defined to focus on each of the three peaks (and thus the three unknown acids) individually.

Once a reasonable  $M_r$  value is determined (using the Solver function) for each acid, compare the values to those noted down as part of the pre-lab exercises to identify which three acids were used. Use the CRC Handbook available in the laboratory to help if there is insufficient data in the group to identify the acids (which are all common diprotic weak organic acids).

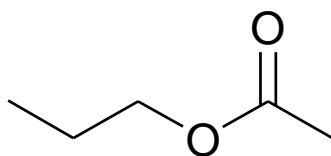
**Show a demonstrator your mean and standard deviation values for each acid, and the suggested identity of each acid.**



**SKILLS**  
**BLOCK 2**  
**EXPERIMENT 7A**

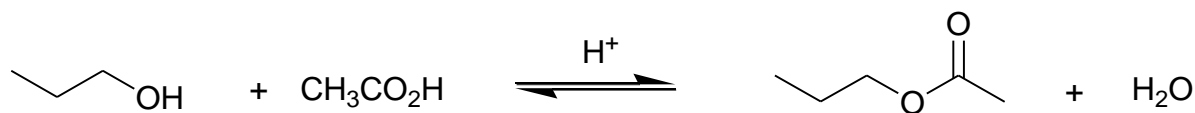
**PREPARATION OF PEAR  
ESSENCE (PROPYL ACETATE)**

## 7A. Preparation of pear essence (propyl acetate)



This ester is prepared by the well-known esterification process that should be familiar from previous studies (the reversible reaction between a C-OH containing compound and an acid to give an ester plus water). Ethanoic acid (acetic acid) is used as an acylating agent in the presence of a catalytic amount of concentrated sulfuric acid.

The esterification process is the most well-known equilibrium-controlled (reversible) reaction in organic chemistry, which is shifted to the right, in favour of the ester, by using a much higher concentration (excess) of one of the starting materials:



### 7A.1 Aims

- To refresh knowledge about use of spectra to identify unknowns from prior knowledge
- To develop a detailed synthetic and purification method from an outline procedure
- To further develop confidence with use of reflux apparatus
- To use distillation as a purification technique
- To determine a melting point of a product to determine purity

### 7A.2 Pre-lab exercises

These exercises must be completed before the experiment, and completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read the instructions in the laboratory manual through carefully and highlight unfamiliar words or apparatus.
2. In DUO, in the Core Chemistry 1A Laboratory Course section, open Foundation Chemistry LabSkills and click to begin. Click on the 'Equipment Glossary' or the 'Reagent Glossary' at the top of the screen to identify any unfamiliar words contained in your laboratory manual.

3. Using Foundation Chemistry LabSkills, look over the Reflux and Filtration (vacuum filtration –Buchner apparatus) sections to ensure familiarity with these techniques which have been used already during this laboratory course.
4. Preparing for use of a separating funnel: Using LabSkills, under the ‘Preparation and Purification’ sub-heading, select ‘Solvent Extraction’ and read the introduction about the use of a separating funnel. Watch the video demonstrating the use of a separating funnel. Use the simulation to practice using the separating funnel, and read about the drying liquids. Read through and pay close attention to the safety aspects of the procedure. It may also be useful to look through the relevant sections on the Chemistry Interactive Lab Primer (<http://chem-ilp.net>) if this technique has not been used before.
5. Preparing for the distillation: Using LabSkills, under the ‘Preparation and Purification’ sub-heading, move the cursor over ‘Distillation’ and then select ‘Technique’ from the pop-up list. Read the first page about distillation, and use the simulation on the second page to practice setting up distillation apparatus. Then select ‘Video’ and watch the procedure again, and then select ‘Safety’ and read through the safety points, focusing particularly on the use of stirrer hotplates. It may also be useful to look through the relevant sections on the Chemistry Interactive Lab Primer (<http://chem-ilp.net>) if this technique has not been used before.
6. Complete the Spectroscopy Exercise, available on DUO. Answers should be presented in a Word document, with all data tabulated with units. Submit the completed document via DUO.
7. Prepare the lab notebook. Include the mechanism for the reaction of R-OH with  $\text{CH}_3\text{CO}_2\text{H}$ , indicating clearly the role of the  $\text{H}^+$  (from  $\text{H}_2\text{SO}_4$ ), using textbooks to assist, if required. [Hint: This is a new mechanism not previously covered at pre-University level in the majority of cases.]

### 7A.3 Risk assessment

CHEMICAL	RISKS	SAFETY
Propan-1-ol	Highly flammable, irritant Risk of serious damage to eyes. Vapours may cause drowsiness and dizziness.	Keep away from sources of ignition. Avoid contact with skin. In case of contact with eyes, rinse immediately with plenty of water. Wear eye/face protection.
Glacial acetic acid (glacial ethanoic acid)	Flammable. Corrosive. Causes severe burns.	Wear gloves. Do not breathe gas/fumes/vapour/spray. In case of contact with eyes, rinse immediately with plenty of water.
Concentrated sulphuric acid	Causes severe burns. Corrosive.	In case of contact with eyes, rinse immediately with plenty of water. Never add water to this product. Wear gloves. Use in fumes cupboard.
Propyl acetate	Highly Flammable. Irritating to the eyes. Repeated exposure may cause skin dryness or cracking. Vapours may cause drowsiness and dizziness	Keep away from sources of ignition. In case of contact with eyes, rinse immediately with plenty of water. Do not empty into drains. Take precautionary measures against static discharges.
5% Sodium bicarbonate solution	Irritant.	In case of contact with eyes, rinse immediately with plenty of water.
Saturated aqueous sodium chloride	Limited evidence of a carcinogenic effect.	Avoid contact with skin and eyes.
Anhydrous magnesium sulfate	-	Do not breathe dust. Avoid contact with skin and eyes.

### 7A.4 Laboratory activity

**Show a demonstrator the prepared lab notebook to confirm completion of the pre-lab exercises.**

Work in pairs but keep individual records in lab notebooks as the experiment proceeds. Choose a work area with a free locker and a fume cupboard to work in, and write the number of these at the top of the lab notebook page for this experiment. **Inform the demonstrator of the locker and fume cupboard number.**

Work in a fume cupboard for the duration of the experiment. Think carefully about use of stoppers and bungs to prevent flammable vapours escaping into the laboratory during any transfer of liquids during the experiment.

1. In a fume cupboard pour  $12\text{ cm}^3$  (9.6 g) of propan-1-ol and  $24\text{ cm}^3$  (24 g) of glacial acetic acid into a  $100\text{ cm}^3$  round bottomed flask. Carefully add  $4\text{ cm}^3$  of concentrated sulfuric acid to the contents of the flask, with swirling. Add a magnetic stirrer bar to the mixture.
2. Assemble a reflux apparatus. **Show a demonstrator the apparatus before beginning to heat the reaction.**<sup>2</sup> Bring the mixture to the boil using a hotplate and heating block. Allow the mixture to reflux for one hour. Remove the heating source and allow the mixture to cool to room temperature. Prepare the next stages while the reaction is refluxing and cooling, and begin work on the exercises at the end of the instructions (remembering to keep the experiment under close observation). Write answers to the exercises onto the left hand page of the lab notebook.
3. In a fume cupboard, pour the cooled mixture into a separating funnel and carefully add  $50\text{ cm}^3$  of cold deionised water. Rinse the reaction flask with  $10\text{ cm}^3$  of cold water and pour the rinsings into the separating funnel. Stopper the separating funnel and shake it several times. Separate the lower aqueous layer from the upper organic layer (density = 0.87 g/ml). Only discard the aqueous layer at the end of the experiment after the pure product has been isolated.
4. Add approximately  $30\text{ cm}^3$  of 5% aqueous sodium bicarbonate (sodium hydrogencarbonate,  $\text{NaHCO}_3$ ) carefully to the organic layer contained in the separating funnel. Swirl the separating funnel gently until carbon dioxide gas is no longer evolved. Stopper and gently shake the funnel, regularly releasing the pressure, until no more  $\text{CO}_2$  is evolved. Remove the lower layer and repeat the above extraction with  $30\text{ cm}^3$  of 5% sodium bicarbonate solution. Remove the lower layer and check to see if it is basic using pH indicator paper (use a glass rod to transfer a drop of solution to a small piece of pH paper on a white tile). If it is not basic, repeat the procedure with additional  $30\text{ cm}^3$  portions of 5% base until the aqueous layer is basic.
5. Wash the organic layer with one  $25\text{ cm}^3$  portion of water, adding  $5\text{ cm}^3$  of saturated aqueous sodium chloride to aid in layer separation. Swirl the mixture gently - do not shake it! Carefully separate the lower aqueous NaCl layer and put it on one side. Pour the

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<sup>2</sup> The demonstrator will check and mark the apparatus set-up, but will also double-check that there is no stopper in the top of the apparatus. A common (and dangerous) mistake is to leave a stopper in the apparatus, sealing it, and then heating. **Heating a sealed system is very dangerous** as it can lead to explosion, so always double-check that your apparatus is not sealed before beginning to heat a reaction mixture.

remaining ester from the separating funnel into a dry 100 cm<sup>3</sup> conical flask. Add about 2 g of anhydrous magnesium sulphate to dry the ester. Stopper the flask to swirl it gently. Allow the crude ester to stand until the liquid is clear; about 15 minutes will be required in order for complete drying to occur (if the solution is still cloudy after 15 minutes, decant the liquid and add to it another 1 g quantity of drying agent.)

6. In the fume cupboard, assemble a dry B10 distillation apparatus<sup>3</sup> with B10 necked pear shaped flask (see display set-up next to the balance bench). Carefully filter the ester into the distillation flask through a fluted filter paper so that the drying agent is excluded. Add three anti-bumping granules and distil the ester, collecting the main fraction, which will have a constant boiling point, in a pre-weighed sample bottle (+ lid). Collect any liquid which distils over before the temperature becomes steady (i.e. impurities) in a separate bottle. Swap receivers when the temperature becomes steady. Note this temperature. Record the mass of the product obtained and calculate the % yield, carefully considering which starting material is yield-determining.
7. Rinse out the apparatus with acetone putting the washings into the C waste, and return all clean apparatus. Discard the basic washings down the sink in the fume cupboard. Tidy the bench space and fume cupboard and turn off all taps and plug sockets.
8. Complete any of the following exercises that have not already been completed during the experiment. **Once complete, show a demonstrator the lab notebook, the tidy fume cupboard, tidy work bench and tidy lab locker to get signed off.**

### Exercises

Write answers to the following questions, including an equation to illustrate the answer to question 2, into the lab notebook.

1. What is the good leaving group in the final stage of this reaction?
2. What reagent is used to remove the excess acetic acid, some of which will be present in the crude ester?
3. As a result of this, what has happened to the sulfuric acid?

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<sup>3</sup> Remember that 'B10' indicates the size of the glass joint on the flask. All of the glassware in the teaching lab is 'B' and is typically B10, B14, B19 or B24 in size. The numbers indicate the diameter of the joint. Thus a B10 joint is probably the one with the smallest diameter in your laboratory locker. The size is often indicated near the neck of the glassware.

**SKILLS  
BLOCK 2  
EXPERIMENT 7B**

**HYDROLYSIS OF  
METHYL BENZOATE**

## 7B. Hydrolysis of methyl benzoate

In Experiment 7A, an ester was prepared from a carboxylic acid and an alcohol using concentrated acid as a catalyst. The synthesis involved the use of reflux apparatus, followed by washing in a separating funnel and distillation to purify the product. Purity was determined by the analysis of the boiling point of the product.

This experiment will involve the reverse reaction, the hydrolysis of an ester, again using reflux, to produce an alcohol and a solid acid. The solid product will be isolated by vacuum filtration and purified by recrystallisation. The purity of the product will be determined by taking a melting point of the solid acid.

### 7B.1 Aims

- To develop a detailed synthetic and purification method from an outline procedure
- To further develop confidence with use of reflux apparatus
- To practice recrystallisation as a purification technique
- To determine a melting point of a product to determine purity

### 7B.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read the instructions in the laboratory manual through carefully and highlight unfamiliar words or apparatus.
2. In DUO, in the Core Chemistry 1A Laboratory Course section, open Foundation Chemistry LabSkills and click to begin. Click on the 'Equipment Glossary' or the 'Reagent Glossary' at the top of the screen to identify any unfamiliar words contained in the laboratory manual.
3. Using Foundation Chemistry LabSkills, look over the Reflux and Filtration (vacuum filtration – Buchner apparatus) sections to ensure familiarity with these techniques which have been used already during this laboratory course.
4. Again using LabSkills, select 'Recrystallisation' from the menu and re-read about the technique and the safety aspects, watch the video and use the simulation to practice recrystallising a solid. This method was used previously in Experiment 3A. No further instructions will be provided about recrystallisation in this laboratory manual, so use LabSkills thoroughly to prepare for the experiment.



5. Select 'Melting Point' from the main menu and read about the technique, watch the video and read the safety information thoroughly. Practice taking a melting point using the simulation. No further instructions will be provided about taking a melting point in this laboratory manual, so use LabSkills thoroughly to prepare for the experiment.
  
6. Prepare the lab notebook for the experiment. Construct the two balanced chemical equations for this experiment. [Hint: the first reaction is between methyl benzoate and sodium hydroxide. The second is between the salt produced and hydrochloric acid to liberate the benzoic acid product]. Expand on the method given in this laboratory manual to ensure that a complete and detailed set of instructions have been prepared for use in the laboratory before the laboratory session begins. This should include a more detailed method for performing the recrystallisation and the melting point determination (using LabSkills to assist), and sizes of all glassware should be determined. Diagrams of any unfamiliar apparatus should be drawn. Look up and note down the density of methyl benzoate.

### 7B.3 Risk assessment

CHEMICAL	RISKS	SAFETY
Methyl benzoate	Flammable. Irritant.	Wear suitable protective clothing.
Sodium hydroxide (2 M)	Causes burns. Corrosive. Take care especially when it is hot.	In case of contact rinse immediately with plenty of water. Wear suitable protective clothing and gloves when handling.
Hydrochloric acid (2 M)	Causes burns, irritating to eyes, skin and respiratory system.	In case of contact rinse immediately with plenty of water. Wear suitable protective clothing and gloves when handling.
Benzoic acid	Harmful, irritant.	In case of contact with eyes, rinse immediately with plenty of water.

#### 7B.4 Laboratory activity

**Show a demonstrator the prepared lab notebook to confirm completion of the pre-lab exercises.**

Work in pairs but keep individual records in lab notebooks as the experiment proceeds. Choose a work area with a free locker and a fume cupboard to work in, and write the number of these at the top of the lab notebook page for this experiment. **Inform the demonstrator of the locker and fume cupboard number.**

Work in a fume cupboard for the duration of the experiment. Think carefully about use of stoppers and bungs to prevent flammable vapours escaping into the laboratory during any transfer of liquids during the experiment.

1. Add 2 cm<sup>3</sup> of methyl benzoate, 30 cm<sup>3</sup> of 2 M sodium hydroxide solution, a few anti-bumping granules and a magnetic stirrer bar into an appropriately sized round bottomed flask.
2. Assemble reflux apparatus. Bring the mixture to the boil using a hotplate and heating block. Allow the mixture to reflux for forty five minutes. Remove the heating source and allow the mixture to cool to room temperature.
3. Pour the cooled mixture into 50 cm<sup>3</sup> of 2 M hydrochloric acid. A white precipitate should form. **Show a demonstrator the beaker containing the white precipitate.**
4. Isolate the product using vacuum filtration and recrystallise the solid in a small conical flask or beaker from hot deionised water.
5. Isolate the pure product using vacuum filtration. Dry as well as possible and record the mass of the product obtained. Calculate the percentage yield and record it, with working, in the lab notebook. [Please remember that this calculation requires you to start by calculating the number of moles of the reactants.] Place the sample in a clean, labelled sample bag (not a weigh bottle!). **Show the % yield calculation to a demonstrator.**
6. Determine a melting point of the pure solid. If time allows, a rough and two accurate readings should be taken before a final melting point is reported. **Show your value to the demonstrator to have the purity of the product assessed. Hand the demonstrator the bagged, labelled sample for assessment.**
7. Dispose carefully of any waste (ask if unsure), wash and return all equipment and ensure all power sockets and hotplates are switched off. **Show a demonstrator the completed lab notebook, the clean and tidy workspace, the clean and tidy fume cupboard and the equipment locker to get them signed off before leaving the laboratory**

## APPENDIX A: Assessment guide for laboratory reports

Reports will be assessed against the following criteria, which are not necessarily equally weighted.

	Structure	Presentation	Technical Content	Results and Discussion
<b>First Class</b>	Excellent, very clear, logical subdivision.	Well written in good English, cogent arguments presented. Conclusions concur with results obtained, results are clearly summarised.	Appropriate theoretical background included. Proper use made of theory expressions, etc.	Critical assessment of the results. Quality of sample based on data (spectra, errors). Graphs neatly plotted and correctly interpreted. Extended interpretation based on analysis of theory section.
<b>Upper Second</b>	Well organised easy to follow and a sense of direction throughout.	Clearly laid out, conclusions and summary evident and clearly written.	Good grasp of the necessary theory and its use.	Results analysed and assessed in sufficiently critical manner. Evidence of an appreciation of sources of error.
<b>Lower Second</b>	Satisfactory but some loss of way evident.	Straightforward to read, satisfactorily written, vagueness or hesitancy in conclusions and summary.	Only the basic theory behind the experiment is presented, no evidence of real understanding.	Satisfactory assessment of results and outcome of experiments. Critical evaluation not overly evident.
<b>Third</b>	No direction, no subdivision. Lack of clarity.	Somewhat disorganised and hard to read. Conclusion and summary incorrect or "off the mark".	Gaps in understanding evident from what was presented.	Poor analysis of the results or sample quality, no attempt to assess sources of error or where things may have gone wrong.
<b>Fail</b>	No evidence of any organisation, absence of basic understanding, no coherence.	Difficult to read; slap dash presentation, absence of conclusion or summary.	No real presentation of background and its appreciation.	No assessment of the results, no discussion.