Learn Chemistry

First Year Undergraduate Chemistry Laboratory Course Manual 2011-2012

Core Chemistry 1A: Skills Block 1

Developed by Dr Jacquie Robson, RSC School Teacher Fellow 2010-2011 at Durham University

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Core Chemistry 1A

First Year Chemistry Laboratory Course Manual 2011-2012

SKILLS BLOCK 1

Name ……

Core Chemistry 1A laboratory session:

Day/Time: …… Group name: ……..

Contents

Safety in the first year laboratory (CG 021)

The Health and Safety at Work Act was introduced in 1974. Since then many regulations have been made under the act, for example, The Control of Substances Hazardous to Health (COSHH). The University has a statutory obligation to comply with these requirements and you, as a student, have a duty to abide by the regulations. The following notes are to guide you in good laboratory practice and to familiarise yourself with the safety aspects of your laboratory work.

Emergency Telephone Numbers

Internal telephones:	FIRE, POLICE, AMBULANCE	9999
	UNIVERSITY EMERGENCY NUMBER	43333

1. Staff with special responsibilities for safety:

Chairman of the Board of Studies:	Professor J S O Evans
Chemistry Safety Officer:	Dr J A G Williams
Undergraduate teaching laboratories:	Dr E Wrede (Physical Chemistry)
	Dr J A G Williams (Inorganic Chemistry)
	Dr E Khosravi (Organic Chemistry)

2. No work is to be carried out unless a member of staff is present.

3. All persons in laboratories (whether or not they are actually doing practical work) must wear safety spectacles and laboratory coats. Academic staff supervising undergraduates enforce this rule. In all laboratories, hair should be secured so that it does not hang below the neck. It is important to wear suitable clothing, and your footwear must incorporate flat heels, slip-resistant soles and uppers fully enclosing the foot.

4. Foods, drinks, cosmetics and cigarettes must not be taken into or used in areas where chemical substances are used or kept.

5. Bags and coats should be placed in the lockers provided outside the laboratory and not left in corridors or on benches.

6. All accidents and dangerous occurrences must be reported immediately to a member of staff or a demonstrator. The first aid box is located in the foyer area and a list of qualified first aiders is on the front. The accident book is kept in room CG 058 and the member of staff in charge of the laboratory must fill out a report for all incidents. An emergency shower is located in the foyer area and there are four eyewash stations beside the sinks. There is a chemical spillage treatment kit in CG195.

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 six weeks, you will complete 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

You will be assigned one laboratory session per week. All experiments that count towards Core Chemistry 1A contain a suffix of 'A' in the title (e.g. Experiment 3A) and will be carried out by everybody. Experiment titles containing a suffix of 'B' (e.g. Experiment 3B) will count towards Core Chemistry 1B and will be carried out only by those studying Core Chemistry 1B. These experiments do not appear in this laboratory manual.

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.

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**
Thurs am	actinium	bismuth	cobalt
Friday am	dysprosium	erbium	fluorine
Friday pm	gadolinium	hafnium	yttrium
Monday am	potassium	lithium	molybdenum
Monday pm	niobium	phosphorus	osmium
Tuesday am	rhodium	strontium	tantalum
Wed am	uranium	vanadium	tungsten

Set lists will appear on DUO before the start of Week 2. Sets will perform experiments according to the following rota:

Week	**Set 1**	**Set 2**	**Set 3**
2	Experiment 2	Experiment 3	Experiment 4
3	Experiment 3	Experiment 4	Experiment 2
4	Experiment 4	Experiment 2	Experiment 3

For example, in week 3, everyone in Set 2 will carry out Experiment 4. As a student studying only Core Chemistry 1A, you will only complete Experiment 4A. Those students also studying Core Chemistry 1B will complete Experiment 4B during their second session of the week. During Week 1, you will find out your allocated set and should write the details onto the front of your laboratory manual.

1.5 Assessment

Pre-lab exercises, mostly carried out via DUO, 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

	Core Chemistry 1A	**Core Chemistry 1B**
Pre-lab exercises (whole year)	10 %	10 %
Laboratory session marks (whole year)	10 %	10 %
SKILLS Experiment 5B: Determination of the enthalpy of vaporisation of ethanol	-	5%
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 % *
	100 %	100 %

*For students completing both Core Chemistry 1A and Core Chemistry 1B, 50 % of the project mark 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 1 – week 2 to week 4 (Thursday 13th October to Wednesday 2nd November 2011)

Senior Demonstrators (staff)	Junior Demonstrators (postgraduate students)
Dr Jacquie Robson* Dr Pippa Coffer Dr Ehmke Pohl	Jonathan Coome Alex Dudgeon Rebecca Edwards Ricardo Girling Helen Mason Hannah Straker Julie Thye

* Laboratory course leader - email: j.m.robson@durham.ac.uk

SKILLS
BLOCK 1
EXPERIMENT 2A

INVESTIGATING CONCEPTS OF EVIDENCE: HOW MUCH WATER CAN A PAPER TOWEL SOAK UP?

2A. Investigating concepts of evidence: measuring how much water a paper towel can soak up?

An understanding of how experiments are designed and structured and ideas about reliability, causality and validity are essential to enable critical evaluation of scientific information. These ideas are touched upon in school science curricula but a thorough understanding of these essential concepts can take time and practice to fully appreciate.

Designing an experiment requires much scientific understanding. All the ideas detailed above need to be carefully considered and the design adapted and refined after testing to produce a procedure that gives good quality evidence or data. Many of the scientific techniques that often need to be learned by rote, such as titration, are protocols developed by previous scientists, with consideration for these key ideas, to ensure that the data produced are of the highest possible quality. Designing a new experimental method is a very different skill to carrying out a learned protocol. This activity allows that skill to be practised with a focus on the processes involved. Experiments are designed to allow the collection of evidence or data. Measurement is central to the collection of scientific evidence, but how a measurement is carried out and the choice of the apparatus used is key to understanding the quality of the evidence.

This activity, set in a very simple context, allows the exploration of some of these issues. Treatment of data, using the mean, the range, the standard deviation and the standard error, is also investigated.

Think carefully about each step of this activity. Try not to focus on the outcome, but on the reasoning behind any experimental decisions or assumptions made. Different people within a group, and different groups, will come up with different ideas to tackle this exercise. This is encouraged as the discussion around each idea is essential to develop understanding. There is no single right answer or expected outcome for this activity, although some methods may give better quality data than others. Learning about and understanding the planning process and the ideas detailed above is the desired outcome. The aim is to develop understanding of some key ideas about data, evidence, measurement and experimental design by exploring many different possibilities.

2A.1 Aims

- To design and carry out a simple procedure to answer a question
- To develop understanding of the ideas behind experiment design and measurement
- To discuss and develop understanding of the use of mean values, ranges, standard deviations and standard errors in reporting experimental findings

2A.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.

If carrying out this activity in Week 2, ensure that all the post-lab exercises from the first laboratory session are completed. This includes setting up the lab notebook ready for use.

Reading exercise:

1. Read through all experimental instructions given in the laboratory manual.
2. 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 is available to all Durham students via the University Library web pages (follow the link on DUO). Ensure that Chapters 2, 3, 6, 7 and 8 are read particularly thoroughly and used to explain any unfamiliar terms in the experiment instructions.

Make sure you understand the following terminology (page numbers refer to Gott and Duggan. Additional information is available in the National Physics Laboratory 'Beginner's Guide to Uncertainty of Measurement' by Stephanie Bell (1999), available via DUO).

- validity and reliability (p6-7)
- precision (p117-119)
- accuracy (p119-123)
- standard deviation (p143-149)
- standard error (p149-p152)

Use the ideas presented in the book to inform any decisions made during the next task. To get the most out of the laboratory activity and the discussions, it is essential that the reading has been done properly before arriving in the laboratory.

Planning exercise:

Available apparatus:

- thermometers
- stopwatches
- measuring cylinders of various sizes
- beakers of various sizes
- droppers
- pipettes
- top pan balances
- funnels
- paper towels

Using apparatus from the list and any other available laboratory apparatus, design a simple procedure to determine how much water a paper towel can soak up. Describe equipment sizes and volumes. Think about the reasons behind the selection of particular apparatus or any particular scale (i.e. what volumes of liquids to use) and consider the ideas presented in the textbook when making these decisions. Prepare the next page of the lab notebook for use in the session, and write the method in. Design and draw a results table in the lab notebook to contain the data to be collected.

A demonstrator will check that you have completed this task before you will be allowed to join the rest of your group to begin the laboratory activity.

2A.3 Laboratory activity

Suggested timings are given for each part of the activity to ensure that groups complete the activity on time.

(30 minutes)

In groups of three or four, each group member is to take turns to present the procedure that was designed in the pre-lab. Discuss the strengths of each procedure, identify any possible areas for improvement and consider any issues or difficulties that each particular method may present. Discuss the validity of the method and any attempts to ensure precision, accuracy and reliability. Remember, there is no single right answer here and it is to be expected that there will be up to four different procedures to discuss, not four versions of an identical method! This exercise is not about identifying which method is 'correct', but about considering a number of different ways of approaching the same problem and considering different aspects of these approaches.

After all procedures have been presented, decide, as a group, which one is the:

- most imaginative
- most complex
- simplest
- fastest method to produce 10 pieces of data

(20 minutes)

Once all of the methods have been considered and ideas about experimental design have been discussed, a single method should be decided upon. This could be one that has already been presented, or could be a new method designed after discussion of the strengths of the different procedures. This agreed method should be reported in the lab notebook of each group member. Note down any decisions that have been made, with reasons, about the experimental design, including any assumptions that have been made or any definitions that have been decided upon. **Show a demonstrator your method before moving on to the next part.**

(45 minutes)

Working efficiently in groups, collect at least 10 pieces of data using the agreed method. Adapt the design if required to ensure the data are as good quality as they can be for the method used, but do not use unnecessarily precise equipment. Ensure that there are a minimum of 10 pieces of data for the experiment, recorded in lab notebooks in an appropriate table. Remember that each member of the group needs a record of the data. **Show a demonstrator the results before moving on to the next part.**

(45 minutes)

Each group should now have 10 pieces of data (x_1, x_2, x_3 ... x_{10}) which should show variation (i.e. they will not all be the same). Having a number of different pieces of data representing the measure of a single thing is usual and common in science. To be able to report a single value (i.e. one single number that represents all 10 pieces of data), some statistical treatment of the numbers is required. The first calculation usually performed on a data set is the determination of a mean value. The best estimate for a single value of x is given by the arithmetic mean. The formula for calculating the arithmetic mean from n data points is:

$$\bar{x} = \frac{x_1 + x_2 + \ldots x_n}{n} \quad \text{or} \quad \bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

Individually, calculate the arithmetic mean for the data set and determine the range of the data. Compare the values with that calculated by the rest of the group and discuss the following, noting findings in the lab notebook:

1. Did everyone choose to use all 10 pieces of data or were some ignored? Why?
2. What information does a mean value give about the data?
3. If the mean value alone is reported, is it a true reflection of the findings from the experiment?
4. Were the data 'good enough' to draw a conclusion for this experiment? Decide upon criteria for 'good enough'.

Show a demonstrator your findings before moving on to the next part.

Each individual value deviates from the mean by d_i where: $d_i = x_i - \bar{x}$

Note that d_i can be both positive and negative and that: $\sum_{i=1}^{n} d_i = 0$

It is usual to report the standard deviation (usually given the symbol s or σ_x) from the mean. This is the square of the individual deviations for each piece of data, divided by n-1, where n is the number

of measurements. The value *n-1* is used, rather than *n*, for good statistical reasons.[1] Use the formula below to calculate the standard deviation for the data set:

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

where σ_x = standard deviation of the sample
x_i = a single measurement
$\bar{x}$ = mean
n = number of measurements / sample size

Compare the values within the group to check the calculation. Discuss the following within the group, noting findings in the lab notebook:

1. What information does the standard deviation give about the data that has been collected?
2. What would the data set 'look' like if the standard deviation was larger?
3. What would the data set 'look' like if the standard deviation was smaller?
4. In what way is reporting a mean with a standard deviation more useful than reporting a mean and a range?
5. What are the limitations of reporting a standard deviation?

Tell a demonstrator your findings before moving on to the next part.

The mean value represents the average of a distribution of 10 different values. The standard error (often referred to as the 'standard deviation of the mean') is also often reported as it gives a measure of the precision to which $\bar{x}$ is known. Calculate the standard error for the data. The mathematical formula for the standard error is:

$$SE = \frac{\sigma_x}{\sqrt{n}}$$

where *SE*= standard error (sometimes shown as $\sigma_{\bar{x}}$)
σ_x = standard deviation
n = the number of readings

[1] For a more in-depth explanation, see p87 of Taylor, J. R., 'An Introduction to Error Analysis: The Study of Uncertainties in Physical Measurements', (1982), University Science Books.

Discuss the following within the group:

1. What different information does the standard deviation and the standard error give about a particular data set?
2. What does the value of the standard error tell us about the data?
3. What simple change could be made to the experiment design to reduce the standard error?
4. Make a claim about the data collected by the group (e.g. How much water can a paper towel soak up?). Consider the mean, the standard deviation and the standard error when reporting your findings.
5. Are the claims of other groups likely to be the same? Why? What are the limitations of the groups' claim?

Report the group's findings to the demonstrator for discussion.

Use of Excel to calculate mean, median, mode, standard deviation and standard error:

Open Excel on one of the laboratory computers and input the group's data set (all 10 pieces of data) in one column. Click on the 'Data Analysis' tab and select 'Descriptive Statistics'. Use the mouse to highlight the data. This should show in the 'Input Range' box. Click for a new worksheet to be generated and tick 'Summary Statistics' box. Clicking OK will generate a table on another sheet that shows the mean, standard error, median, mode and standard deviation (among other things) for the data. Compare the values to those you calculated. This tool can be used in Excel to analyse data sets collected during other practical work in this course.

Tidy up the work area and put equipment away. **Show a demonstrator the lab notebook to be signed off before leaving the laboratory.**

2A.4 Post-laboratory work

Review the Gott and Duggan textbook after the session to consolidate your learning during the laboratory activity. This book is available for open access by members of the University via the library website (the link is available on DUO):
Gott, R. and Duggan, S. "Understanding Scientific Evidence – How to critically evaluate data" 2003, Sage Publications, London, Thousand Oaks, New Delhi.

You should also review the National Physics Laboratory Measurement Good Practice Guide No. 11: Beginners Guide to Uncertainty in Measurement by Stephanie Bell (1999), available on DUO.

SKILLS
BLOCK 1
EXPERIMENT 3A

DETERMINATION OF THE M_r OF AN UNKNOWN SOLID ACID USING TITRATION

3A. Determination of the M_r of an unknown solid acid using titration

In Experiment 2A, many of the issues that can arise when planning an experiment to collect good quality data are explored. Certain experimental techniques have evolved over time to give an agreed standard procedure that chemists can follow to produce results that are precise, accurate and reliable (assuming the procedure is followed carefully). These procedures take account of all the issues explored in experiment 2A. Learning how to perform these standard procedures is a key part of your training as a good practical chemist. The procedures in this experiment include the preparation of a standard solution and the use of titration. These procedures should be familiar to you from your previous studies, but it is important that you follow the pre-lab exercises through from start to finish to revise the methods and to be fully prepared for your laboratory class.

3A.1 Pre-lab exercises

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

1. If completing this activity in Week 2, ensure that all the post-lab exercises from the first laboratory session are completed. This includes setting up the lab notebook ready for use.

2. Read your Experiment Information carefully and highlight unfamiliar words or apparatus.

3. 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 find the meaning of any unfamiliar words contained in your laboratory manual.

4. Under the 'Quantitative analysis' sub-heading, move the cursor over 'Titration' and then select 'Video' from the pop-up list. Watch each of the video clips in turn to familiarise yourself with the technique.

5. Return to the main menu and again move the cursor over 'Titration'. This time, select 'Technique' and work through the buttons on the yellow and blue bar at the bottom, beginning with reading the 'What is titration' introduction page, then completing the interactive exercises in 'Setting up equipment', 'Taking readings', 'Finding an End-Point' and 'Analysing results'. Keep working through these until you have practiced and are comfortable with the required procedure for the experiment. This includes being comfortable with the calculations you will be required to do. You may find it helpful at this point to write a more detailed method into the lab notebook, using the information

provided on LabSkills and the Experiment Information provided in this laboratory manual, ready for use in your laboratory session. This will ensure that you are able to work independently and confidently. You should also plan or prepare your results table ready for use during your experiment.

6. The accepted uncertainty in the laboratory burettes is ±0.05 cm^3. This means that the glassware has been manufactured to guarantee that any reading taken using it will be within 0.05 cm^3 of the true value. Thus the burettes should be read to the nearest 0.05 cm^3 (and not any values in between). LabSkills allows you to practice reading a burette before arrival in the laboratory. Practice this on LabSkills to perfect technique (this task is not assessed so take as many attempts as required to ensure that readings are being done correctly). Look at the picture in Figure 3A.1. It can be seen that the meniscus sits just below 1.40 cm^3, but not as far as 1.45 cm^3. It is, however, closer to 1.40 cm^3, thus this is the reading from this burette. Remember to record this value in the results table to 2 decimal places. i.e. for the reading in Figure 1, the results table would show a value of 1.40 cm^3, not 1.4 cm^3. All readings in the results table should be of the form X.X0 cm^3 or X.X5 cm^3. Remember to record readings to this level of precision during the experiment.

Figure 3A.1

7. View the 'Safety' section from the 'Titration' menu and ensure you have clicked on all information points to read about the safety aspects of your experiment.

8. From LabSkills Main Menu, go to the 'Basic Skills' section on the right hand side, and select 'Preparing Solutions'. Work through all of this revision material to prepare for making the standard solution of Acid A during the experiment. It may also be useful to read and view the 'Acid/Base Titration' section (under 'Titration' and then 'Common Experiments').

9. Complete the pre-lab quiz (available on DUO in the Experiment 3A folder). Results for the first attempt at this test will be stored and will count towards the summative mark for the pre-lab. Review all the answers after completion of the test to receive feedback on the responses and corrections to any mistakes.

3A.2 Aims

- To practice appropriate use of titration apparatus following the accepted procedure
- To prepare a standard solution following the accepted procedure
- To use acid-base titration to determine concentration of an unknown
- To use acid-base titration to determine the M_r of an unknown solid acid
- To use an analytical balance

3A.3 Risk assessment

CHEMICAL RISKS	RISKS	SAFETY
Hydrochloric acid	At this concentration, treat as irritant.	Wear gloves.
Sodium hydroxide	At this concentration, treat as irritant.	Wear gloves.
Unknown Acid A	Treat as hazardous. Do not allow contact with eyes or skin.	In case of contact rinse immediately with plenty of water. Wear suitable protective clothing and gloves when handling.
Phenolphthalein	Limited evidence of a carcinogenic effect. Irritating to eyes, respiratory system and skin.	Wear suitable protective clothing and gloves.

3A.4 Laboratory activity

Please note that the analytical glassware such as volumetric flasks, glass pipettes and burettes should not be placed in the ovens as this can distort the glass and thus affect the calibration of the apparatus. Only less accurately calibrated glassware, such as beakers, conical flasks, Quick-fit and test tubes, should be placed in the glass oven**.**

This experiment revises techniques you will have learned before arriving in Durham. You will therefore perform this experiment on your own. In Part 1 you will standardise (determine the concentration of) a solution of sodium hydroxide using a solution of hydrochloric acid of known concentration. In Part 2 you will make up a standard solution of unknown solid acid **A**, and use the standardised solution of sodium hydroxide to perform a further titration to allow you to calculate the M_r of acid **A**.

Part 1 – Standardisation of the NaOH solution

Choose a work area with a free locker, and write the locker number at the top of the lab notebook page for this experiment. You must also inform the demonstrator of the locker number. Following the procedure outlined and practiced in the LabSkills software during the pre-lab exercises, titrate 25.00 cm^3 aliquots of the NaOH solution (to be standardised) against the hydrochloric acid solution of known concentration. Use two to three drops of phenolphthalein as the indicator. Record the volume at the end point and calculate the titre value. This is the rough titre. Using the rough titre as a guide, repeat the titration more carefully until two consecutive, concordant (±0.05 cm^3) results are obtained. This will take a minimum of three titrations, or it may take more if the titration is not performed very carefully. **Ask the supervising demonstrator to check one of your end point readings**. Use the results from your titration to calculate the concentration of the sodium hydroxide solution and record to 3 significant figures. Use this value in calculating your answer to Part 2. **Show your value for the concentration to the demonstrator to assess and note down.**

*Part 2 – Determination of the Mr of acid **A***

Record the mass of an empty weighing bottle and lid on an analytical balance. Then place the bottle on a top pan balance and tare (set it to zero). Add between 1.9 g and 2.1 g of acid **A** to the bottle (ensure the mass is within this range). Place the lid back on the weighing bottle and return it to the analytical balance and record the mass of the weighing bottle, lid and acid **A**. Transfer the acid **A** to a 250 cm^3 beaker. Re-weigh the weighing bottle and lid (and any residual acid **A**) using an analytical balance and record the mass. Determine the mass of acid **A** in the beaker.

Fully dissolve the solid acid **A** in no more than 100 cm^3 of deionised water before transferring the solution carefully and quantitatively to a 200.00 cm^3 volumetric flask. Transfer the washings from the beaker to ensure all the acid **A** is transferred to the volumetric flask. Make the solution up to the mark **and ask the supervising demonstrator to check that the meniscus is on the line**. Once checked, stopper the flask and invert a number of times to mix thoroughly.

Using the technique revised on LabSkills, titrate 20.00 cm^3 aliquots of the sodium hydroxide solution used in Part 1 (now of known concentration) with this freshly prepared solution of acid **A**, using phenolphthalein as the indicator. Repeat until two consecutive titrations differ by no more than 0.05 cm^3. Remember to read the burette to the nearest 0.05 cm^3 and record your results to two decimal places. **Show the supervising demonstrator your titration results.**

Assuming that acid **A** is a monobasic acid, calculate the amount (in moles) of acid **A** that reacts in the titration with NaOH. Use this information, and the mass of acid **A** used to prepare the standard solution, to calculate the relative molecular mass, M_r, of acid **A**. Report the result to an appropriate degree of precision. **Show your M_r value to the supervising demonstrator for them to assess and**

note down. Acid **A** is an analytical standard, so it is anticipated that results will be in the range ±0.5% of the actual M_r.

Discard the remaining solution of acid **A** down the sink with plenty of water. Clean and wash all equipment used, and leave apparatus in a good condition for the next person working in that area. Ensure all taps are turned off. **Ask a demonstrator to mark your lab notebook and to sign off your tidy work space before leaving the laboratory**. **Ensure that the demonstrator has made a note of the concentration of NaOH determined in Part 1, and the M_r value calculated in Part 2 before leaving the laboratory.**

SKILLS
BLOCK 1
EXPERIMENT 4A

PURIFICATION OF AN UNKNOWN SOLID BY RECRYSTALLISATION

4A. Purification of an unknown solid by recrystallisation

In this experiment you will be recrystallising an impure solid. This is a common purification method.[2] The purpose of recrystallisation is to remove both soluble and insoluble impurities from a solid. To do this, a solvent must be found in which the bulk of the material will dissolve when it is hot but not when it is cold. The insoluble impurities can then be filtered from the hot solution. On cooling, the desired material crystallises leaving the soluble impurities still in solution. The pure compound can therefore be filtered from the cold solution. You will be using reflux apparatus and handling other common laboratory apparatus. Some of these techniques may be familiar to you from your pre-University studies, but it is important that you follow the pre-lab exercises through from start to finish. This will allow you to revise the procedures and prepare for the laboratory class.

4A.1 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. If completing this activity in Week 2, ensure that all the post-lab exercises from the first laboratory session are completed. This includes setting up the lab notebook ready for use.

2. Read the instructions in your laboratory manual through carefully and highlight unfamiliar words or apparatus.

3. 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.

4. Preparing for the recrystallisation: Under the 'Preparation and Purification' sub-heading, move the cursor over 'Recrystallisation' and then select 'Technique' from the pop-up list. Read the first page about recrystallization, and practice the simulation on the second page until you have successfully recrystallized the solid. Then select 'Video' and watch the procedure again, and then select 'Safety' and read through the safety points, focusing particularly on use of stirrer hotplates.

[2] For further information about recrystallisation, see pp92-101 of Dean, J. R. et al, 'Practical Skills in Chemistry', (2002), Prentice Hall or p127 of Harwood, L. M. and Moody, C. J., 'Experimental Organic Chemistry: Principles and Practice' (1989), Blackwell Scientific Publications.

5. Preparing for using reflux apparatus: Solids can be recrystallised from water using a conical flask and stirrer hotplate, as shown in the simulation in LabSkills or, for more volatile and flammable solvents, alternative apparatus can be used. The solid in this experiment, although being recrystallised from water, will be dissolved using reflux apparatus to provide students with experience in setting up this glassware. It would, however, be more usual[3] to use reflux apparatus for more volatile and flammable solvents than water! To become familiar with the required apparatus, go back to the Main Menu of LabSkills and under the 'Preparation and Purification' sub-heading, move the cursor over 'Reflux' and then select 'Video' and watch the procedure. Next, select 'Technique' and read about the use of the apparatus and then have a go at setting it up using the simulation. Keep practising with the simulation until you have successfully reached reflux. Remember that this experiment still involves a simple recrystallisation, just using reflux apparatus instead of a conical flask. Finally, view the safety information provided about reflux apparatus.

6. From the Foundation Chemistry LabSkills main menu, select 'Filtration'. Select 'Technique' from the menu and read through the information, clicking along the progress bar at the bottom to move through. When complete, click on 'Gravity Filtration Video' and watch all stages, then click on 'Buchner filtration video' and watch through. Both of these methods of filtration should be familiar from prior studies, but, if not, take time to learn about the differences between them and their uses.

7. Preparing for taking a melting point: Select 'Melting Point' from the LabSkills 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.

8. It is likely that previous practical work has usually required the use of a Bunsen burner for heating. In the undergraduate laboratory, it is more likely that a hot plate will be used, accompanied by a magnetic stirrer bar (also known as a 'stir flea'). To prepare for using this apparatus, open the Chemistry Interactive Lab Primer, either via DUO or using a web browser (http://chem-ilp.net). Select the 'Lab Techniques' tab and select 'Heating' from the menu. Click on 'Hot Plates' and read all information about the use of stirrer hotplates and magnetic stirrer bars.

9. Prepare the lab notebook ready to begin the laboratory session. This should include drawing diagrams of reflux apparatus and Buchner filtration apparatus and any necessary notes

[3] See p96 of Dean, J. R. et al, 'Practical Skills in Chemistry', (2002), Prentice Hall for further discussion.

about taking a melting point to assist during the experiment. The diagrams will not be provided in the laboratory manual.

4A.2 Aims

- To develop confidence with use of reflux apparatus
- To purify an unknown solid by recystallisation
- To practice using different methods of filtration

4A.3 Risk assessment

CHEMICAL	RISKS	SAFETY
Unknown solid	Harmful. Irritant.	Wear suitable protective clothing and gloves.

4A.4 Laboratory activity

1. Work in pairs for this experiment. 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. **Inform the demonstrator of the locker and fume cupboard number**.

2. Using a weighing bottle or weighing boat, weigh out approximately 2.0 g of impure solid. Over a piece of paper, carefully transfer the solid to a 100 cm^3 B19 round bottomed flask.[4] Keep a small amount in the weigh bottle for melting point determination. Record the mass of impure solid used.

3. Add 20 cm^3 of deionised water and a magnetic stirrer bar. Set up the apparatus for heating at reflux as practised in the pre-lab exercises. **Show a demonstrator the reflux equipment set-up before switching on the heat source.** Heat the mixture using a hotplate and heating block with hotplate on full. Once reflux has been reached, remove from the hotplate, observe how much solid material is remaining, add more water (5 cm^3) down top of condenser and re-heat. Repeat this process until no more solid material dissolves. Note that not all of it will dissolve because the sample contains a solid impurity. Remember that recrystallisation involves the use of the minimum amount of solvent to prepare a saturated solution, so do not add too much.

4. Filter the hot saturated solution through a fluted filter paper (see Appendices 1 and 2, page 29) in a pre-heated large filter funnel into a preheated, insulated 100 cm^3 conical flask. This

[4] 'B19' indicates the size of the glass joint on the flask. All of our glassware is 'B' and is typically B10, B14, B19 or B24 in size. The numbers indicate the diameter of the joint. Thus a B19 joint is larger in diameter than a B10 and B14 but smaller than a B24. The size is often indicated near the neck of the glassware.

process removes insoluble materials (e.g. dust). If the product crystallises in the filter paper, add a small amount of boiling water and allow it to filter. Repeat until the crystals are fully through the filter paper.

5. Allow the saturated solution to cool slowly in the insulated flask, whereupon crystals will form.[5] **Show a demonstrator the crystals in the conical flask**. Filter this solution using a Buchner funnel, and when all the liquid phase is just disappearing, wash the crystals on the filter paper with fresh, cold water, keeping the vacuum on and still sucking. Suck the crystals as dry as possible and press down the crystals with a stopper or spatula. Transfer the crystals to a pre-weighed watch glass and dry by gently pressing a filter paper down onto the crystals until they are as dry as possible. If they are very wet, place in an oven on the watch glass for half an hour (ensure that the oven is not too hot). Record the mass of the dry crystals. **Show a demonstrator the crystals produced, and the mass recorded.**

6. Determine the melting point of the recrystallised material and the impure starting material. Follow the procedure practised on LabSkills: Fill melting point tubes with each of the two materials and perform a rough determination of the melting point of one by heating rapidly (i.e. at the maximum rate); observe the tube closely through the viewfinder and note the approximate melting temperature. Cool the heating block to 20 ^{o}C below the approximate melting point, insert a fresh tube and reheat SLOWLY. Record the temperature at which the solid first begins to melt, and the temperature at which it has all melted, i.e. a temperature range. If time, perform accurate repeats of the melting point determination. If enough data has been collected, report one single melting point for each solid. **Show a demonstrator the melting point results.**

7. Wash up (where required) and put away all equipment. Ensure contaminated filter papers are placed into the 'solid waste' receptacle provided, with all instructions about packaging the waste followed carefully. Ensure locker space and fume cupboard is clean and tidy. **Ask a demonstrator to sign off the locker and work space.**

8. Discuss the following questions within pairs and write answers into the lab notebook. **Show a demonstrator the lab notebook for discussion of answers and for signing off before leaving the laboratory.**

[5] The solution remaining after recrystallisation is often referred to as the *mother liquor*. The crystals are usually isolated from the mother liquor by filtration, leaving the mother liquor in the receiving flask (the filtrate).

Question 1

What are the melting point criteria for judging whether an organic solid is pure?

Question 2

Which way does the melting point of an increasingly purified solid change – upwards or downwards?

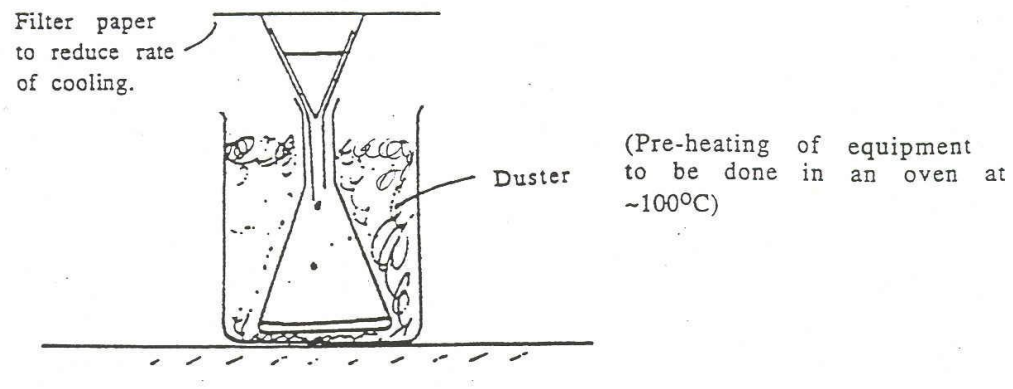
4A.5 Appendix 1 – Procedure for fluting a filter paper

1. Fold the paper in half and open it out flat again
2. Rotate through 90^{o} and repeat step 1 (the paper is now folded in quarters)
3. Fold in half again so that two quarters are bisected (the easy way to do this is to match up the previous folds) and again open it out flat.
4. Repeat step 3. Your paper should now be folded into eighths.
5. Turn the paper over.
6. Fold in half to bisect two of the eighths (again, match up some of the previous folds) and open it out flat.
7. Rotate the paper and repeat step 6; keep going until all the eighths have been folded in half. Your paper should now be in sixteenths, with each alternate fold going the opposite way.
8. Fold your paper into a concertina shape – you are now ready to filter!

A video demonstrating how to flute a filter paper is available on DUO. If you are struggling with the origami, you might want to use a free computer in the laboratory and follow along with the video to fold your paper.

4A.6 Appendix 2 – Procedure for performing a hot gravity filtration

1. Line a large beaker with a duster and insert your conical flask and filter funnel.
2. Prepare a fluted filter paper as in Appendix B; place it in the filter funnel
3. Place the whole set up in a hot oven and leave for about 10 min.
4. Carry out the filtration of the hot solution immediately; place a filter paper over the mouth of the funnel between additions of solution to slow down the rate of cooling.



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**
First Class	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.
Upper Second	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.
Lower Second	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.
Third	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.
Fail	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.