

# INTERNATIONAL AS AND A-LEVEL CHEMISTRY (9620)

Practical handbook

This is the **Chemistry** version of this practical handbook.

The sections on tabulating data, significant figures, uncertainties, graphing, and subject specific vocabulary are particularly useful for students and could be printed as a student booklet by schools.

The information in this document is correct, to the best of our knowledge as of September 2018.

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## INTRODUCTION

Practical work brings science to life, helping students make sense of the universe around them. That's why we've put practical work at the heart of our Biology, Chemistry and Physics International AS and A-levels. Practical science allows scientific theory to transform into deep knowledge and understanding – scientific thinking. Through investigation, students uncover the important links between their personal observations and scientific ideas.

"In the best schools visited, teachers ensured that pupils understood the 'big ideas' of science. They made sure that pupils mastered the investigative and practical skills that underpin the development of scientific knowledge and could discover for themselves the relevance and usefulness of those ideas."

Ofsted report

Maintaining curiosity in science

November 2013, No. 130135

### THE PURPOSE OF THIS PRACTICAL HANDBOOK

This handbook has been developed to support you in advancing your students to fluency in science.

Over the years, there have been many rules developed for practical work in Biology, Chemistry and Physics. Some have been prescriptive; some have been intended as guidance. Although we have always attempted to be consistent within subjects, differences have emerged over time. For example, students taking Physics may also be taking Biology and find themselves confronted with contradictory rules and guidance.

This practical handbook is an attempt to harmonise the rules and guidance for International Biology, Chemistry and Physics. There are occasions where these will necessarily be different, but we will try to explain why on the occasions where that happens.

We have worked with teachers, technicians and examiners to produce this handbook. This has been an evolving document, but one that we hope you will be able to use with your students, whether they're doing International AS or A-level Biology, Chemistry or Physics, or a combination of subjects, to improve their practical skills: in the classroom, in the laboratory, in exams and on to university or the workplace.

Unless specified, all guidance is common to Biology, Chemistry and Physics at both International AS and A-level and subject-specific examples are for illustration only. However, the extent to which a particular aspect is assessed will differ. Teachers should refer to the specifications and specimen materials on our website for more information.

### THE PURPOSE OF PRACTICAL WORK

There are three interconnected, but separate reasons for doing practical work in schools and colleges. They are:

1. To support and consolidate scientific concepts (knowledge and understanding).

This is done by applying and developing what is known and understood of abstract ideas and models. Through practical work we are able to make sense of new information and observations, and provide insights into the development of scientific thinking.

- 2. To develop **investigative skills**. These transferable skills include:
  - devising and investigating testable questions
  - identifying and controlling variables
  - analysing, interpreting and evaluating data.
- 3. To build and master **practical skills** such as:
  - using specialist equipment to take measurements
  - handling and manipulating equipment with confidence and fluency
  - recognising hazards and planning how to minimise risk.

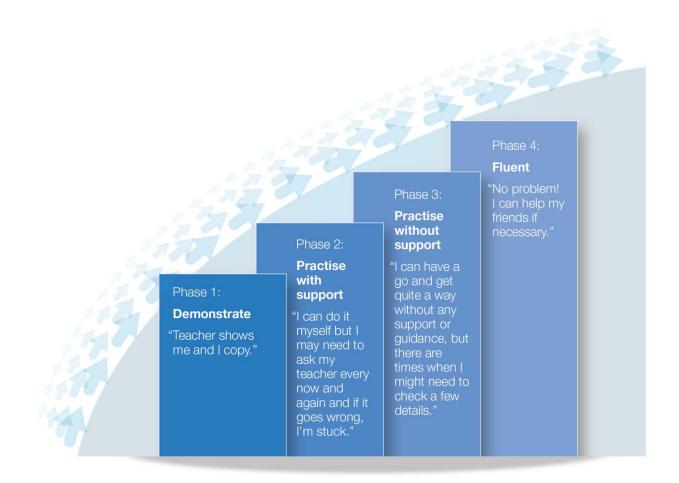
By focusing on the reasons for carrying out a particular practical, teachers will help their students understand the subject better, to develop the skills of a scientist and to master the manipulative skills required for further study or jobs in STEM subjects.

The International AS and A-levels in Biology, Chemistry and Physics separate the ways in which practical work is assessed. This is discussed in the next section.

### FLUENCY IN SCIENCE PRACTICAL WORK

At the beginning of a Year 12 course, students will need support and guidance to build their confidence. This could involve, for example, breaking down practicals into discrete sections or being more explicit in instructions. Alternatively, a demonstration of a key technique followed by students copying may support their development. This could be a better starting point than 'setting students loose' to do it for themselves.

### Progression in the mastery of practical skills and techniques shows increasing independence and confidence.



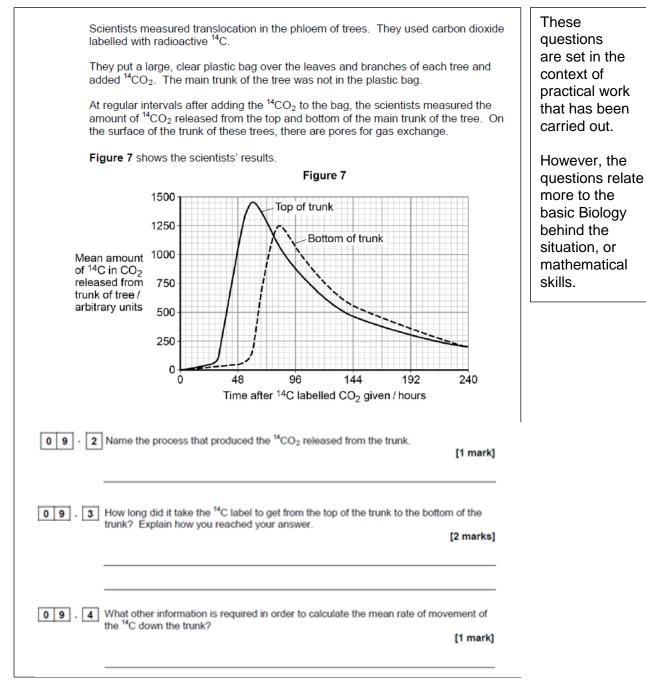
Safety is always the responsibility of the teacher. No student should be expected to assess risks and then carry out their science practical without the support and guidance of their teacher.

## PRACTICAL SKILLS ASSESSMENT IN QUESTION PAPERS

The International AS and A-level papers will contain the following types of questions which relate to practical work:

1. Questions set in a practical context, where the question centres on the science, not the practical work.

### **EXAMPLE (A-LEVEL BIOLOGY SPECIMEN PAPER 1)**



### INTERNATIONAL AS AND A-LEVEL CHEMISTRY (9620) PRACTICAL HANDBOOK

### **EXAMPLE (AS CHEMISTRY SPECIMEN PAPER 1)**

4	Colourless solutions of $X(aq)$ and $Y(aq)$ react to form an orange solution of $Z(aq)$ according to the following equation.
	$\mathbf{X}(aq) + 2\mathbf{Y}(aq) \rightleftharpoons \mathbf{Z}(aq)  \Delta H = -20 \text{ kJ mol}^{-1}$
	A student added a solution containing 0.50 mol of <b>X</b> (aq) to a solution containing 0.50 mol of <b>Y</b> (aq) and shook the mixture. After 30 seconds, there was no further change in colour. The amount of <b>Z</b> (aq) at equilibrium was 0.20 mol.
04.1	Deduce the amounts of <b>X</b> (aq) and <b>Y</b> (aq) at equilibrium. [2 marks]
	Amount of <b>X</b> (aq) =mol Amount of <b>Y</b> (aq) =mol

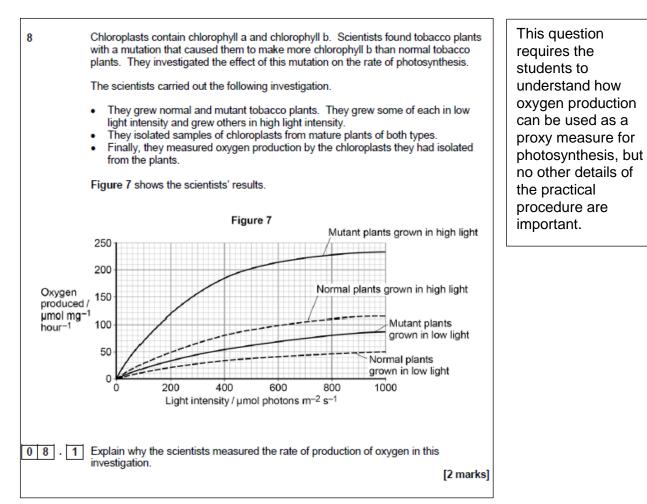
This question requires an understanding of the underlying chemistry, not the practical procedure undertaken.

### **EXAMPLE (A-LEVEL PHYSICS SPECIMEN PAPER 3)**

02.6	The experiment is performed with a capacitor of nominal value 680 $\mu$ F and a manufacturing tolerance of $\pm$ 5 %. In this experiment the charging current is maintained at 65 $\mu$ A. The data from the experiment produces a straight-line graph for the variation of pd with time. This shows that the pd across the capacitor increases at a rate of 98 mV s <sup>-1</sup> . Calculate the capacitance of the capacitor. [2 marks]	This question is set in a practical context, and particular readings need to be used to calculate the answer, but the specific practical set-up is not important.
	capacitance =µF	

2. Questions that require specific aspects of a practical procedure to be understood in order to answer a question about the underlying science.

### EXAMPLE (A-LEVEL BIOLOGY SPECIMEN PAPER 2)



To answer this question, the student must understand the process of yield calculation (which will have been gained through practical work), but again the details of the practical

procedure are

unimportant.

H<sub>2</sub>O

[2 marks]

### **EXAMPLE (AS CHEMISTRY SPECIMEN PAPER 2)**

0 1 . 4 The effect of gentle heat on maleic acid is shown below.

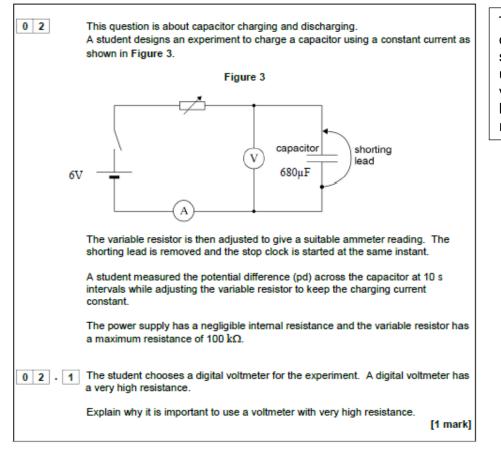
were obtained

A student predicted that the yield of this reaction would be greater than 80%.

Is the student correct? Justify your answer with a calculation using these data.

In an experiment, 10.0 g of maleic acid were heated and 6.53 g of organic product

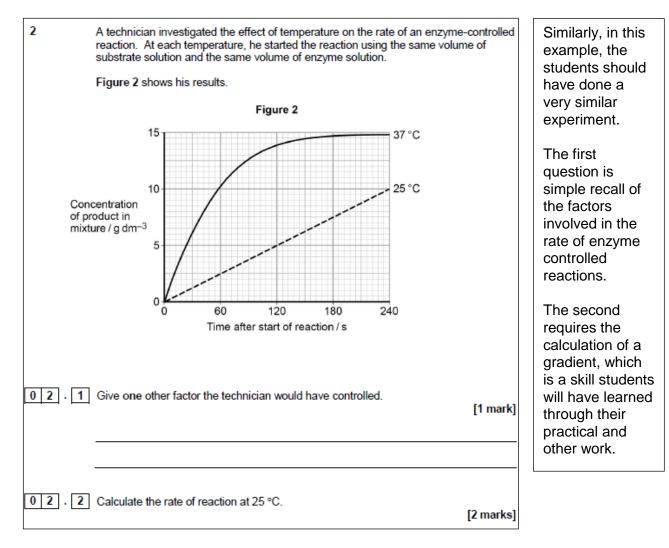
### **EXAMPLE (A-LEVEL PHYSICS SPECIMEN PAPER 5)**



To answer this question, the student must understand why a voltmeter needs to have a very high resistance.

### 3. Questions directly on the required practical procedures.

### **EXAMPLE (AS BIOLOGY SPECIMEN PAPER 1)**



### INTERNATIONAL AS AND A-LEVEL CHEMISTRY (9620) PRACTICAL HANDBOOK

### **EXAMPLE (A-LEVEL CHEMISTRY SPECIMEN PAPER 3)**

3	A peptide is hydrolysed to form a solution containing a mixture of a mixture is then analysed by silica gel thin-layer chromatography (TI solvent. The individual amino acids are identified from their Rr value Part of the practical procedure is given below.	LC) using a toxic es. have completed the related required practical
	<ol> <li>Wearing plastic gloves to hold a TLC plate, draw a penc the bottom of the plate.</li> </ol>	il line 1.5 cm from will have a greater understanding of
	<ol> <li>Use a capillary tube to apply a very small drop of the solution the mid-point of the pencil line.</li> </ol>	each of the steps
	<ol><li>Allow the spot to dry completely.</li></ol>	in the procedure
	<ol> <li>In the developing tank, add the developing solvent to a dep than 1 cm.</li> </ol>	
	<ol><li>Place your TLC plate in the developing tank.</li></ol>	explain each in
	<ol> <li>Place your TLC plate in the developing tank.</li> <li>Allow the developing solvent to rise up the plate to the top.</li> </ol>	turn.
	<ol><li>Remove the plate and quickly mark the position of the solve pencil.</li></ol>	
	8. Allow the plate to dry in a fume cupboard.	This type of question is likely
03.1	Parts of the procedure are in bold text.	to be fairly rare, to avoid predictable
	For each of these parts, consider whether it is essential and justify	

### **EXAMPLE (A-LEVEL PHYSICS SPECIMEN PAPER 5)**

0 2 . 6 The student decides to confirm the value of the capacitance by first determining the time constant of the circuit when the capacitor discharges through a fixed resistor.

Describe an experiment to do this. Include in your answer:

- a circuit diagram
- an outline of a procedure
- an explanation of how you would use the data to determine the time constant. [4 marks]

This question focuses on a particular aspect of one of the required practicals, and is related to the discharging of a capacitor through a fixed resistor.

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4. Questions applying the skills from the required practical procedures and the apparatus and techniques list.

### EXAMPLE (A-LEVEL BIOLOGY SPECIMEN PAPER 5)

2	Fibrin is a protein. Congo red is a dye that binds to fibrin molecules and colours them red. When a suspension of Congo-red fibrin is digested, the dye goes into solution.
	You are provided with <ul> <li>fibrin powder that has been dyed with Congo red</li> <li>trypsin, an enzyme that hydrolyses fibrin</li> <li>any other laboratory apparatus that you might need.</li> </ul>
	Plan an investigation to the find effect of pH on the rate of hydrolysis of fibrin by Trypsin.
02.1	Describe how you would change the independent variable. Include the steps that you would take to ensure that confounding variables were kept constant and any controls that you would set up.
	[4 marks]

### EXAMPLE (A-LEVEL CHEMISTRY SPECIMEN PAPER 3)

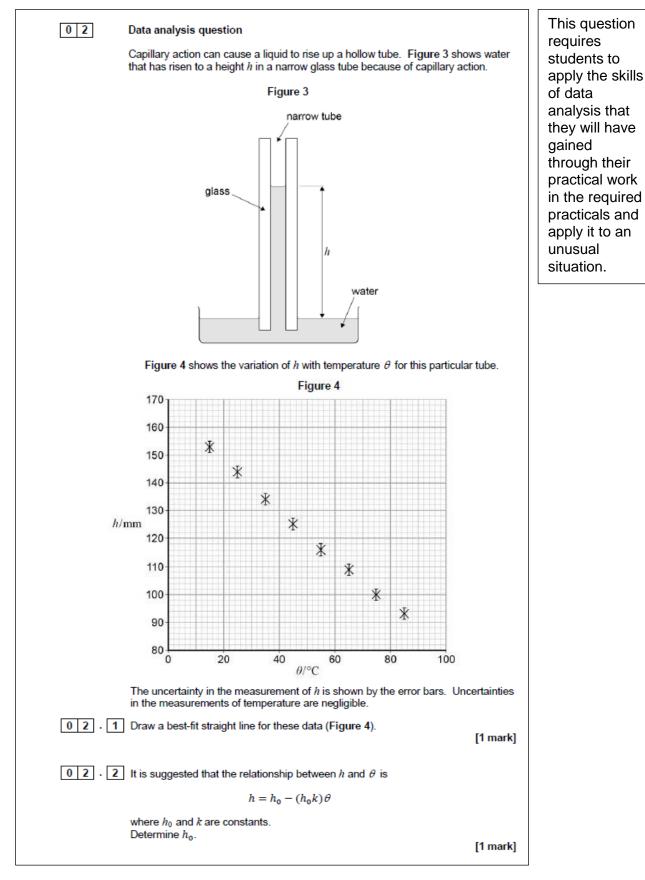
	Tab	le 1	
d	ethanol	ethanal	ethanoic aci
oint / °C	78	21	118

This question expects students to understand distillation which is one of the required practicals. It is not necessary for students to have carried out this precise experiment to understand the requirements.

requirements.

the

This question expects students to understand enzyme controlled reactions, which is one of the required practicals. It is not necessary for students to have used Congo Red to understand



### **EXAMPLE (AS PHYSICS SPECIMEN PAPER 2)**

## GUIDELINES TO SUPPORTING STUDENTS IN PRACTICAL WORK

Developed in collaboration with National Foundation for Educational Research and CLEAPSS

### CLARIFY THE IMPORTANCE OF KEEPING A LAB BOOK OR OTHER RECORDS OF PRACTICAL WORK

Explain that students need a record of their achievements to guide their learning. Lab books also can be an opportunity to develop a skill used both by scientists and in business. They allow students to accurately and clearly record information, ideas and thoughts for future reference which is a very useful life skill.

### WARN STUDENTS AGAINST PLAGIARISM AND COPYING

Explain the meaning of the term plagiarism and that the use of acknowledged sources is an encouraged and acceptable practice, but trying to pass off other people's work as their own is not, and will not help them learn. Show students how sources should be cited.

### **EXPLAIN THE LEARNING CRITERIA FOR EACH SKILL**

This will help students learn and allow them to know when they have met the criteria. The student lab book contains the criteria, but they own the process and have the responsibility for collecting appropriate evidence of success.

### USE CLEARLY DEFINED LEARNING OUTCOMES

For example, if you are running a practical session to teach students how to use a microscope and staining techniques safely and efficiently, then make sure they know why they are learning this. This will also make it much easier for them to know when they have met the criteria.

### START WITH SIMPLE TASKS

Students need to become confident with the apparatus and concepts of practical work before they can proceed to more complicated experiments. It may be more effective to start with simple manipulation skills and progress to the higher order skills.

### TEACH PRACTICAL WORK IN YOUR PREFERRED ORDER

Teach the skills as you see fit and that suit your circumstances – the assessment process is aimed to be flexible and help you teach practical work, not to dictate how it should be done.

### USE FEEDBACK AND PEER ASSESSMENT

Feedback is essential to help students develop skills effectively. Allowing self and peer review will allow time for quality feedback as well as provide powerful learning tools. However, this is a decision for teachers. The scheme is designed to be flexible while promoting best practice.

Research shows that feedback is the best tool for learning in practical skills. Students who normally only receive numerical marks as feedback for work will need to be trained in both giving and receiving comment-based feedback. Provided it is objective, focused on the task and meets learning outcomes, students will quickly value this feedback.

Feedback does not need to be lengthy, but it does need to be done while the task is fresh in the students' mind. Not everything needs written feedback but could be discussed with students, either individually or as a class. For example, if a teacher finds that many students cannot calculate percentage change, the start of the next lesson could be used for a group discussion about this.

The direct assessment of practical work is designed to allow teachers to integrate student-centred learning (including peer review), into day-to-day teaching and learning. This encourages critical skills. Research indicates these are powerful tools for learning. For example, teachers could ask students to evaluate each other's data objectively. The students could identify why some data may be useful and some not. This can be a very good way of getting students to understand why some conventions are used, and what improves the quality of results. This also frees up marking time to concentrate on teaching.

### **DON'T GIVE MARKS**

We have deliberately moved away from banded criteria and marks to concentrate on the mastery of key practical competencies. The purpose of marking should be changed to emphasise learning. Students should find it easier to understand and track their progress, and focus their work. We would expect most students, with practice and the explicit teaching of skills and techniques, to have confidence to approach practical work related exam questions.

### **USE GROUP WORK**

This is a very useful skill, allowing students to build on each other's ideas. For example, planning an experiment can be done as a class discussion. Alternatively, techniques such as snowballing can be used, in which students produce their own plan then sit down in a small group to discuss which are the best collective ideas. From this, they revise their plan which is then discussed to produce a new 'best' plan.

## **USE OF LAB BOOKS**

Students do **not** need to write up every practical that they do in detail. However, it is good practice to have a record of all they do. A lab book could contain this record. It is a student's personal book and may contain a range of notes, tables, jottings, reminders of what went wrong, errors identified and other findings. It is a live document that can function as a learning journal.

Lab books are **not** a requirement of the OxfordAQA AS and A-level specifications in Biology, Chemistry or Physics. They are, however, highly valued by colleagues in higher education.

Each institution has its own rules on lab book usage. The following guidelines are based on those from a selection of companies and universities that use lab books. They are designed to help students and teachers in preparing to use lab books for university but do not represent the only way that books could be used for International AS and A-level sciences. Teachers may wish to vary the following points to suit their purposes.

### THE PURPOSE OF A LAB BOOK

A lab book is a complete record of everything that has been done in the laboratory. As such, it becomes important both to track progress of experiments, and also, in industry and universities, to prove who developed an idea or discovered something first.

A lab book is a:

- source of data that can be used later by the experimenter or others
- complete record of what has been done so that experiments could be understood or repeated by a competent scientist at some point in the future
- tool that supports sound thinking and helps experimenters to question their results to ensure that their interpretation is the same one that others would come to
- record of why experiments were done.

### TYPE OF BOOK

A lab book is often a hard-backed book with bound pages. Spiral bound notebooks are not recommended as it is too easy to rip a page out and start again. It is generally advisable that a lab book has a cover that won't disintegrate the moment it gets slightly wet.

### STYLE

Notes should be recorded as experiments are taking place. They should not be a "neat" record written at a later date from scraps of paper. However, they should be written clearly, in legible writing and in language which can be understood by others.

Many lab books are used in industry as a source of data, and so should be written in indelible ink.

To ensure that an observer can be confident that all data are included when a lab book is examined, there should be no blank spaces. Mistakes should be crossed out and re-written. Numbers should not be overwritten, erased, nor should Tippex be used. Pencil should not be used for anything other than graphs and diagrams.

### EACH PAGE SHOULD BE DATED

Worksheets, graphs, printed information, photographs and even flat "data" such as chromatograms or TLC plates can all be stuck into a lab book. They should not cover up any information so that photocopying the page shows all information in one go. Anything glued in should lie flat and not be folded.

### CONTENT

Generally, lab books will contain:

- title and date of experiment
- notes on the objectives of the experiment notes on the method, including all details (eg temperatures, volumes, settings of pieces of equipment) with justification where necessary
- · estimates of the uncertainty of measurements
- sketches of how equipment has been set up can be helpful. Photographs pasted in are also acceptable
- data and observations input to tables (or similar) while carrying out the experiment
- calculations annotated to show thinking
- graphs and charts
- summary, discussions and conclusions
- cross-references to earlier data and references to external information.

This list and its order are not prescriptive. Many experiments change as they are set up and trials run. Often a method will be given, then some data, then a brief mention of changes that were necessary, then more data and so on.

## **REQUIRED PRACTICAL ACTIVITIES**

The OxfordAQA Exams required practicals have been designed to give students a range of practical experience. Carrying out the required practicals will mean that students will have experienced the use of most of the standard pieces of equipment and techniques expected when students move to further study in the subject at university. Teachers are encouraged to develop students' abilities by inclusion of other opportunities for skills development, as exemplified in the schemes of work for each subject.

Teachers are encouraged to vary their approach to the required practical activities. Some are more suitable for highly structured approaches that develop key techniques. Others allow opportunities for students to develop investigative approaches.

This list is not designed to limit the practical activities carried out by students. A rich practical experience for students will include more than the required practical activities. The explicit teaching of practical skills builds students' competence. Many teachers will also use practical approaches to the introduction of content knowledge in the course of their normal teaching.

### STUDENTS WHO MISS A REQUIRED PRACTICAL ACTIVITY

The required practical activities are part of the specification. As such, exam papers will test knowledge and understanding of the procedures involved and require evaluation of the techniques adopted. Students may need to interpret specimen results.

A student who misses a particular practical activity may be at a disadvantage when answering questions in the exams. It will often be difficult to set up a practical a second time for students to catch up, although if at all possible an attempt should be made. Teachers will need to decide on a case by case basis whether they feel it is important for the student to carry out that particular practical. This is no different from when teachers make decisions about whether to re-teach a particular topic if a student is away from class when it is first taught.

### CHEMISTRY REQUIRED PRACTICAL ACTIVITIES

AS	S practical activities	A2	practical activities
list kn pro teo	udents must carry out the practical activities ted below. The AS written papers test owledge and understanding of the ocedures as well as evaluation of the chniques adopted. They may require udents to interpret specimen results.	Students must carry out the practical activities below. The A2 written papers test knowledge and understanding of the procedures involved, as well as evaluation of the techniques adopted. They may require students to interpret specimen results.	
Pr	actical activity	Practical activity	
1	Make up a volumetric solution and carry out a simple acid-base titration.	6	Investigate how pH changes when a weak acid reacts with a strong base and when a strong acid reacts with a weak base.
2	Measure an enthalpy change.	7	Measure the rate of reaction by an initial
3	Carry out simple test-tube reactions to identify:	1	rate method and a continuous monitoring method.
	<ul> <li>cations – Group 2, NH<sub>4</sub><sup>+</sup></li> <li>anions – Group 7 (halide ions), OH<sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup></li> </ul>	8	Measure the electromotive force (EMF) of an electrochemical cell.
4	Distil a product from a reaction.	9	Carry out simple test–tube reactions to identify transition metal ions in aqueous solution.
5	Carry out tests for alcohols, aldehydes, alkenes and carboxylic acids.	10	Prepare a pure organic solid and test its purity.

## TABULATING DATA

It is important to keep a record of data while carrying out practical work. Tables should have clear headings with units indicated using a forward slash before the unit.

Time/min	Temperature/°C
0	14.8
1	14.7
2	14.6

Although using a forward slash (solidus) is the standard format for post-16 studies, other formats are generally acceptable. For example:

Volume in cm <sup>3</sup>	Time taken in s
15	23
25	45
35	56

Concenti (mol dr	Time (s)
1.0	152
1.5	93
2.0	54

It is good practice to draw a table before an experiment commences and then enter data straight into the table. This can sometimes lead to data points being in the wrong order. For example, when investigating the pH change in an acid-base titration, a student may do a number of pH measurements at 10, 20, 25, 30 and 35 cm<sup>3</sup> of reagent added, and then investigate the range between 20 and 30 further by adding readings at 22, 24, 24.5, 25, 25, 26, 28. Whilst this is perfectly acceptable, it is generally a good idea to make a copy of the table in ascending order of volume to enable patterns to be spotted more easily. Reordered tables should follow the original data if using a lab book, data should not be noted down in rough before it is written up.

It is also expected that the independent variable is the left hand column in a table, with the following columns showing the dependent variables. These should be headed in similar ways to measured variables. The body of the table should not contain units.

### TABULATING LOGARITHMIC VALUES

When the logarithm is taken of a physical quantity, the resulting value has no unit. However, it is important to be clear about which unit the quantity had to start with. The logarithm of a time in seconds will be very different from the logarithm of the same time in minutes.

These should be included in tables in the following way:

Reading number	time/s	log (time/s)
1	2.3	0.36
2	3.5	0.54
3	5.6	0.75

## **SIGNIFICANT FIGURES**

Data should be written in tables to the same number of significant figures. This number should be determined by the resolution of the device being used to measure the data or the uncertainty in measurement. For example, a sample labelled as "1 mol dm<sup>-3</sup> acid" should not be recorded in a table of results as 1.0 mol dm<sup>-3</sup> acid.

There is sometimes confusion over the number of significant figures when readings cross multiples of 10. Changing the number of decimal places across a power of ten retains the number of significant figures **but changes the precision.** The same number of decimal places should therefore generally be used, as illustrated below.

0.97	99.7
0.98	99.8
0.99	99.9
1.00	100.0
1.10	101.0

It is good practice to write down all digits showing on a digital meter.

Calculated quantities should be shown to the number of significant figures of the data with the least number of significant figures.

### Example:

Calculate the concentration, in mol dm<sup>-3</sup>, of a solution of sodium hydroxide that contains 0.28 mol of NaOH in 465 cm<sup>3</sup> of water.

Concentration =  $\frac{0.28}{475} \times 1000 = 0.59$ 

Note that the concentration can only be quoted to two significant figures as the number of moles is only quoted to two significant figures.

Equipment measuring to half a unit (eg a thermometer measuring to 0.5 °C) should have measurements recorded to one decimal place (eg 1.0 °C, 2.5 °C). The uncertainty in these measurements would be  $\pm 0.25$ , but this would be rounded to the same number of decimal places (giving measurements quoted with uncertainty of  $(1.0 \pm 0.3)$  °C etc).

## UNCERTAINTIES

### SOURCES OF UNCERTAINTIES

Students should know that every measurement has some inherent uncertainty.

The important question to ask is whether an experimenter can be confident that the true value lies in the range that is predicted by the uncertainty that is quoted. Good experimental design will attempt to reduce the uncertainty in the outcome of an experiment. The experimenter will design experiments and procedures that produce the least uncertainty and to provide a realistic uncertainty for the outcome.

In assessing uncertainty, there are a number of issues that have to be considered. These include:

- the resolution of the instrument used
- the manufacturer's tolerance on instruments
- the judgments that are made by the experimenter
- the procedures adopted (eg repeated readings)
- the size of increments available (eg the size of drops from a pipette).

Numerical questions will look at a number of these factors. Often, the resolution will be the guiding factor in assessing a numerical uncertainty. There may be further questions that would require candidates to evaluate arrangements and procedures. Students could be asked how particular procedures would affect uncertainties and how they could be reduced by different apparatus design or procedure.

A combination of the above factors means that there can be no hard and fast rules about the actual uncertainty in a measurement. What we can assess from an instrument's resolution is the **minimum** possible uncertainty. Only the experimenter can assess the other factors, based on the arrangement and use of the apparatus. A rigorous experimenter would draw attention to these factors and take them into account.

### **READINGS AND MEASUREMENTS**

It is useful, when discussing uncertainties, to separate measurements into two forms.

- Readings: the values found from a single judgement when using a piece of equipment.
- Measurements: the values taken as the difference between the judgements of two values.

#### **Examples**

When using a thermometer, a student only needs to make one judgement (the height of the liquid). This is a reading. It can be assumed that the zero value has been correctly set.

For burettes and rulers, both the starting point and the end point of the measurement must be judged, leading to two uncertainties

The following list is not exhaustive, and the way that the instrument is used will determine whether the student is taking a reading or a measurement.

Reading (one judgement only)	Measurement (two judgements required)
thermometer	ruler
pH meter	stopwatch
top pan balance	analogue meter
measuring cylinder	burette
volumetric flask	

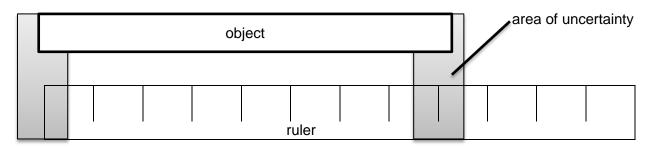
The uncertainty in a **reading** when using a particular instrument is **no smaller** than plus or minus half of the smallest division or greater. For example, a temperature measured with a thermometer is likely to have an uncertainty of  $\pm 0.5$  °C if the graduations are 1 °C apart.

Students should be aware that readings are often written with the uncertainty. An example of this would be to write a voltage as  $(2.40 \pm 0.01)$  V. It is usual for the uncertainty quoted to be the same number of decimal places as the value. Unless there are good reasons otherwise (eg an advanced statistical analysis), students at this level should quote the uncertainty in a measurement to the same number of decimal places as the value.

### Measurement example: length

When measuring length, **two** uncertainties must be included: the uncertainty of the placement of the zero of the ruler and the uncertainty of the point the measurement is taken from.

As both ends of the ruler have a  $\pm 0.5$  scale division uncertainty, the measurement will have an uncertainty of  $\pm 1$  division.



For most rulers, this will mean that the uncertainty in a measurement of length will be ±1 mm.

This "initial value uncertainty" will apply to any instrument where the user can set the zero (incorrectly), but would not apply to equipment such as balances or thermometers where the zero is set at the point of manufacture.

In summary

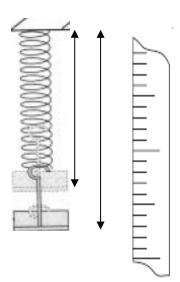
- The uncertainty of a reading (one judgement) is at least ±0.5 of the smallest scale reading.
- The uncertainty of a measurement (two judgements) is at least ±1 of the smallest scale reading.

The way measurements are taken can also affect the uncertainty.

### Measurement example: the extension of a spring

Measuring the extension of a spring using a metre ruler can be achieved in two ways

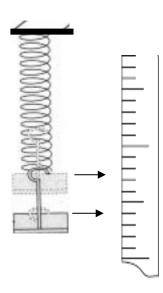
1. Measuring the total length unloaded and then loaded.



Four readings must be taken for this: the start and end point of the unloaded spring's length and the start and end point of the loaded spring's length.

The minimum uncertainty in each measured length is  $\pm 1$  mm using a meter ruler with 1 mm divisions (the actual uncertainty is likely to be larger due to parallax in this instance). The extension would be the difference between the two readings, so the minimum uncertainty would be  $\pm 2$  mm.

2. Fixing one end and taking a scale reading of the lower end.



Two readings must be taken for this: the end point of the unloaded spring's length and the end point of the loaded spring's length. The start point is assumed to have zero uncertainty, as it is fixed.

The minimum uncertainty in each reading would be  $\pm 0.5$  mm, so the minimum extension uncertainty would be  $\pm 1$  mm.

Even with other practical uncertainties this second approach would be better.

Realistically, the uncertainty would be larger than this and an uncertainty in each reading of 1 mm or would be more sensible. This depends on factors such as how close the ruler can be mounted to the point as at which the reading is to be taken.

### **OTHER FACTORS**

There are some occasions where the resolution of the instrument is not the limiting factor in the uncertainty in a measurement.

Best practice is to write down the full reading and then to write to fewer significant figures when the uncertainty has been estimated.

#### Examples:

A stopwatch has a resolution of hundredths of a second, but the uncertainty in the measurement is more likely to be due to the reaction time of the experimenter. Here, the student should write the full reading on the stopwatch (eg 12.20 s), carry the significant figures through for all repeats, and reduce this to a more appropriate number of significant figures after an averaging process later.

If a student measures the length of a piece of wire, it is very difficult to hold the wire completely straight against the ruler. The uncertainty in the measurement is likely to be higher than the  $\pm 1$  mm uncertainty of the ruler. Depending on the number of "kinks" in the wire, the uncertainty could be reasonably judged to be nearer  $\pm 2$  or 3 mm.

The uncertainty of the reading from digital voltmeters and ammeters depends on the electronics and is not strictly the last figure in the readout. Manufacturers usually quote the percentage uncertainties for the different ranges. Unless otherwise stated it may be assumed that  $\pm 0.5$  in the least significant digit is to be the uncertainty in the measurement. This would generally be rounded up to  $\pm 1$  of the least significant digit when quoting the value and the uncertainty together. For example (5.21  $\pm 0.01$ ) V. If the reading fluctuates, then it may be necessary to take a number of readings and do a mean and range calculation.

### **UNCERTAINTIES IN GIVEN VALUES**

In written exams, students can assume the uncertainty to be  $\pm 1$  in the last significant digit. For example, if a boiling point is quoted as being 78 °C, the uncertainty could be assumed to be  $\pm 1$  °C. The uncertainty may be lower than this but without knowing the details of the experiment and procedure that lead to this value there is no evidence to assume otherwise.

#### **REPEATED MEASUREMENTS**

Repeating a measurement is a method for reducing the uncertainty.

With many readings one can also identify those that are exceptional (that are far away from a significant number of other measurements). Sometimes it will be appropriate to remove outliers from measurements before calculating a mean. On other occasions, particularly in Biology, outliers are important to include. For example, it is important to know that a particular drug produces side effects in one person in a thousand.

If measurements are repeated, the uncertainty can be calculated by finding half the range of the measured values.

#### For example:

<b>Distance/m</b> 1.23 (1.32) 1.27 (1.22)	Repeat	1	2	3	4
	Distance/m	1.23	( 1.32 )	1.27	( 1.22 )

1.32 - 1.22 = 0.10 therefore

Mean distance: (1.26 ± 0.05) m

### PERCENTAGE UNCERTAINTIES

The percentage uncertainty in a measurement can be calculated using:

percentage uncertainty =  $\frac{\text{uncertainty}}{\text{value}} \times 100\%$ 

The percentage uncertainty in a repeated measurement can also be calculated using:

percentage uncertainty =  $\frac{\text{uncertainty}}{\text{mean value}} \times 100\%$ 

### **Further examples:**

Example 1. Some values for diameter of a wire

Repeat	1		2	3		4	
Diameter/mm	0.35	(	0.37	0.36	(	0.34	)

The exact values for the mean is 0.36 mm and for the uncertainty is 0.015 mm.

This could be quoted as such or recorded as  $0.36 \pm 0.02$  mm given that there is a wide range and only 4 readings. Given the simplistic nature of the analysis then giving the percentage uncertainty as 5% or 6% would be acceptable.

Example 2. Different values for the diameter of a wire

Repeat	1	2	3	
Diameter/mm	0.35	0.36	0.35	

The mean here is 0.35 mm with uncertainty of 0.005 mm.

The percentage uncertainty is 1.41% so may be quoted as 1% but really it would be better to obtain further data.

### TITRATION

Titration is a special case where a number of factors are involved in the uncertainties in the measurement.

Students should carry out a rough titration to determine the amount of titrant needed. This is to speed up the process of carrying out multiple samples. The value of this titre should be ignored in subsequent calculations.

In titrations one single titre is never sufficient. The experiment is usually done until there are at least two titres that are concordant ie within a certain allowable range, often 0.10 cm<sup>3</sup>. These values are then averaged.

#### For example:

Titration	Rough	1	2	3
Final reading	24.20	47.40	24.10	47.35
Initial reading	0.35	24.20	0.65	24.10
Titre/cm <sup>3</sup>	23.85	23.20	23.45	23.25

Here, titres 1 and 3 are within the allowable range of 0.10 cm<sup>3</sup> so are averaged to 23.23 cm<sup>3</sup>.

Unlike in some Biology experiments (where anomalous results are always included unless there is good reason not to), in Chemistry it is assumed that repeats in a titration should be concordant. If they are not, there is likely to have been some experimental error. For example, the wrong volume of solution added from the burette, the wrong amount of solution measuring the pipette or the end point might have been misjudged.

The total error in a titre is caused by three factors:

Error	Uncertainty
Reading the burette at the start of the titration	Half a division = $\pm 0.05$ cm <sup>3</sup>
Reading the burette at the end of the titration	Half a division = $\pm 0.05$ cm <sup>3</sup>
Judging the end point to within one drop	Volume of a drop = $\pm 0.05$ cm <sup>3</sup>
Total	± 0.15 cm <sup>3</sup>

This will, of course, depend on the glassware used, as some burettes are calibrated to a higher accuracy than others.

### **UNCERTAINTIES IN EXAMS**

Wherever possible, questions in exams will be clear on whether students are being asked to calculate the uncertainty of a reading, a measurement, or given data.

Where there is ambiguity, mark schemes will allow alternative sensible answers and credit clear thinking.

It is important that teachers read the reports on the examination following each series to understand common mistakes to help their students improve in subsequent years.

### **UNCERTAINTIES IN PRACTICAL WORK**

Students are expected to develop an understanding of uncertainties in measurements through their practical work.

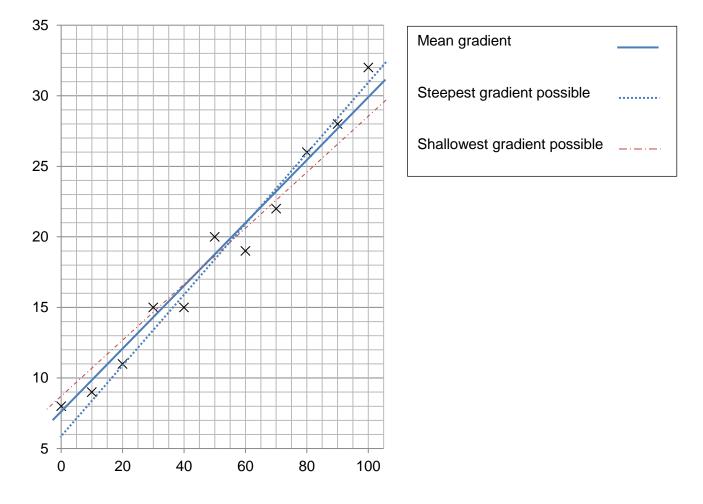
### **UNCERTAINTIES FROM GRADIENTS**

To find the uncertainty in a gradient, two lines should be drawn on the graph. One should be the "best" line of best fit. The second line should be the steepest or shallowest gradient line of best fit possible from the data. The gradient of each line should then be found.

The uncertainty in the gradient is found by:

percentage uncertainty = 
$$\frac{1}{2} \left( \frac{\text{steeper gradient - shallowest gradient}}{\text{mean gradient}} \right)$$

Note the modulus bars meaning that this percentage will always be positive.



In the same way, the percentage uncertainty in the y-intercept can be found:

percentage uncertainty =  $\frac{1}{2}$  ( $\frac{\text{greatest intercept -smallest intercept}}{\text{mean intercept}}$ )

### **COMBINING UNCERTAINTIES**

Combination	Operation	Example
Adding or subtracting values a = b + c	Add the absolute uncertainties $\Delta a = \Delta b + \Delta c$	Initial volume in burette = $3.40 \pm 0.05 \text{ cm}^3$ Final volume in burette = $28.50 \pm 0.05 \text{ cm}^3$ Titre = $25.10 \pm 0.10 \text{ cm}^3$
Multiplying values $a = b \times c$	Add the percentage uncertainties εa = εb + εc	Mass = $50.0 \pm 0.1 \text{ g}$ Temperature rise (T) = $10.9 \pm 0.1 \text{ °C}$ Percentage uncertainty in mass = $0.20\%$ Percentage uncertainty in T = $0.92\%$ Heat change = $2278 \text{ J}$ Percentage uncertainty in heat change = $1.12\%$ Absolute uncertainty in heat change = $\pm 26 \text{ J}$ (Note – the uncertainty in specific heat is taken to be zero)
Dividing values $a = \frac{b}{c}$	Add the percentage uncertainties εa = εb + εc	Mass of salt in solution= $100 \pm 0.1$ g Volume of solution = $250 \pm 0.5$ cm <sup>3</sup> Percentage uncertainty in mass = $0.1\%$ Percentage uncertainty in volume = $0.2\%$ Concentration of solution = $0.400$ g cm <sup>-3</sup> Percentage uncertainty of concentration = $0.3\%$ Absolute uncertainty of concentration = $\pm 0.0012$ g cm <sup>-3</sup>
Power rules $a = b^c$	Multiply the percentage uncertainty by the power εa = c × εb	Concentration of H <sup>+</sup> ions = $0.150 \pm 0.001 \text{ mol dm}^{-3}$ rate of reaction = $k[\text{H}^+]^2 = 0.207 \text{ mol dm}^{-3} \text{ s}^{-1}$ (Note – the uncertainty in <i>k</i> is taken as zero and its value in this reaction is $0.920 \text{ dm}^6 \text{ mol}^{-2} \text{ s}^{-1}$ ) Percentage uncertainty in concentration = $0.67\%$ Percentage uncertainty in rate = $1.33\%$ Absolute uncertainty in rate = $\pm 0.003 \text{ mol dm}^{-3} \text{ s}^{-1}$

Percentage uncertainties should be combined using the following rules:

Note: Absolute uncertainties (denoted by  $\Delta$ ) have the same units as the quantity.

Percentage uncertainties have no units.

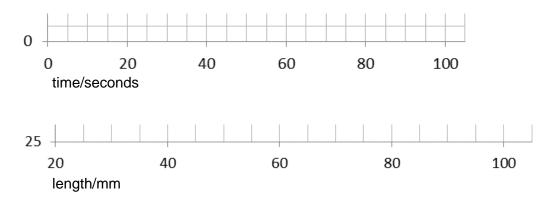
Uncertainties in trigonometric and logarithmic functions will not be tested in A-level exams.

## GRAPHING

Graphing skills can be assessed in the written papers for the International AS and A-level grade. Students should recognise that the type of graph that they draw should be based on an understanding of the type of data they are using and the intended analysis of the data. The rules below are guidelines which will vary according to the specific circumstances.

### LABELLING AXES

Axes should always be labelled with the variable being measured and the units. These should be separated with a forward slash (solidus):



Axes should not be labelled with the units on each scale marking.

### **DATA POINTS**

Data points should be marked with a cross. Both  $\times$  and + marks are acceptable, but care should be taken that data points can be seen against the grid.

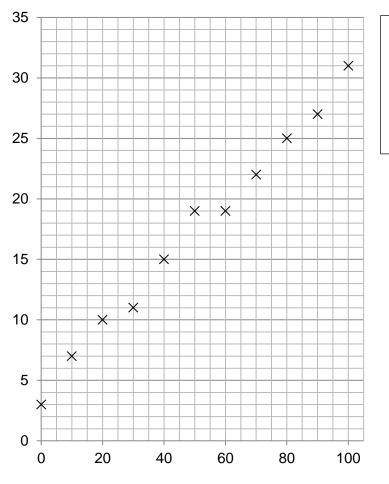
Error bars, standard deviation and ranges can take the place of data points where appropriate.

### SCALES AND ORIGINS

Students should attempt to spread the data points on a graph as far as possible without resorting to scales that are difficult to deal with. Students should consider:

- the maximum and minimum values of each variable
- the size of the graph paper
- whether 0.0 should be included as a data point
- how to draw the axes without using difficult scale markings (eg multiples of 3, 7, 11 etc)
- in exams, the plots should cover at least half of the grid supplied for the graph.

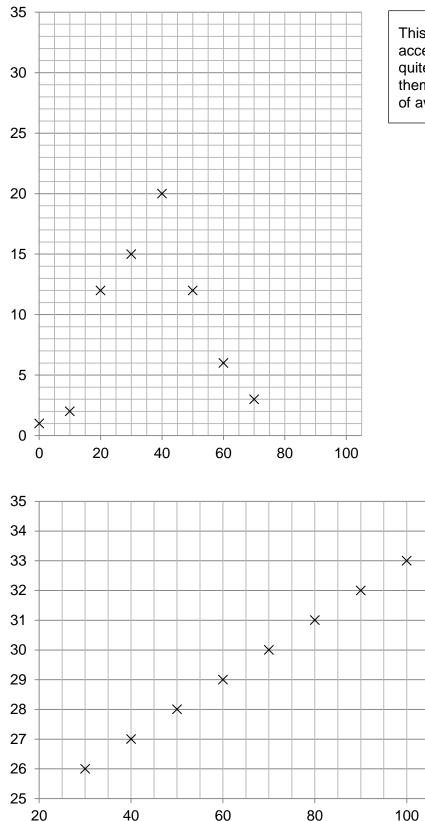
Please note that in the Graphing section, many generic graphs are used to illustrate the points made. For example, the following three graphs are intended to illustrate the information above relating to the spread of data points on a graph. Students producing such graphs on the basis of real practical work or in examination questions would be expected to add in axes labels and units.



This graph has well-spaced marking points and the data fills the paper.

Each point is marked with a cross (so points can be seen even when a line of best fit is drawn).

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This graph is on the limit of acceptability. The points do not quite fill the page, but to spread them further would result in the use of awkward scales.

> At first glance, this graph is well drawn and has spread the data out sensibly.

However, if the graph were to later be used to calculate the equation of the line, the lack of a y-intercept could cause problems. Increasing the axes to ensure all points are spread out but the y-intercept is also included is a skill that requires practice and may take a couple of attempts.

### LINES OF BEST FIT

Lines of best fit should be drawn when appropriate. Students should consider the following when deciding where to draw a line of best fit:

- are the data likely to be following an underlying equation (for example, a relationship governed by a physical law)? This will help decide if the line should be straight or curved
- are there any anomalous results?
- are there uncertainties in the measurements? The line of best fit should fall within error bars, if drawn.

There is no definitive way of determining where a line of best fit should be drawn. A good rule of thumb is to make sure that there are as many points on one side of the line as the other. Often the line should pass through, or very close to, the majority of plotted points. Graphing programs can sometimes help, but tend to use algorithms that make assumptions about the data that may not be appropriate.

Lines of best fit should be continuous and drawn as a thin pencil that does not obscure the points below and does not add uncertainty to the measurement of gradient of the line.

Not all lines of best fit go through the origin. Students should ask themselves whether a 0 in the independent variable is likely to produce a 0 in the dependent variable. This can provide an extra and more certain point through which a line must pass. A line of best fit that is expected to pass through (0,0), but does not, would imply some systematic error in the experiment. This would be a good source of discussion in an evaluation.

### DEALING WITH ANOMALOUS RESULTS

At International GCSE, students are often taught automatically to ignore anomalous results. At International AS and A-level, students should think carefully about what could have caused the unexpected result and therefore whether it is anomalous. A student might be able to identify a reason for the unexpected result and so validly regard it as an anomaly. For example, an anomalous result might be explained by a different experimenter making the measurement, a different solution or a different measuring device being used. In the case where the reason for an anomalous result occurring can be identified, the result should be recorded and plotted but may then be ignored.

Anomalous results should also be ignored where results are expected to be the same.

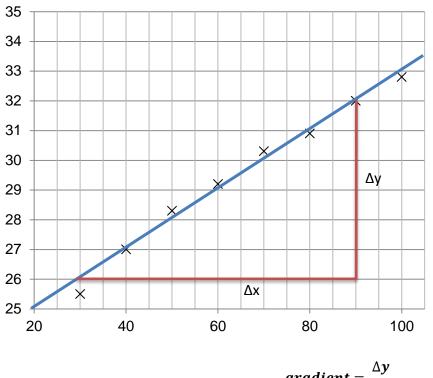
Where there is no obvious error and no expectation that results should be the same, anomalous results should be included. This will reduce the possibility that a key point is being overlooked.

Please note: when recording results it is important that all data are included. Anomalous results should only be ignored at the data analysis stage.

It is best practice whenever an anomalous result is identified for the experiment to be repeated. This highlights the need to tabulate and even graph results as an experiment is carried out.

### **MEASURING GRADIENTS**

When finding the gradient of a line of best fit, students should show their working by drawing a triangle on the line. The hypotenuse of the triangle should be at least half as big as the line of best fit.



The line of best fit here has an equal number of points on both sides. It is not too wide so points can be seen under it.

The gradient triangle has been drawn so the hypotenuse includes more than half of the line.

In addition, it starts and ends on points where the line of best fit crosses grid lines so the points can be read easily (this is not always possible).

$$gradient = \frac{\Delta y}{\Delta x}$$

When finding the gradient of a curve, eg the rate of reaction at a time that was not sampled, students should draw a tangent to the curve at the relevant value of the independent variable (xaxis).

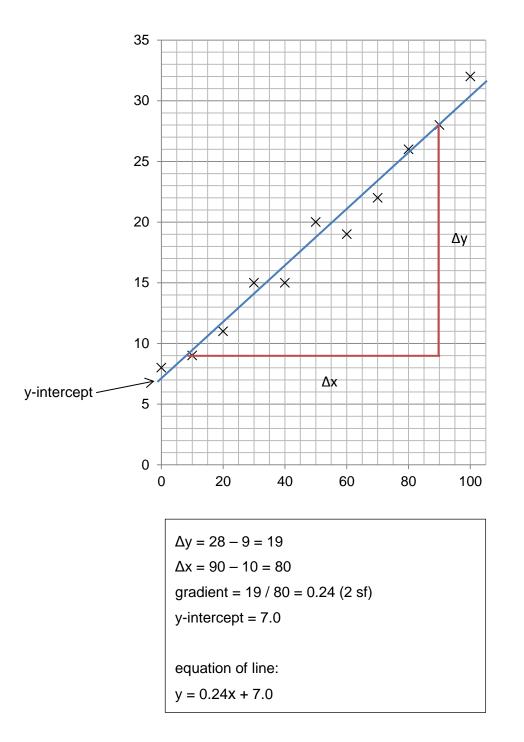
Use of a set square to draw a triangle over this point on the curve can be helpful in drawing an appropriate tangent.

### THE EQUATION OF A STRAIGHT LINE

Students should be able to translate graphical data into the equation of a straight line.

### y = mx + c

Where y is the dependent variable, m is the gradient, x is the independent variable and c is the y-intercept.

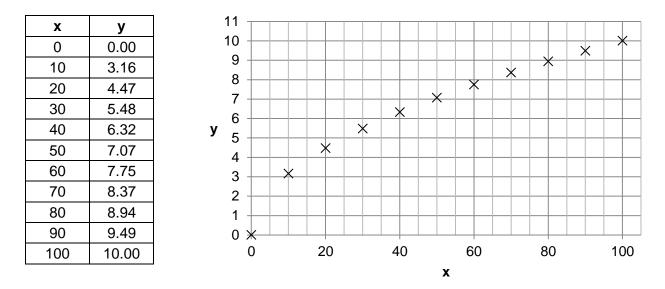


#### **TESTING RELATIONSHIPS**

Sometimes it is not clear what the relationship between two variables is. A quick way to find a possible relationship is to manipulate the data to form a straight line graph from the data by changing the variable plotted on each axis.

For example:

#### Raw data and graph

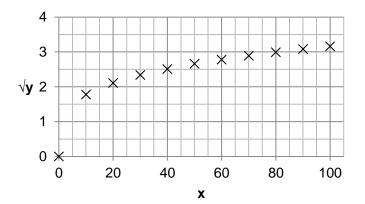


This is clearly not a straight line graph. The relationship between x and y is not clear.

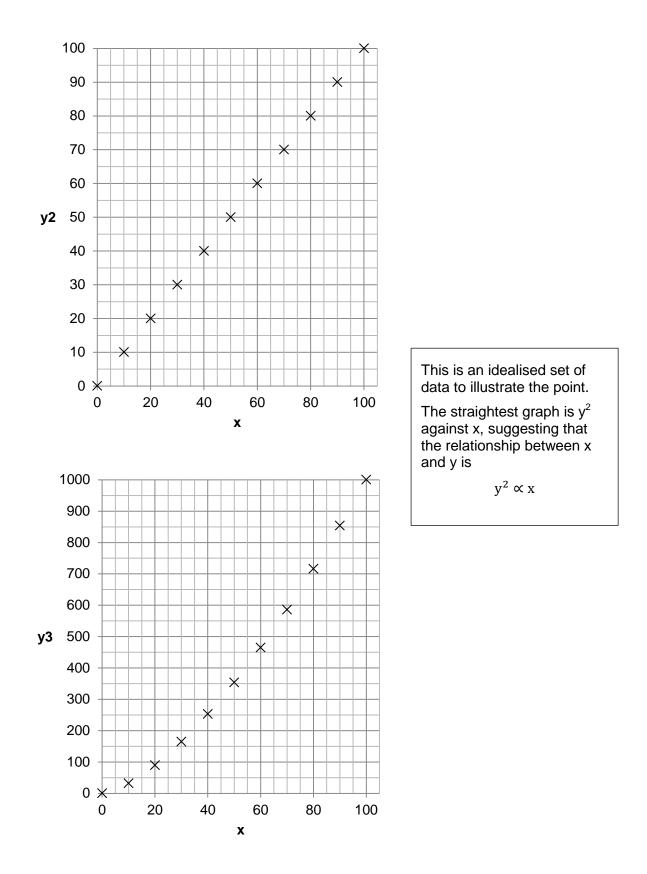
#### MANIPULATED DATA AND GRAPHS

A series of different graphs can be drawn from these data. The one that is closest to a straight line is a good candidate for the relationship between x and y.

X	У	√y	y <sup>2</sup>	y <sup>3</sup>
0	0.00	0.00	0.00	0.00
10	3.16	1.78	10.00	32
20	4.47	2.11	20.00	89
30	5.48	2.34	30.00	160
40	6.32	2.51	40.00	250
50	7.07	2.66	50.00	350
60	7.75	2.78	60.00	470
70	8.37	2.89	70.00	590
80	8.94	2.99	80.00	720
90	9.49	3.08	90.00	850
100	10.00	3.16	100.00	1000



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#### MORE COMPLEX RELATIONSHIPS

Graphs can be used to analyse more complex relationships by rearranging the equation into a form similar to y=mx+c.

#### Example one: testing power laws

A relationship is known to be of the form  $y=Ax^n$ , but n is unknown.

Measurements of y and x are taken.

A graph is plotted with log(y) plotted against log(x).

The gradient of this graph will be n, with the y intercept log(A), as log(y) = n(log(x)) + log(A)

#### Example two

The equation that relates the rate constant of a reaction to temperature is

$$k = Ae^{-\frac{E_a}{RT}}$$

This can be rearranged into

$$\ln(k) = -\frac{E_a}{R} \left(\frac{1}{T}\right) + \ln A$$

So a graph of  $\ln(k)$  against  $\left(\frac{1}{T}\right)$  should be a straight line, with a gradient of  $-\frac{E_a}{R}$  and a y-intercept of lnA

# SUBJECT SPECIFIC VOCABULARY

## THE LANGUAGE OF MEASUREMENT

The following subject specific vocabulary provides definitions of key terms used in our International AS and A-level science specifications.

#### ACCURACY

A measurement result is considered accurate if it is judged to be close to the true value.

#### CALIBRATION

Marking a scale on a measuring instrument. This involves establishing the relationship between indications of a measuring instrument and standard or reference quantity values, which must be applied.

For example, placing a thermometer in melting ice to see whether it reads 0 °C, in order to check if it has been calibrated correctly.

#### DATA

Information, either qualitative or quantitative, that has been collected.

#### ERRORS

See also uncertainties.

#### **Measurement error**

The difference between a measured value and the true value.

#### Anomalies

These are values in a set of results which are judged not to be part of the variation caused by random uncertainty.

#### **Random error**

These cause readings to be spread about the true value, due to results varying in an unpredictable way from one measurement to the next.

Random errors are present when any measurement is made, and cannot be corrected. The effect of random errors can be reduced by making more measurements and calculating a new mean.

#### Systematic error

These cause readings to differ from the true value by a consistent amount each time a measurement is made.

Sources of systematic error can include the environment, methods of observation or instruments used.

Systematic errors cannot be dealt with by simple repeats. If a systematic error is suspected, the data collection should be repeated using a different technique or a different set of equipment, and the results compared.

#### Zero error

Any indication that a measuring system gives a false reading when the true value of a measured quantity is zero, eg the needle on an ammeter failing to return to zero when no current flows. A zero error may result in a systematic uncertainty.

#### **EVIDENCE**

Data which has been shown to be valid.

#### **FAIR TEST**

A fair test is one in which only the independent variable has been allowed to affect the dependent variable.

#### **HYPOTHESIS**

A proposal intended to explain certain facts or observations.

#### **INTERVAL**

The quantity between readings, eg a set of 11 readings equally spaced over a distance of 1 metre would give an interval of 10 centimetres.

#### PRECISION

Precise measurements are ones in which there is very little spread about the mean value. Precision depends only on the extent of random errors – it gives no indication of how close results are to the true value.

#### PREDICTION

A prediction is a statement suggesting what will happen in the future, based on observation, experience or a hypothesis.

#### RANGE

The maximum and minimum values of the independent or dependent variables; important in ensuring that any pattern is detected.

For example a range of distances may be quoted as either: 'from 10 cm to 50 cm' or 'from 50 cm to 10 cm'

#### REPEATABLE

A measurement is repeatable if the original experimenter repeats the investigation using same method and equipment and obtains the same results.

#### REPRODUCIBLE

A measurement is reproducible if the investigation is repeated by another person, or by using different equipment or techniques, and the same results are obtained.

#### RESOLUTION

This is the smallest change in the quantity being measured (input) of a measuring instrument that gives a perceptible change in the reading.

#### SKETCH GRAPH

A line graph, not necessarily on a grid, that shows the general shape of the relationship between two variables. It will not have any points plotted and although the axes should be labelled they may not be scaled.

#### **TRUE VALUE**

This is the value that would be obtained in an ideal measurement.

#### UNCERTAINTY

The interval within which the true value can be expected to lie, with a given level of confidence or probability, eg "the temperature is 20 °C  $\pm$  2 °C, at a level of confidence of 95%.

#### VALIDITY

Suitability of the investigative procedure to answer the question being asked. For example, an investigation to find out if the rate of a chemical reaction depended upon the concentration of one of the reactants would not be a valid procedure if the temperature of the reactants was not controlled.

#### VALID CONCLUSION

A conclusion supported by valid data, obtained from an appropriate experimental design and based on sound reasoning.

#### VARIABLES

These are physical, chemical or biological quantities or characteristics.

#### **Categoric variables**

Categoric variables have values that are labels, eg names of plants or types of material.

#### **Continuous variables**

Continuous variables can have values (called a quantity) that can be given a magnitude either by counting (as in the case of the number of shrimp) or by measurement (eg light intensity, flow rate etc).

#### **Control variables**

A control variable is one which may, in addition to the independent variable, affect the outcome of the investigation and therefore has to be kept constant or at least monitored.

#### **Dependent variables**

The dependent variable is the variable of which the value is measured for each and every change in the independent variable.

#### Independent variables

The independent variable is the variable for which values are changed or selected by the investigator.

#### Nominal variables

A nominal variable is a type of categoric variable where there is no ordering of categories (eg red flowers, pink flowers, blue flowers).

# REQUIRED PRACTICAL ACTIVITIES: EXEMPLAR EXPERIMENTS

During the development of our International AS and A-levels in Biology, Chemistry and Physics, we have spoken to hundreds of teachers. Teachers helped us to develop every part of the specification, including its contents and layout, what is examined in which paper and the question types we include. Teachers also helped us to decide which practical activities to include in our required practicals for each subject.

Teachers asked us for full, comprehensive instructions on how to carry out each of the 10 required practicals. In response, we have included a **sample** method for each practical in this section. These have been prepared so that a reasonably equipped school can cover the required activity with their students. It gives **one possible version** of the experiment that teachers could use. They will help inform planning the laboratory time required and helping schools to ensure they have the right equipment. Many are based on existing tasks as we know that they work well and schools have been using them for a number of years in the current AQA UK specifications.

This document should only be seen as a starting point. We do not intend to stifle innovation and would encourage teachers to try different methods. Students will not be examined on the specific practical work exemplified within this section but on the skills and understanding they build up through their practical work. Teachers can vary all experiments to suit their and their students' needs.

#### SAFETY

At all times, the teacher is responsible for safety in the classroom. Teachers should intervene whenever they see unsafe working. Risk assessments should be carried out before working, and advice from CLEAPSS and other organisations should be followed.

It is appropriate to give students at International AS and A-level more independence when making decisions about safety. They should be taught how to assess risks and how to write risk assessments when appropriate. They should also understand the appropriate use of safety equipment and how to put measures in place to reduce risks.

# To support teachers further, Mary Philpott, Biology Adviser, previously from CLEAPSS, outlines the difference between identification of major hazards, associated risk and control measures and a full risk assessment:

The risk assessment should always be complete, as it is this that prevents injury or ill-health.

The risk assessment is fundamentally the **thinking** that has taken place before and during an activity, so that any foreseeable risk is reduced to a minimum. A written record is necessary only to show that the thinking has taken place.

We tend to get caught up in the paperwork that provides evidence for the risk assessment, but the guidance from the Health and Safety Executive is that the written record should be on a **point-of-use document** and there is no particular form etc that needs to be filled in.

The tables/forms etc that many schools use are simply planning documents that the teachers use to provide the point of use risk assessment for each of their lessons. Incidentally, CLEAPSS members must refer to our current advice when preparing their point-of-use documents.

The student is not responsible for their risk assessment. In a large part, therefore, the student's risk assessment will be that they carry through the safety measures that the teacher has put in place. It is therefore fine if the student makes a note on their point-of use document that shows they have thought about how to behave safely, and carried it through. The teacher will also be able to record

what they have seen in a practical that shows that the student's risk assessment is effective. For example, the student's written risk assessment could be as simple as making notes on a method sheet about where they will put on eye protection or how they will arrange any heating equipment so that there is a minimum risk of scalding or burning themselves or the person next to them.

The teacher's observation notes will refer to whether they have carried out their written plans.

It might help the students to think safely if the teacher gives them a little time at the start of each practical to highlight or make notes about the safety aspects, and a class discussion about safety could show up any safety aspects that perhaps the teacher had not considered.

The students may also note where they have reminded other students about any safety issues.

If the students are planning their own practical activities, they could use the safety advice given in the CLEAPSS Student Safety Sheets. In this case, they could identify hazards, risks and control measures.

In this case, they would make their own point of use document, with the control measures clearly identified.

The teacher would need to check that the risk assessment is adequate before they let the students proceed with the activity.

These are examples of 10 experiments that can be done as part of the International AS/A-level Chemistry course. The methods are written using commonly used reagents and techniques, although teachers can modify the methods and reagents as desired.

#### TRIALLING

All practicals should be trialled before use with students.

#### **RISK ASSESSMENT AND RISK MANAGEMENT**

Risk assessment and risk management are the responsibility of the centre.

Safety is the responsibility of the teacher and the centre. It is important that students are taught to act safely in the laboratory at all times, including the wearing of goggles at all times and the use of additional safety equipment where appropriate.

#### **NOTES FROM CLEAPSS**

Technicians/teachers should follow CLEAPSS guidance, particularly that found on Hazcards and recipe sheets. The worldwide regulations covering the labelling of reagents by suppliers are currently being changed. Details about these changes can be found in leaflet GL101, which is available on the CLEAPSS website. You will need to have a CLEAPSS login.

Make up a volumetric solution and carry out a simple acid-base titration: To prepare a solution of sodium hydrogensulfate that has a known concentration.

### TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

#### REQUIREMENTS

In addition to general laboratory apparatus, each student needs:

- weighing bottle or boat
- 250 cm<sup>3</sup> volumetric (graduated) flask
- sodium hydrogensulfate solid (see below)
- filter funnel
- spatula
- deionised or distilled water in a wash bottle
- 250 cm<sup>3</sup> beaker
- glass rod
- digital mass balance (reading to 2 or 3 decimal places).

The composition of the **sodium hydrogensulfate** should be known; either anhydrous (and the purest available) or the monohydrate. Students need to be advised which they are using. Suppliers can also call this reagent sodium bisulfate.

If using anhydrous, make sure it is not too old as it will have picked up water and therefore not be as accurate a mass.

In trials, 3.5 g of anhydrous and 4.0 g of the monohydrate were used.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

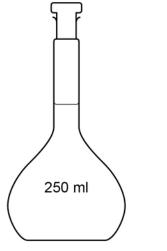
Make up a volumetric solution and carry out a simple acid-base titration: To prepare a solution of sodium hydrogensulfate that has a known concentration.

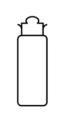
# STUDENT SHEET

#### REQUIREMENTS

You are provided with the following:

- weighing bottle or boat
- 250 cm<sup>3</sup> volumetric (graduated) flask
- sodium hydrogensulfate solid
- filter funnel
- spatula
- deionised or distilled water in a wash bottle
- 250 cm<sup>3</sup> beaker
- glass rod
- digital mass balance (reading to 2 or 3 decimal places).









Volumetric flask

Weighing bottle

Weighing boat

Wash bottle

#### SUGGESTED METHOD

The task is to prepare 250 cm<sup>3</sup> of a solution of sodium hydrogensulfate with a known concentration in the range 0.090 to 0.110 mol dm<sup>-3</sup>

The procedure is as follows:

- a) Calculate the mass of sodium hydrogensulfate solid needed to produce 250 cm<sup>3</sup> of a 0.100 mol dm<sup>-3</sup> solution. Show your working. If you are using the anhydrous solid, the mass to weigh out will be between 2.7 and 3.3 g, and if you are using the monohydrate, the mass to weigh out should be between 3.1 and 3.8 g.
- b) Weigh a clean dry weighing bottle (or weighing boat).
- c) Place the weighing bottle on the pan of a digital balance and, using a spatula, place into the bottle **approximately** the mass of sodium hydrogensulfate that you have calculated to be necessary.
- d) Weigh the weighing bottle and its contents accurately and record the **precise** mass.
- e) Pour the contents of the weighing bottle into a beaker and re-weigh the weighing bottle (which may still contain traces of sodium hydrogensulfate).
- f) Calculate the mass of sodium hydrogensulfate that you have transferred. Remember to record all weighings to the resolution of the balance that you have used.
- g) Add approximately 100 cm<sup>3</sup> of deionised (or distilled) water to the beaker containing the solid and use a glass rod to stir the contents of the beaker until all of the sodium hydrogensulfate dissolves.
- h) Using a funnel, pour the contents of the beaker into a 250 cm<sup>3</sup> volumetric (graduated) flask and then using the wash bottle rinse the beaker and funnel into the same volumetric flask. Rinse the glass rod into these washings.
- i) Make the volumetric flask up to the graduated mark by carefully adding deionised water from the wash bottle. You will need to be careful so that you do not over-shoot the mark.
- j) Stopper the volumetric flask and shake it thoroughly to mix the contents of the flask.
- k) Calculate the exact concentration in mol dm<sup>-3</sup> of your solution quoting the value to the appropriate precision. Show all of your working.

Make up a volumetric solution and carry out a simple acid-base titration: To determine the concentration of a solution of sodium hydroxide by titration using a sodium hydrogensulfate solution that has a known concentration.

# TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

#### REQUIREMENTS

In addition to general laboratory apparatus, each student needs:

- burette
- stand and clamp
- 25 cm<sup>3</sup> pipette
- pipette filler
- two 250 cm<sup>3</sup> conical flasks
- two 250 cm<sup>3</sup> beakers
- funnel
- wash bottle
- phenolphthalein indicator
- standard sodium hydrogensulfate solution (150 cm<sup>3</sup>)
- sodium hydroxide solution (150 cm<sup>3</sup>) labelled as "Sodium hydroxide solution of unknown concentration".

The sodium hydrogensulfate solution needs to be a **solution** with an accurately known concentration between 0.090 and 0.100 mol  $dm^{-3}$  or could be the solution which the student prepared as part a of this required practical.

Students should use a sodium hydroxide solution with an accurately known concentration between 0.090 and 0.100 mol dm<sup>-3</sup> but labelled as "Sodium hydroxide solution of unknown concentration".

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

Sample masses per dm<sup>3</sup>, for example:

3.6 g > 1 dm<sup>3</sup> = 0.090 mol dm<sup>-3</sup> 3.8 g > 1 dm<sup>3</sup> = 0.095 mol dm<sup>-3</sup> 4.0 g > 1 dm<sup>3</sup> = 0.100 mol dm<sup>-3</sup>

#### SAMPLE RESULTS

The following table is a sample results table using results from the trial of this experiment.

	Titre 1/cm <sup>3</sup>	Titre 2/cm <sup>3</sup>	Titre 3/cm <sup>3</sup>
Initial reading	0.10	0.00	0.10
Final reading	24.95	25.10	25.10
Titre	24.85	25.10	25.00
		~	✓

All titre readings to 2 decimal places.

Concordant results should be within  $0.10 \text{ cm}^3$  of each other. In this case, titre 2 and titre 3 are concordant.

The average titre is 25.05 cm<sup>3</sup>

Make up a volumetric solution and carry out a simple acid-base titration: To determine the concentration of a solution of sodium hydroxide by titration using a sodium hydrogensulfate solution that has a known concentration.

## STUDENT SHEET

#### REQUIREMENTS

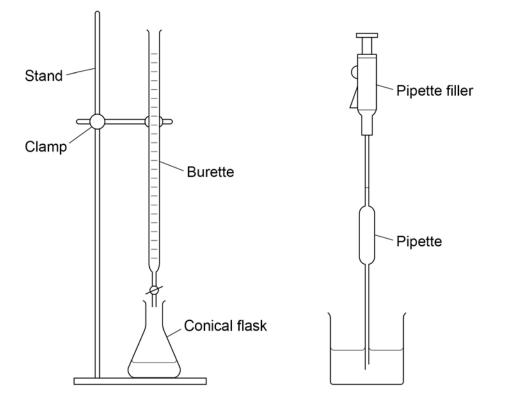
You are provided with the following:

- burette
- stand and clamp
- 25 cm<sup>3</sup> pipette
- pipette filler
- two 250 cm<sup>3</sup> conical flasks
- two 250 cm<sup>3</sup> beakers
- funnel
- wash bottle
- phenolphthalein indicator
- sodium hydrogensulfate solution
- sodium hydroxide solution.

The sodium hydrogensulfate solution may be the solution which you prepared in part a of this experiment or it could be a solution provided to you by your teacher.

#### SUGGESTED METHOD

- a) Pour approximately 100 cm<sup>3</sup> of the sodium hydrogensulfate solution into a clean, dry beaker that is labelled 'sodium hydrogensulfate'. Use a small volume of this solution to rinse the burette before filling it with the sodium hydrogensulfate solution.
- b) Pour approximately 100 cm<sup>3</sup> of the sodium hydroxide solution into a second clean, dry beaker labelled 'sodium hydroxide'.
- c) Rinse a 25 cm<sup>3</sup> pipette with the sodium hydroxide solution provided and then, using a pipette filler, pipette exactly 25.0 cm<sup>3</sup> of sodium hydroxide solution into a 250 cm<sup>3</sup> conical flask (which has been rinsed with deionised water).
- d) Add **two to three drops** of phenolphthalein indicator to the solution in the conical flask and note the colour of the indicator in alkali.
- e) Before you start to titrate, construct a table ready to record your results.
- f) Record the initial burette reading. Make sure that all your burette readings are to the appropriate precision.
- g) Titrate the contents of the conical flask by adding sodium hydrogensulfate solution to it from the burette. Add the sodium hydrogensulfate solution slowly, swirling the flask gently to mix the solution. Add the sodium hydrogensulfate solution dropwise near the end-point until the indicator undergoes a definite colour change; this is the end-point of the titration. Record the colour change in your results. Record the final burette reading in your table of results.



- h) Calculate and record in your table of results the volume of sodium hydrogensulfate solution used.
- i) Repeat the titration until you obtain two results which are concordant. You should normally carry out at least three titrations. Record all of the results that you obtain.
- j) Calculate and record the mean volume of sodium hydrogensulfate solution used in the titration. Show your working.
- k) Use your results to calculate the concentration of the sodium hydroxide. Show your working.

#### Measure an enthalpy change:

To determine an enthalpy change which cannot be measured directly. The reaction involves the conversion of anhydrous copper(II) sulfate into hydrated copper(II) sulfate.

# TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

#### REQUIREMENTS

In addition to general laboratory apparatus, each student needs:

- hydrated copper(II) sulfate (small) crystals (~ 6.20–6.30 g)
- anhydrous copper(II) sulfate powder (~ 3.90–4.10 g)
- polystyrene cups (as a calorimeters) with lids
- 250 cm<sup>3</sup> or 400<sup>3</sup> cm beaker (as appropriate for holding the cup)
- stand and clamp
- 0 °C to 50 °C glass or digital thermometer (0.1 °C or 0.2 °C divisions are desirable but not essential)
- two 25 cm<sup>3</sup> measuring cylinders
- two weighing bottles
- stopwatch
- graph paper
- stirrer
- deionised or distilled water
- access to a digital mass balance (measuring to 2 decimal places).

The calorimeter is a polystyrene cup (an ordinary coffee cup) fitted into the beaker which will provide some insulation, and also act as a support.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

#### **ADDITIONAL NOTES**

If the stock of anyhydrous copper(II) sulfate powder is not white, place in an evaporation dish in an oven at a low temperature or heat gently over a Bunsen burner before storing in a desiccator.

Results obtained could vary considerably depending on the type of cup used, whether or not a lid was used of if there is a high temperature in the laboratory on the day. Lids on the polystyrene cups help to minimise heat loss and maximise change in temperature.

If weighing bottles are not available, a weighing boat can be used if students work quickly.

#### SAFETY

Each student will use a fairly large amount of copper sulfate(VI) and it has an environmental warning. Waste will be an issue so solutions should be collected, filtered and allowed to evaporate so that copper sulfate(VI) can be recycled.

#### SAMPLE RESULTS

The following table is a sample results table using results from the trial of this experiment.

Temperature/°C	Experiment 1	Experiment 2
0	19.8	19.8
1	19.6	19.8
2	19.6	19.8
3	19.6	19.8
4		
5	27.4	18.2
6	27.2	18.2
7	26.8	18.2
8	26.6	18.2
9	26.4	18.2
10	26.2	18.2
11	26.2	18.2
12	26.2	18.2
13	26.2	18.2
14	26.2	18.2
15	26.2	18.2

4.94 g anhydrous copper(II) sulfate and 7.72 g hydrated copper(II) sulfate were used.

#### Measure an enthalpy change:

To determine an enthalpy change which cannot be measured directly. The reaction involves the conversion of anhydrous copper(II) sulfate into hydrated copper(II) sulfate.

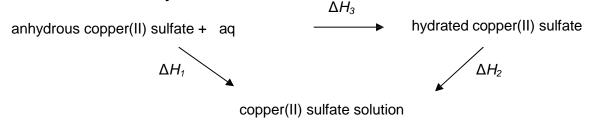
### STUDENT SHEET

#### REQUIREMENTS

You are provided with the following:

- hydrated copper(II) sulfate crystals
- anhydrous copper(II) sulfate powder
- polystyrene cups with lids
- 250 cm<sup>3</sup> or 400<sup>3</sup> cm beaker (as appropriate for holding the cup)
- stand and clamp
- 0 °C to 50 °C glass or digital thermometer
- two 25 cm<sup>3</sup> measuring cylinders
- two weighing bottles
- stopwatch
- graph paper
- stirrer
- deionised or distilled water
- access to a digital mass balance (measuring to 2 decimal places).

#### Consider the Hess's Law cycle below



We can measure accurately by experiment both the values  $\Delta H_1$  and  $\Delta H_2$ 

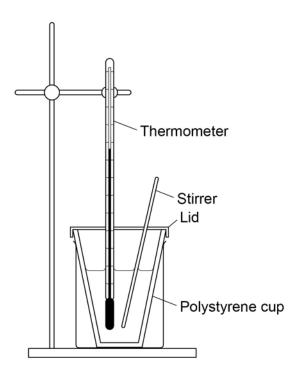
By applying Hess's Law, we can calculate the value for  $\Delta H_3$ , since the two routes from anhydrous copper(II) sulfate to copper(II) sulfate solution have the same overall enthalpy change.

ie	$\Delta H_1$	=	$\Delta H_2$	+	$\Delta H_3$
therefore	$\Delta H_3$	=	$\Delta H_1$	_	$\Delta H_2$

#### Suggested method

#### Experiment 1 - Collecting data for the determination of $\Delta H_1$

- a) Weigh out between 3.90 g and 4.10 g of anhydrous copper(II) sulfate in a dry stoppered weighing bottle, keeping the stock of solid in a closed container during weighing.
   Take care to avoid skin contact. The precise mass should be recorded.
- b) Construct a suitable table of results to allow you to record temperatures at minute intervals up to 15 minutes.
- c) Using a measuring cylinder, place 25 cm<sup>3</sup> of deionised water into a polystyrene cup and record its temperature at the beginning (t=0), start the timer and then record the temperature again every minute, stirring the liquid continuously.



- d) At the fourth minute, add the powdered anhydrous copper(II) sulfate rapidly to the water in the polystyrene cup and continue to stir, but do not record the temperature. At the fifth minute and for every minute up to 15 minutes, stir and record the temperature of the solution in the polystyrene cup.
- e) Plot a graph of temperature (on the *y*-axis) against time. Draw two separate best fit lines; one, which joins the points before the addition, and one, which joins the points after the addition, extrapolating both lines to the fourth minute.
- f) Use your graph to determine the temperature change at the fourth minute, which theoretically should have occurred immediately on addition of the solid.

#### Experiment 2 - Collecting data for the determination of $\Delta H_2$

- a) Weigh out between 6.20 g and 6.30 g of hydrated copper(II) sulfate in a dry stoppered weighing bottle. The precise mass should be recorded.
- b) Construct a suitable table of results to allow you to record temperatures at minute intervals up to 15 minutes as you did for Experiment 1.
- c) Using a measuring cylinder, place 24 cm<sup>3</sup> of deionised water into a polystyrene cup. Since the hydrated crystals contain water, the total amount of water will be approximately the same as in Experiment 1.
- d) Repeat the procedure adopted in Experiment 1. Add the copper(II) sulfate crystals to the water in the polystyrene cup and obtain temperature data for 15 minutes.
- e) Plot a graph similar to that in Experiment 1 and determine the temperature change in this experiment.

#### Analysing the data and calculating $\Delta H_3$

You should be familiar with the expression

Heat change = mass x specific heat capacity x temperature change

#### Heat change = $m c \Delta T$

#### In this experiment, we will ignore heat loss to the surroundings.

The specific heat capacity of the polystyrene cup is negligible when compared to the mass of water and the aqueous solutions can be considered to have the same specific heat capacity as water.

(For many aqueous chemical reactions, it can be assumed that the only substance heated is water).

In each of Experiment 1 and Experiment 2 you need the **mass of water, m (in g)**, which has changed in temperature. As the density of water can be assumed to be 1 g cm<sup>-3</sup> the mass can be directly taken from the volume of water ie 25 g in each case. Do **not** add on the mass of the solid used.

You will also need the **temperature change**,  $\Delta T$  (in K), from your graph in order to be able to calculate the heat change.

For water, the specific heat capacity,  $\mathbf{c} = 4.18 \text{ J K}^{-1} \text{ g}^{-1}$  and, so, the value that you obtain for the heat change in each experiment will be in **joules**. You can convert this value into kilojoules by dividing it by 1000.

You can then calculate the enthalpy changes,  $\Delta H_1$  and  $\Delta H_2$ , in **kJ mol<sup>-1</sup>**, using the masses of the solids used in each experiment.

You need to use the values that you have obtained for  $\Delta H_1$  and  $\Delta H_2$  and apply Hess's

Law to calculate  $\Delta H_3$  in **kJ mol**<sup>-1</sup> for the hydration of copper(II) sulfate.

 $CuSO_4(s) + aq \rightarrow CuSO_4.5H_2O(s)$ 

Carry out simple test-tube reactions to identify:

- cations Group 2,  $NH_4^+$
- anions Group 7 (halide ions),  $OH^-$ ,  $CO_3^{2-}$ ,  $SO_4^{2-}$

To carry out tests for the presence of cations and anions and to make accurate observations.

## TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

It is advisable to split the content of this practical over a number of sessions so that the material is carefully completed.

#### REQUIREMENTS

#### General

- test tubes and stoppers
- test-tube racks
- plastic graduated dropping pipettes
- deionised or distilled water
- forceps.

In addition to general laboratory apparatus, each student needs the following for each test:

#### Test 1

- 0.1 mol dm<sup>-3</sup> barium chloride solution
- 0.6 mol dm<sup>-3</sup> sodium hydroxide solution
- 0.1 mol dm<sup>-3</sup> calcium bromide solution (or calcium nitrate/potassium bromide)
- 0.1 mol dm<sup>-3</sup> magnesium chloride solution
- 0.1 mol dm<sup>-3</sup> strontium chloride solution.

- 0.1 mol dm<sup>-3</sup> barium chloride solution
- 1.0 mol dm<sup>-3</sup> sulfuric acid
- 0.1 mol dm<sup>-3</sup> calcium bromide solution (or calcium nitrate/potassium bromide)
- 0.1 mol dm<sup>-3</sup> magnesium chloride solution
- 0.1 mol dm<sup>-3</sup> strontium chloride solution.

#### Test 3

- 0.1 mol dm<sup>-3</sup> ammonium chloride •
- 0.4 mol dm<sup>-3</sup> sodium hydroxide solution
- red litmus paper
- kettle
- water bath.

#### Test 4

- 0.4 mol dm<sup>-3</sup> sodium hydroxide solution
- red litmus paper (or universal indicator paper)
- 1.0 mol dm<sup>-3</sup> ammonia solution (freshly prepared)
- petri dish with lid.

#### Test 5

- 0.5 mol dm<sup>-3</sup> sodium carbonate solution
- 0.5 mol dm<sup>-3</sup> hydrochloric acid
- $0.02 \text{ mol } \text{dm}^{-3}$  calcium hydroxide solution (limewater).

#### Test 6

- 0.1 mol dm<sup>-3</sup> barium chloride solution
- $0.1 \text{ mol dm}^{-3}$  magnesium sulfate solution.

#### Test 7

- 0.1 mol  $dm^{-3}$  potassium chloride solution 0.1 mol  $dm^{-3}$  potassium bromide solution •
- 0.1 mol dm<sup>-3</sup> potassium iodide solution
- 0.1 mol dm<sup>-3</sup> nitric acid
- 0.05 mol dm<sup>-3</sup> silver nitrate solution
- concentrated ammonia solution (in a fume cupboard)
- 2.0 mol dm<sup>-3</sup> ammonia solution (in a fume cupboard, freshly prepared and labelled 'ammonia solution').

#### Test 8

- potassium chloride solid •
- potassium bromide solid
- potassium iodide solid
- 0.1 mol dm<sup>-3</sup> lead nitrate solution (or lead ethanoate solution) •
- blue litmus paper
- filter paper •
- small spatula.
- concentrated sulfuric acid in dropping bottles (in a fume cupboard)
- 0.5 mol dm<sup>-3</sup> acidified potassium dichromate(VI) solution (in a fume cupboard, see below).

The acidified potassium dichromate(VI) solution should be made by dissolving 3 g of potassium dichromate in 100 cm<sup>3</sup> of 1.0 mol dm<sup>-3</sup> sulfuric acid.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

#### ADDITIONAL NOTES

In test 3, step (b) will work slowly at room temperature or use water from a recently boiled kettle poured into a beaker.

In test 8, step (b), only 2 to 5 drops of concentrated sulfuric acid should be added and this should be done slowly.

#### SAMPLE RESULTS

Test 1

	Barium chloride	Calcium bromide	Magnesium chloride	Strontium chloride
Initial	colourless solution	colourless solution	colourless solution	colourless solution
10 drops of 0.6 mol dm <sup>-3</sup> NaOH	colourless solution	slight white precipitate	slight white precipitate	slight white precipitate
Excess NaOH	colourless solution	slight white precipitate	white precipitate	slight white precipitate

#### Test 2

	Barium chloride	Calcium bromide	Magnesium chloride	Strontium chloride
10 drops of 1.0 mol dm <sup>-3</sup> H₂SO₄	white precipitate	slight white precipitate	slight white precipitate	white precipitate
Excess H <sub>2</sub> SO <sub>4</sub>	white precipitate	slight white precipitate	colourless solution	white precipitate

#### Test 3

Ammonium chloride + sodium hydroxide - Damp red litmus paper = blue

#### Test 4

Sodium hydroxide – Damp red litmus paper = blue Ammonia solution – Damp red litmus paper = blue

#### Test 5

Limewater - colourless solution to cloudy

#### Test 6

Test of sulfate ions - white precipitate forms

#### Test 7

	+ HNO <sub>3</sub>	+ 2 M NH <sub>3</sub>	+ concentrated NH <sub>3</sub>
Potassium chloride	white precipitate	colourless solution	colourless solution
Potassium bromide	cream precipitate	cream precipitate	colourless solution
Potassium iodide	yellow precipitate	yellow precipitate	yellow precipitate

	Concentrated sulfuric acid	Result of paper test
Potassium chloride	effervescence	red
Potassium bromide	effervescence brown gas produced solution turns deep brown/red	
Potassium iodide	solution goes red/brown immediately brown gas produced	turns black/grey

Carry out simple test-tube reactions to identify:

- cations Group 2, NH<sub>4</sub><sup>+</sup>
- anions – Group 7 (halide ions),  $OH^-$ ,  $CO_3^{2-}$ ,  $SO_4^{2-}$

To carry out tests for the presence of cations and anions and to make accurate observations.

### STUDENT SHEET

These tests may be split over several lessons.

#### REQUIREMENTS

You are provided with the following:

#### **GENERAL**

- test tubes and stoppers
- test-tube racks
- plastic graduated dropping pipettes •
- deionised or distilled water •
- forceps.

#### Test 1

- 0.1 mol dm<sup>-3</sup> barium chloride solution
- 0.6 mol dm<sup>-3</sup> sodium hydroxide solution
- 0.1 mol dm<sup>-3</sup> calcium bromide solution (or calcium nitrate/potassium bromide) •
- 0.1 mol dm<sup>-3</sup> magnesium chloride solution
- 0.1 mol dm<sup>-3</sup> strontium chloride solution.

#### Test 2

- 0.1 mol dm<sup>-3</sup> barium chloride solution •
- 1.0 mol dm<sup>-3</sup> sulfuric acid •
- 0.1 mol  $dm^{-3}$  calcium bromide solution (or calcium nitrate/potassium bromide) 0.1 mol  $dm^{-3}$  magnesium chloride solution
- •
- 0.1 mol  $dm^{-3}$  strontium chloride solution.

- 0.1 mol dm<sup>-3</sup> ammonium chloride •
- 0.4 mol dm<sup>-3</sup> sodium hydroxide solution
- red litmus paper
- kettle
- water bath.

#### Test 4

- 0.4 mol dm<sup>-3</sup> sodium hydroxide solution •
- red litmus paper (or universal indicator paper)
- 1.0 mol dm<sup>-3</sup> ammonia solution
- petri dish with lid.

#### Test 5

- 0.5 mol dm<sup>-3</sup> sodium carbonate solution
- 0.5 mol dm<sup>-3</sup> hydrochloric acid •
- 0.02 mol dm<sup>-3</sup> calcium hydroxide solution (limewater).

#### Test 6

- 0.1 mol  $dm^{-3}$  barium chloride solution 0.1 mol  $dm^{-3}$  magnesium sulfate solution. •

#### Test 7

- 0.1 mol dm<sup>-3</sup> potassium chloride solution 0.1 mol dm<sup>-3</sup> potassium bromide solution 0.1 mol dm<sup>-3</sup> potassium iodide solution 0.1 mol dm<sup>-3</sup> nitric acid
- •
- 0.05 mol dm<sup>-3</sup> silver nitrate solution •
- concentrated ammonia solution
- 2.0 mol dm<sup>-3</sup> ammonia solution.

- potassium chloride solid •
- potassium bromide solid
- potassium iodide solid
- 0.1 mol dm<sup>-3</sup> lead nitrate solution (or lead ethanoate solution) •
- blue litmus paper
- filter paper •
- small spatula. •
- concentrated sulfuric acid in dropping bottles •
- 0.5 mol dm<sup>-3</sup> acidified potassium dichromate(VI) solution.

#### Suggested method

In every case, you should present all of your observations in a neat table. The presentation of a clearly organised record of your observations is an important skill which you will be expected to demonstrate.

#### Tests 1 and 2: Testing for Group 2 metal cations

Test 1: Dilute sodium hydroxide

- a) Place about 10 drops of 0.1 mol dm<sup>-3</sup> barium chloride in a clean test tube.
- b) Add about 10 drops of 0.6 mol dm<sup>-3</sup> sodium hydroxide solution, mixing well.
- c) Now continue to add this sodium hydroxide solution, dropwise with gentle shaking, until in excess.

The test tube should **not** be more than half full. Once completed, dispose of the contents by placing the test tube in a bowl of water.

d) Repeat this test with the calcium bromide, magnesium chloride and strontium chloride.

#### Test 2: Dilute sulfuric acid

- a) Place about 10 drops of 0.1 mol dm<sup>-3</sup> barium chloride in a clean test tube.
- b) Add about 10 drops of 1.0 mol  $dm^{-3}$  sulfuric acid, mixing well.
- c) Now continue to add this dilute sulfuric acid, dropwise with gentle shaking, until in excess.

The test tube should not be more than half full. Once completed, dispose of the contents by placing the test tube in a bowl of water.

d) Repeat this test with the calcium bromide, magnesium chloride and strontium chloride.

#### Test 3: Testing for ammonium ions

- a) Place about 10 drops of 0.1 mol dm<sup>-3</sup> ammonium chloride in a clean test tube.
- b) Add about 10 drops of 0.4 mol dm<sup>-3</sup> sodium hydroxide solution. Shake the mixture.
- c) Warm the mixture in the test tube gently using a water bath.
- d) Test the fumes released from the mixture by using forceps to hold a piece of damp red litmus paper in the mouth of the test tube.
- e) Dispose of the contents by using the previous method.

#### Tests 4, 5, and 7: Tests for anions in aqueous solution

Test 4: Test for hydroxide ions in aqueous solution

- a) Test about 1 cm<sup>3</sup> of 0.4 mol dm<sup>-3</sup> sodium hydroxide solution in a test tube with red litmus paper or universal indicator paper.
- b) Record your observations. Dispose of the test tube contents.

This approach can also be used to test for the alkaline gas, ammonia, which forms hydroxide ions when it comes into contact with water.

- c) Take 5 drops of 1 mol dm<sup>-3</sup> ammonia solution and place on a filter paper and place inside a petri dish with lid. Dampen a piece of red litmus paper with deionised water and place on the other side of the petri dish. Replace the lid and observe over a few minutes.
- d) Record your observations.

Test 5: Test for carbonate ions in aqueous solution

- a) Have about 2 cm<sup>3</sup> of calcium hydroxide (limewater) ready in a test tube.
- b) To about 3 cm<sup>3</sup> of 0.5 mol dm<sup>-3</sup> sodium carbonate solution in a test tube, add an equal volume of 1.0 mol dm<sup>-3</sup> dilute hydrochloric acid.
- c) Immediately put in delivery tube with open end into the limewater test tube. Make sure that the end of the tube is below the level of the liquid.
- d) Record your observations. Dispose of the test tube contents.

Test 6: Test for sulfate ions in aqueous solution

- a) To about 1 cm<sup>3</sup> of 0.1 mol dm<sup>-3</sup> magnesium sulfate solution in a test tube, add an equal volume of dilute hydrochloric acid followed by an equal volume of 0.1 mol dm<sup>-3</sup> barium chloride solution.
- b) Record your observations. Dispose of the test tube contents.

#### Test 7: Test for halide ions in aqueous solution

Test for chloride, bromide and iodide ions in aqueous solution

- a) Place about 10 drops of 0.1 mol dm<sup>-3</sup> potassium chloride in a clean test tube.
- b) Add about 5 drops of dilute nitric acid. Shake well.
- c) To the solution add another 10 drops of 0.05 mol  $dm^{-3}$  silver nitrate solution.
- d) Then add an excess of 2 mol dm<sup>-3</sup> ammonia solution and shake to mix thoroughly. Dispose of the tube contents.
- e) Repeat steps a) and b), but this time add an excess of concentrated ammonia solution, working in a fume cupboard. Dispose of the tube contents.
- f) Repeat steps a) to d) but using potassium bromide and then potassium iodide instead of potassium chloride.

#### Test 8: Test for halide ions in solid salts using concentrated sulfuric acid

Test for chloride, bromide and iodide ions in solid potassium halides

Note: Gloves must be worn for this procedure.

#### These experiments must be done in a fume hood

- a) Place a small spatula of solid potassium chloride in a clean dry test tube.
- b) Slowly add a few (2 to 5) drops of concentrated sulfuric acid.
- c) Record what happens.
- d) Test the gas evolved with moist blue litmus paper, taking care that the paper does not touch the sides of the test tube.
- e) Repeat this experiment with solid potassium bromide, but this time test the gas produced using a narrow strip of filter paper that has been dipped in acidified potassium dichromate solution.
- f) Repeat this experiment with potassium iodide, but this time test the gas produced using a narrow strip of filter paper that has been dipped in lead nitrate solution.

Distil a product from a reaction:

To prepare ethanal by the oxidation of ethanol and to distil the ethanal from the reaction mixture.

## TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

#### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- simple distillation apparatus OR Quickfit apparatus
- acidified potassium dichromate(VI) (see below)
- protective gloves
- stand and clamp
- 10 cm<sup>3</sup> measuring cylinder
- 25 cm<sup>3</sup> measuring cylinder
- anti-bumping granules
- test tube
- thermometer (-10 °C to 110 °C)
- two 250 cm<sup>3</sup> beakers
- ethanol
- teat pipette
- 0.05 mol dm<sup>-3</sup> silver nitrate solution
- 2 mol dm<sup>-3</sup> dilute ammonia solution
- 2 mol dm<sup>-3</sup> sodium hydroxide solution
- 1 mol dm<sup>-3</sup> dilute sulfuric acid.

The student sheet assumes that **simple distillation apparatus** will be used and this can be made by using a boiling tube fitted with a bung with a right-angled glass delivery tube. The delivery tube needs to be long enough to go into a test tube immersed in cold water in a beaker. A 400 cm<sup>3</sup> beaker can be used for the water bath as this will be big enough to immerse the test tube. Ideally, a lab jack should be available for the beaker to collect the product in. If not available, several heat proof mats or an inverted trough with a mat on top will work.

The alternative is for the centre to provide Quickfit apparatus and guidance to students in its assembly. This apparatus will lead to a more ethanal being collected because it is condensed more efficiently using a water-cooled condenser.

Spare supplies of all reagents specified in these notes should be available for student use

(if needed).

#### Distil a product from a reaction:

To prepare ethanal by the oxidation of ethanol and to distil the ethanal from the reaction mixture.

### STUDENT SHEET

#### REQUIREMENTS

You are provided with the following:

- simple distillation apparatus OR Quickfit apparatus •
- acidified sodium dichromate(VI) •
- protective gloves •
- stand and clamp •
- 10 cm<sup>3</sup> measuring cylinder •
- 25 cm<sup>3</sup> measuring cylinder
- anti-bumping granules
- test tube •
- thermometer •
- two 250 cm<sup>3</sup> beakers •
- ethanol
- teat pipette •
- 0.05 mol dm<sup>-3</sup> silver nitrate solution
- 2 mol  $dm^{-3}$  dilute ammonia solution 2 mol  $dm^{-3}$  sodium hydroxide solution
- 1 mol dm<sup>-3</sup> dilute sulfuric acid.

#### SUGGESTED METHOD

#### The oxidation of ethanol to ethanal

- a) Using a 25 cm<sup>3</sup> measuring cylinder, carefully measure out 12 cm<sup>3</sup> of the solution of acidified sodium dichromate(VI). Pour this oxidising agent into a boiling tube. You should wear protective gloves when handling the corrosive oxidising agent.
- b) Cool the boiling tube in cold water in a beaker.
- c) Using a 10 cm<sup>3</sup> measuring cylinder, carefully measure out 2 cm<sup>3</sup> of ethanol.
- Using a teat pipette, **slowly** add the 2 cm<sup>3</sup> of ethanol **dropwise**, to the oxidising agent in the d) cooled boiling tube (immersed in cold water in a beaker), shaking the tube gently to mix the contents.
- e) After the addition of ethanol, add a few anti-bumping granules to the boiling tube and attach to it a bung fitted with a right-angled glass delivery tube.
- Clamp the boiling tube at about 45° in a beaker of water. Heat this beaker of water gently f) and **slowly** distil off approximately 5 cm<sup>3</sup> of liquid distillate into a test tube which is immersed in cold water in a beaker. Keep the test tube cool to avoid loss of the volatile ethanal.
- g) Carry out the test described below on the distillate to confirm that ethanal has been formed in this reaction.

#### Test on the distillate to confirm the formation of ethanal

#### Tollens' silver mirror test:

- a) Prepare a sample of Tollens' reagent by adding 5 drops of sodium hydroxide solution to 2 cm<sup>3</sup> of silver nitrate solution in a test tube.
- b) To this test tube add **just enough** dilute ammonia solution to dissolve the brown precipitate completely.
- c) Using a beaker of hot water (50–60 °C), **gently warm** approximately 5 cm<sup>3</sup> of this test reagent in a test tube.
- d) Add 10 drops of the distillate containing ethanal to the warmed Tollens' reagent in the test tube. Wait a few minutes and note what happens. You should have produced a silver mirror on the walls of the tube.

Make sure that you dispose of the Tollens' reagent thoroughly by rinsing it away with plenty of water and then rinsing any glassware that has contained the reagent with a little dilute sulfuric acid when you are finished.

Carry out tests for alcohols, aldehydes, alkenes and carboxylic acids:

# TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

#### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- ethanol
- ethanal or propanal
- cyclohexene
- 1-bromobutane
- dilute ethanoic acid
- small pieces of metallic sodium under petroleum ether \*

   (a beaker of ethanol should be available for safe disposal of any excess sodium)
- Fehling's solution A
- Fehling's solution B
- bromine water
- sodium carbonate solution
- sodium hydrogencarbonate solid
- sodium hydroxide solution
- silver nitrate solution (0.05 mol dm<sup>-3</sup>)
- dilute nitric acid
- 250 cm<sup>3</sup> beaker
- anti-bumping granules
- test tubes, boiling tubes and a test-tube holder
- thermometer (-10 °C to 110 °C)
- plastic graduated dropping pipettes.

The concentrations of the aqueous solutions in these experiments need to be sufficient to ensure that obvious reactions take place. In practice, this is likely to mean  $2 \text{ mol } dm^{-3}$  for most solutions.

\* The small pieces of metallic sodium should be approximately the size of a grain of rice.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

Carry out tests for alcohols, aldehydes, alkenes and carboxylic acids:

STUDENT SHEET

#### REQUIREMENTS

You are provided with the following:

- ethanol
- ethanal or propanal
- cyclohexene
- 1-bromobutane
- dilute ethanoic acid
- small pieces of metallic sodium under petroleum ether
   (a beaker of ethanol should be available for safe disposal of any excess sodium)
- Fehling's solution A
- Fehling's solution B
- bromine water
- sodium carbonate solution
- sodium hydrogencarbonate solid
- sodium hydroxide solution
- silver nitrate solution (0.05 mol dm<sup>-3</sup>)
- dilute nitric acid
- 250 cm<sup>3</sup> beaker
- anti-bumping granules
- test tubes, boiling tubes and a test-tube holder
- thermometer
- plastic graduated dropping pipettes.

#### SUGGESTED METHOD

This experiment is divided into five parts.

In every case, you should present all of your observations in a neat table. The presentation of a clearly organised record of your observations is an important skill which you will be expected to demonstrate as part of this assessment.

#### Part 1 – A test for an alcohol

- a) To about 1 cm<sup>3</sup> of ethanol in a dry test tube, add a small piece of metallic sodium.
- b) Record your observations.
- c) Make sure that you dispose safely of any excess sodium using the beaker of ethanol provided.

### Part 2 – A test for an aldehyde using Fehling's solution.

- a) In a clean test tube mix together equal volumes of Fehling's solution A and Fehling's solution B. The resultant Fehling's test reagent should be a clear dark blue solution.
- b) Add 5 drops of this test reagent to about 1 cm<sup>3</sup> of sodium carbonate solution in a test tube containing a few anti-bumping granules and then add about 1 cm<sup>3</sup> of ethanal (or propanal) to this same test tube.
- c) Warm the test tube gently for approximately 2 minutes in a beaker half- filled with hot water and then gradually bring the beaker of water to boiling and maintain this temperature for a few minutes.
- d) Using the test tube holder, carefully lift the test tube out of the boiling water and allow its contents to stand for several minutes. Record your observations.

### Part 3 – A test for an alkene (a test for unsaturation)

- a) To about 2 drops of cyclohexene in a test tube, add about 1 cm<sup>3</sup> of bromine water and shake the contents of the tube vigorously from side to side.
- b) Record your observations.

### Part 4 – A test for a carboxylic acid

- a) Place one small spatula measure of solid sodium hydrogencarbonate in a boiling tube tube and add to it about 2 cm<sup>3</sup> of dilute ethanoic acid.
- b) Record your observations.

### Part 5 – A test for a halogenoalkane

- a) Using a teat pipette, add 5 drops of 1-bromobutane to about 1 cm<sup>3</sup> of sodium hydroxide solution in a test tube. Warm the contents of the test tube for a few minutes, by placing it into a beaker filled with hot water at approximately 60 °C.
- b) Acidify the contents of the test tube by adding 2 cm<sup>3</sup> of dilute nitric acid and then add about 1 cm<sup>3</sup> of silver nitrate solution.
- c) Record your observations.

Investigate how pH changes when a weak acid reacts with a strong base and when a strong acid reacts with a weak base:

Investigation of how the pH of a solution of ethanoic acid changes as sodium hydroxide solution is added.

## TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- two 50 cm<sup>3</sup> burettes
- two funnels
- 100 cm<sup>3</sup> beaker
- deionised (or distilled) water in a wash bottle
- stand and clamp
- pH meter/probe
- 0.100 mol dm<sup>-3</sup> sodium hydroxide solution
- 0.100 mol dm<sup>-3</sup> ethanoic acid solution
- standard pH buffer solutions at pH 4.00, 7.00 and 9.20
- stirring rod
- graph paper.

Teachers may choose to use other weak acid/strong base combinations or strong acid/weak base combinations to fully fulfil the specification requirements.

The pH buffer solutions can be bought in ready-made or as tables which can then be made up.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

Investigate how pH changes when a weak acid reacts with a strong base and when a strong acid reacts with a weak base:

Investigation of how the pH of a solution of ethanoic acid changes as sodium hydroxide solution is added.

### STUDENT SHEET

This experiment investigates how the pH of a solution of ethanoic acid changes as sodium hydroxide solution is added.

The results are plotted in a graph which shows the general pattern of how the pH changes when a weak acid reacts with a strong base.

It is necessary initially to calibrate a pH meter so as to give accurate pH values for each pH reading.

### REQUIREMENTS

You are provided with the following:

- two 50 cm<sup>3</sup> burettes
- two funnels
- 100 cm<sup>3</sup> beaker
- deionised (or distilled) water in a wash bottle
- stand and clamp
- pH meter/probe
- 0.100 mol dm<sup>-3</sup> sodium hydroxide solution
- 0.100 mol dm<sup>-3</sup> ethanoic acid solution
- standard pH buffer solutions at pH 4.00, 7.00 and 9.20
- stirring rod
- graph paper.

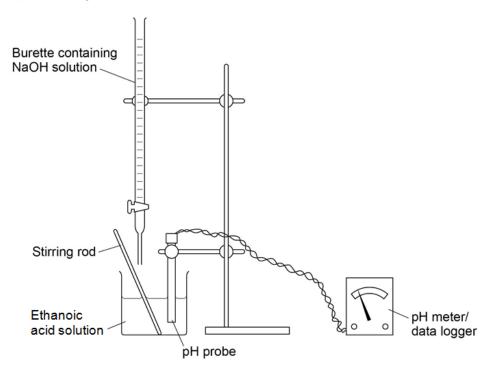
### SUGGESTED METHOD

### Part 1 Calibrate the pH meter

- a) Rinse the pH probe thoroughly with deionised water, and shake it gently to remove excess water. Place the probe in the standard pH 7.00 buffer solution provided, ensuring that the bulb is fully immersed. Record the pH reading in a suitable table.
- b) Repeat this process using the standard pH 4.00 and 9.20 buffer solutions. Rinse the pH probe thoroughly with deionised water before taking each reading. Record the pH readings in your table.
- c) Plot a graph of your recorded pH reading (*x*-axis) against the pH of the buffer solution. Your graph may be a straight line or a curve. This calibration graph will be used in the next part of the experiment to convert pH readings into more accurate pH values.

### Part 2 The measurement of the pH of the mixture of acid and alkali

- a) Rinse a burette with the 0.100 mol dm<sup>-3</sup> solution of ethanoic acid provided and then fill the burette with this solution, ensuring that it is filled below the tap. Label this burette so that you do not confuse it with the second burette.
- b) Use the burette to transfer exactly 20.0 cm<sup>3</sup> of ethanoic acid to a clean 100 cm<sup>3</sup> beaker.
- c) Rinse a second burette with the 0.100 mol dm<sup>-3</sup> NaOH solution provided and then fill this second burette with this solution, ensuring that it is filled below the tap.
- d) Rinse the pH probe with distilled or deionised water and clamp it so that its bulb is fully immersed in the ethanoic acid solution in the beaker. Use a rod to stir the solution gently and record the pH reading in a suitable table.



e) Using the second burette, add exactly 2.0 cm<sup>3</sup> of the NaOH solution to the beaker containing the ethanoic acid. Stir the mixture gently with the glass rod and measure the pH of the mixture. Record the pH reading.

Add the NaOH solution in 2.0 cm<sup>3</sup> portions from the second burette to the ethanoic acid in the beaker until 18 cm<sup>3</sup> of the NaOH solution have been added. Take a pH reading after each addition of NaOH solution, and in each case record the pH reading in your table. Then add the NaOH solution in 0.20 cm<sup>3</sup> portions until 22.0 cm<sup>3</sup> is reached.

Then add the NaOH solution in 2.0 cm<sup>3</sup> portions again until 40 cm<sup>3</sup> have been added.

f) Rinse the pH probe with distilled or deionised water when you have taken all of your readings.

### ANALYSING THE DATA

- a) Use your calibration graph from Part 1 to adjust, as appropriate, the pH readings obtained in your experiment in Part 2. These corrected pH values should be entered into a new column in your table of results.
- b) Plot a graph of the corrected pH values from Part 2 (*y*-axis) against volume of sodium hydroxide solution added.
- c) Join the points in the most appropriate way.
- d) Comment on the shape of the curve.

Measure the rate of reaction by an initial rate method and a continuous monitoring method: An 'lodine Clock' experiment: To investigate the reaction of iodide(V) ions with hydrogen peroxide in acidic solution and to determine the order of the reaction with respect to iodide ions.

### TEACHERS' NOTES

This worksheet provides a method with which students can measure the rate of reaction using an initial rate method. Students will also have to complete a further practical activity and measure the rate of reaction using a continuous monitoring method. This could be based on our exemplar practical in worksheet 6b or any alternative practical work that fulfils this requirement.

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

### INTRODUCTION

The 'lodine Clock' experiment can be used to determine the effect of a change in concentration of iodide ions on the reaction between hydrogen peroxide and iodide ions.

Hydrogen peroxide reacts with iodide ions to form iodine and the thiosulfate ion immediately reacts with iodine as shown below.

 $H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2H_2O(I)$ 

 $2S_2O_3{}^{2-}(aq) + I_2(aq) \rightarrow 2I^{-}(aq) + S_4O_6{}^{2-}(aq)$ 

When the  $I_2$  produced has reacted with all of the limited amount of thiosulfate ions present, excess  $I_2$  remains in solution. Reaction with the starch then forms a dark blue-black colour.

By varying the concentration of I<sup>-</sup>, you can determine the order of reaction with respect to I<sup>-</sup> ions.

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- 125 cm<sup>3</sup> of 0.25 mol dm<sup>-3</sup> dilute sulfuric acid
- 100 cm<sup>3</sup> of 0.10 mol dm<sup>-3</sup> potassium iodide solution
- 25 cm<sup>3</sup> of 0.05 mol dm<sup>-3</sup> sodium thiosulfate solution
- 50 cm<sup>3</sup> of 0.10 mol dm<sup>-3</sup> hydrogen peroxide solution (freshly prepared)
- 5 cm<sup>3</sup> of 1% starch solution (freshly prepared)
- 50 cm<sup>3</sup> burette
- funnel suitable for filling a burette
- stand and clamp
- white tile
- plastic dropping pipette (or starch solution provided in a dropping bottle)
- 25 cm<sup>3</sup> measuring cylinder
- 50 cm<sup>3</sup> measuring cylinder
- 100 cm<sup>3</sup> beaker
- 250 cm<sup>3</sup> beaker
- stirrer
- stopwatch
- paper towels to dry beakers
- plentiful supply of distilled or deionised water.

The exemplar method provided is based on each student having access to communal burettes (approximately one set for every five students) containing hydrogen peroxide and sodium thiosulfate solutions. Teachers are advised that frequent refilling of these burettes will be required.

The hydrogen peroxide solution must be freshly prepared on the day of the practical from a recently purchased, more concentrated solution.

The 1% starch solution must be freshly prepared on the day of the practical.

The hydrogen peroxide is the reagent controlling the time of the reaction and the concentration stated above can be varied as required.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

Measure the rate of reaction by an initial rate method and a continuous monitoring method: An 'lodine Clock' experiment: To investigate the reaction of iodide(V) ions with hydrogen peroxide in acidic solution and to determine the order of the reaction with respect to iodide ions.

### STUDENT SHEET

The 'lodine Clock' experiment can be used to determine the effect of a change in concentration of iodide ions on the reaction between hydrogen peroxide and iodide ions.

### INTRODUCTION

Hydrogen peroxide reacts with iodide ions to form iodine and the thiosulfate ion immediately reacts with iodine as shown below.

 $H_2O_2(aq) + 2H^+(aq) + 2I^-(aq) \rightarrow I_2(aq) + 2H_2O(I)$ 

 $2S_2O_3{}^{2-}(aq) + I_2(aq) \rightarrow 2I^{-}(aq) + S_4O_6{}^{2-}(aq)$ 

When the  $I_2$  produced has reacted with all of the limited amount of thiosulfate ions present, excess  $I_2$  remains in solution. Reaction with the starch then forms a dark blue-black colour.

By varying the concentration of I<sup>-</sup>, you can determine the order of reaction with respect to I<sup>-</sup> ions.

### REQUIREMENTS

You are provided with the following:

- 0.25 mol dm<sup>-3</sup> dilute sulfuric acid
- 0.10 mol dm<sup>-3</sup> potassium iodide solution
- 0.05 mol dm<sup>-3</sup> sodium thiosulfate solution (in a shared burette)
- 0.10 mol dm<sup>-3</sup> hydrogen peroxide solution (in a shared burette)
- starch solution
- 50 cm<sup>3</sup> burette
- funnel suitable for filling a burette
- stand and clamp
- white tile
- plastic dropping pipette
- 25 cm<sup>3</sup> measuring cylinder
- 50 cm<sup>3</sup> measuring cylinder
- 100 cm<sup>3</sup> beaker
- 250 cm<sup>3</sup> beaker
- stirrer
- stopwatch
- paper towels to dry beakers
- plentiful supply of distilled or deionised water.

### SUGGESTED METHOD

### **Experiment 1**

- a) Rinse a 50 cm<sup>3</sup> burette with potassium iodide solution. Fill the burette with potassium iodide solution.
- b) Transfer 10.0 cm<sup>3</sup> of hydrogen peroxide solution from the shared burette provided to a clean, dry **100 cm<sup>3</sup>** beaker. You will use this in step (h).
- c) Use a 50 cm<sup>3</sup> measuring cylinder to add 25 cm<sup>3</sup> of sulfuric acid to a clean, dry **250 cm<sup>3</sup>** beaker.
- d) Use a 25 cm<sup>3</sup> measuring cylinder to add 20 cm<sup>3</sup> of distilled or deionised water into the 250 cm<sup>3</sup> beaker.
- e) Use a plastic dropping pipette to add about 1 cm<sup>3</sup> of starch solution to this beaker.
- f) Use your burette to add 5.0 cm<sup>3</sup> of potassium iodide solution to the mixture in the 250 cm<sup>3</sup> beaker.
- g) Finally, add 5.0 cm<sup>3</sup> of sodium thiosulfate solution from the shared burette provided to the mixture in the 250 cm<sup>3</sup> beaker. Make sure this sodium thiosulfate solution is added last.
- h) Stir the mixture in the 250 cm<sup>3</sup> beaker. Pour the hydrogen peroxide solution from the 100 cm<sup>3</sup> beaker into the 250 cm<sup>3</sup> beaker and **immediately** start the timer. Stir the mixture.
- i) Stop the timer when the mixture in the 250 cm<sup>3</sup> beaker turns blue-black. Record the time to an

appropriate precision in a table of your own design. This experiment could take several minutes.

j) Rinse the 250 cm<sup>3</sup> beaker with distilled or deionised water and dry it with a paper towel.

### **Experiments 2–5**

k) Repeat steps (b) to (j) in four further experiments using the volumes shown in the following table.

Volumes of solutions added to 250 cm <sup>3</sup> beaker						Volume in 100 cm <sup>3</sup> beaker
Experiment	Sulfuric acid 0.25 M/cm <sup>3</sup>	Starch/ cm <sup>3</sup>	Water/ cm³	Potassium iodide 0.10 M/cm <sup>3</sup>	Sodium thiosulfate 0.05 M/cm <sup>3</sup>	Hydrogen peroxide 0.10 M/ cm <sup>3</sup>
1	25	1	20	5.0	5.0	10.0
2	25	1	15	10.0	5.0	10.0
3	25	1	10	15.0	5.0	10.0
4	25	1	5	20.0	5.0	10.0
5	25	1	0	25.0	5.0	10.0

Measure the rate of reaction by an initial rate method and a continuous monitoring method: The reaction between magnesium and hydrochloric acid.

### TEACHERS' NOTES

This worksheet provides a method with which students can measure the rate of reaction using a continuous monitoring method. Students will also have to complete a further practical activity and measure the rate of reaction using an initial rate method. This could be based on our exemplar practical in worksheet 6aor any alternative practical work that fulfils this requirement.

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- 6 cm strips of magnesium ribbon
- 75 cm<sup>3</sup> of 0.8 mol dm<sup>-3</sup> hydrochloric acid
- 50 cm<sup>3</sup> measuring cylinder
- 100 cm<sup>3</sup> conical flask
- rubber bung and delivery tube to fit conical flask
- 100 cm<sup>3</sup> gas syringe OR trough/plastic container with 100 cm<sup>3</sup> measuring cylinder
- stand, boss and clamp
- stopwatch or timer
- distilled or deionised water.

In the suggested method, the magnesium ribbon is provided as 6 cm strips, pre-cut by the technician.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

From trials, a 100 cm<sup>3</sup> gas syringe was suitable for the volume of gas produced in 2.5 minutes. However, you can replace this with a 250 cm<sup>3</sup> gas syringe based on your own trial results.

### SAMPLE RESULTS

The following table is a sample results table using results from the trial of this experiment.

	Volume of gas collected/cm <sup>3</sup>			
Time/s	0.4 mol dm <sup>−3</sup> hydrochloric acid	0.8 mol dm <sup>-3</sup> hydrochloric acid		
0	0.0	0.0		
15	4.0	15.0		
30	6.0	29.0		
45	9.0	43.0		
60	17.0	54.0		
75	20.0	66.0		
90	22.0	75.0		
105	24.0	84.0		
120	27.0	91.0		
135	30.0	95.0		
150	32.0	96.0		

These were obtained using 0.8 mol  $dm^{-3}$  hydrochloric acid and 0.4 mol  $dm^{-3}$  hydrochloric acid with 6 cm strips of magnesium ribbon.

Measure the rate of reaction by an initial rate method and a continuous monitoring method: The reaction between magnesium and hydrochloric acid.

### STUDENT SHEET

### REQUIREMENTS

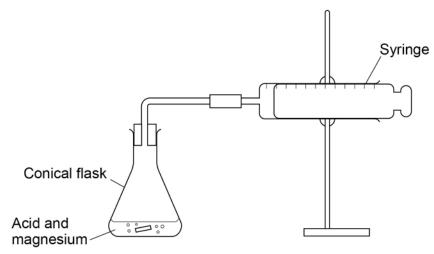
You are provided with the following:

- magnesium ribbon
- 0.8 mol dm<sup>-3</sup> hydrochloric acid
- 50 cm<sup>3</sup> measuring cylinder
- 100 cm<sup>3</sup> conical flask
- rubber bung and delivery tube to fit conical flask
- 100 cm<sup>3</sup> gas syringe OR trough/plastic container with 100 cm<sup>3</sup> measuring cylinder
- stand, boss and clamp
- stopwatch or timer
- distilled or deionised water.

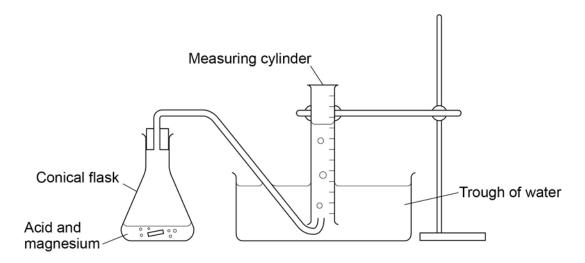
### SUGGESTED METHOD

- a) Measure 50 cm<sup>3</sup> of the 0.8 mol dm<sup>-3</sup> hydrochloric acid and add to conical flask.
- b) Set up the gas syringe in the stand (or alternative gas collection method as shown by your teacher).

### Using a gas syringe



### Using a trough



- c) Add one 6 cm strip of magnesium ribbon to the conical flask, place the bung firmly into the top of the flask and start the timer.
- d) Record the volume of hydrogen gas collected every 15 seconds for 2.5 minutes.

Repeat steps (a) to (d) using 0.4 mol dm<sup>-3</sup> hydrochloric acid, made by mixing 25 cm<sup>3</sup> of the 0.8 mol dm<sup>-3</sup> hydrochloric acid with 25 cm<sup>3</sup> of distilled or deionised water.

### Analysis

- a) Plot a graph of volume of hydrogen produced on the *y*-axis against time in seconds for each hydrochloric acid concentration. Draw a line of best fit.
- b) Draw a tangent to each line of best fit at time, t = 0 s
- c) Calculate the gradient of each tangent in order to deduce the rate of each reaction.
- d) Compare the two rate values obtained.

Measure the electromotive force (EMF) of an electrochemical cell.

## TEACHERS' NOTES

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- pieces of copper and zinc foil (about 2 cm × 5 cm)
- propanone
- 2.0 mol dm<sup>-3</sup> NaCl solution
- 1.0 mol dm<sup>-3</sup> CuSO<sub>4</sub> solution
- 1.0 mol dm<sup>-3</sup> ZnSO<sub>4</sub> solution
- emery paper or fine grade sandpaper
- two 100 cm<sup>3</sup> beakers
- plastic or glass U-tube
- cotton wool or a strip of filter paper (soaked in sodium chloride solution)
- voltmeter (digital or high impedance)
- two electrical leads with connectors for the voltmeter at one end and crocodile clips at the other end
- samples of metals, which could include titanium, iron, calcium, silver.

In the second part of the practical, magnesium can be used as the standard instead of copper.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

Measure the electromotive force (EMF) of an electrochemical cell.

### STUDENT SHEET

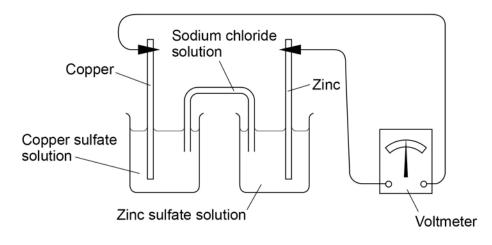
### REQUIREMENTS

You are provided with the following:

- pieces of copper and zinc foil (about 2 cm × 5 cm)
- propanone
- 2.0 mol dm<sup>-3</sup> NaCl solution
- 1.0 mol dm<sup>-3</sup> CuSO<sub>4</sub> solution
- 1.0 mol dm<sup>-3</sup> ZnSO<sub>4</sub> solution
- emery paper or fine grade sandpaper
- two 100 cm<sup>3</sup> beakers
- plastic or glass U-tube
- cotton wool soaked in sodium chloride solution
- voltmeter (digital or high impedance)
- two electrical leads with connectors for the voltmeter at one end and crocodile clips at the other end
- samples of metals.

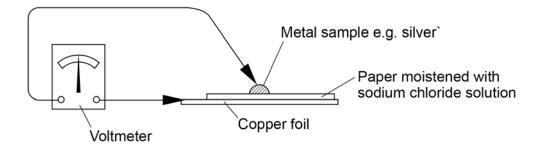
### Suggested method for setting up a standard cell

- a) Clean a piece of copper and a piece of zinc using emery paper or fine grade sandpaper.
- b) Degrease the metal using some cotton wool and propanone.
- c) Place the copper into a 100 cm<sup>3</sup> beaker with about 50 cm<sup>3</sup> of 1 mol dm<sup>-3</sup> CuSO<sub>4</sub> solution.
- d) Place the zinc into a 100 cm<sup>3</sup> beaker with about 50 cm<sup>3</sup> of 1 mol dm<sup>-3</sup> ZnSO<sub>4</sub> solution.
- e) Lightly plug one end of the plastic tube with cotton wool and fill the tube with the solution of 2 mol dm<sup>-3</sup> NaCl provided.
- f) Plug the free end of the tube with cotton wool which has been soaked in sodium chloride. Join the two beakers with the inverted U-tube so that the plugged ends are in the separate beakers.
- g) Connect the Cu(s)|Cu<sup>2+</sup>(aq) and Zn(s)|Zn<sup>2+</sup>(aq) half-cells by connecting the metals (using the crocodile clips and leads provided) provided to the voltmeter and read off the voltage.



### Suggested method for measuring comparative electrode potentials of different metals

- a) Clean a piece of copper using emery paper or fine grade sandpaper.
- b) Connect the positive terminal of the voltmeter to the copper using a crocodile clip and one of the leads.
- c) Cut a piece of filter paper to about the same area as the copper, moisten the filter paper with the sodium chloride solution and place on top of the copper.
- d) Connect the second lead to the voltmeter and use the crocodile clip on the other end of the lead to grip a piece of another metal.
- e) Hold the metal against the filter paper and note the voltage reading and sign.



- f) Repeat steps (d) and (e) with different metals and record your results in a table.
- g) Write the conventional representation for each of the cells that you have constructed
- h) Suggest how you could construct the cell with the largest EMF from the metals provided.

Carry out simple test-tube reactions to identify transition metal ions in aqueous solution: An investigation of some transition metal compounds.

### TEACHERS' NOTES

### An investigation of some transition metal compounds

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- iron(III) nitrate solution labelled 'Solution Q'
- copper(II) chloride solution labelled 'Solution R'
- ammonium iron(II) sulfate solution labelled 'Solution S'
- sodium hydroxide solution
- sodium carbonate solution
- silver nitrate solution
- 12 test tubes
- 7 dropping pipettes
- test-tube rack
- 250 cm<sup>3</sup> beaker
- access to hot water
- plentiful supply of distilled or deionised water.

The following concentrations were used to produce the sample results on the next page:

- 0.2 mol dm<sup>-3</sup> iron(III) nitrate solution
- 0.2 mol dm<sup>-3</sup> copper(II) chloride solution
- 0.5 mol dm<sup>-3</sup> ammonium iron(II) sulfate solution
- 1.0 mol dm<sup>-3</sup> sodium hydroxide solution
- 1.0 mol dm<sup>-3</sup> sodium carbonate solution
- 0.05 mol dm<sup>-3</sup> silver nitrate solution.

Additional guidance in previous practical assessments has included the advice to make up the solution of ammonium iron(II) sulfate in water, no more than one day before the practical. If any solution forms a precipitate, just sufficient drops of dilute sulfuric acid should be added to produce a clear solution.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

### SAMPLE RESULTS

### Test 1(a) and (b)

	Q	R	S
Initial	yellow solution	light blue solution	pale green solution
Add NaOH	orange/brown precipitate	deep blue precipitate	grey/green precipitate
On standing in hot water	no visible change	no visible change	no visible change

### Test 2

	Q	R	S
Addition of sodium carbonate	orange/brown precipitate and effervescence	blue/green precipitate	grey/green precipitate

### Test 3

	Q	R	S
Addition of silver nitrate	no visible change	white precipitate	light brown precipitate

Carry out simple test-tube reactions to identify transition metal ions in aqueous solution: An investigation of some transition metal compounds.

### STUDENT SHEET

Most transition metal compounds are coloured. Some of them are used as dyes and pigments. A dye is a soluble coloured compound. A pigment is an insoluble coloured compound. Both dyes and pigments have to be resistant to chemical change.

Three solutions, labelled **Q**, **R** and **S**, have been provided by a supplier as possible dyes.

- You will carry out tests on these solutions.
- You will record what you **observe** for each test.
- You should ensure that you record observations on dropwise addition, on addition to excess and on standing.
- Where no visible change is observed, write 'no visible change'.

In this task, you are **not** required to identify any of the solutions or any of the reaction products.

### REQUIREMENTS

You are provided with the following:

- three solutions labelled 'Solution Q', 'Solution R' and 'Solution S'
- sodium hydroxide solution
- sodium carbonate solution
- silver nitrate solution
- 12 test tubes
- 7 dropping pipettes
- test-tube rack
- 250 cm<sup>3</sup> beaker
- access to hot water
- plentiful supply of distilled or deionised water.

### SUGGESTED METHOD

### Test 1(a)

- a) Place about 10 drops of solution **Q** in a test tube.
- b) Add sodium hydroxide solution, dropwise with gentle shaking, until in excess.
- c) Do not discard this mixture.
- d) Repeat this test with solution **R** and then solution **S**.

### Test 1(b)

- a) Half fill a 250 cm<sup>3</sup> beaker with the freshly boiled water provided.
- b) Allow the four test tubes containing the mixtures from **Test 1(a)** to stand in the beaker of hot water for about 10 minutes.
- c) While you are waiting, begin **Test 2**.

#### Test 2

- a) Place about 10 drops of sodium carbonate solution in a test tube.
- b) Add about 10 drops of solution **Q** and shake the mixture gently.
- c) Repeat this procedure with solution  ${\bm R}$  and then solution  ${\bm S}.$

### Test 3

- a) Place about 10 drops of solution **Q** in a test tube.
- b) Add about 10 drops of silver nitrate solution and shake the mixture gently.
- c) Repeat this procedure with solution **R** and then solution **S**.
- d) Allow the four test tubes to stand for about 10 minutes.

### Prepare a pure organic solid and test its purity: To prepare a sample of aspirin

## TEACHERS' NOTES

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

This practical is likely to require at least two practical sessions.

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

### Part 1

- salicylic (2-hydroxybenzenecarboxylic) acid (~ 6.0 g)
- 100 cm<sup>3</sup> conical flask
- 10 cm<sup>3</sup> measuring cylinder
- ethanoic anhydride (~ 10 cm<sup>3</sup>)
- concentrated sulfuric acid in a dropping bottle
- 400 cm<sup>3</sup> beaker
- tripod, gauze and Bunsen burner
- thermometer (-10 °C to 110 °C)
- 250 cm<sup>3</sup> beaker
- reduced pressure filtration apparatus
- filter paper
- stirring rod
- deionised or distilled water in a wash bottle
- spatula.

### **ADDITIONAL NOTES**

For the preparation in part 1, 2 g of salicylic acid and 4 cm<sup>3</sup> of ethanoic anhydride could be used. The concentrated sulfuric acid could also be replaced by 5 drops of phosphoric acid.

These quantities fit in a pear-shaped flask and give a good yield.

Relatively new ethanoic anhydride should be used to ensure a good yield.

### Part 2

- 25 cm<sup>3</sup> measuring cylinder
- boiling tube
- ethanol (~ 15 cm<sup>3</sup>)
- thermometer (-10 °C to 110 °C)
- deionised or distilled water in a wash bottle
- 250 cm<sup>3</sup> beaker
- 100 cm<sup>3</sup> conical flask
- stirring rod
- kettle
- digital mass balance (reading to 2 decimal places).

Salicylic acid is unpleasant to work with as there is a hazard associated with skin contact, which should be avoided. Consider the use of protective gloves.

### To test the purity of an organic solid by measuring its melting point

### REQUIREMENTS

In addition to general laboratory apparatus, each student needs the following:

- pure benzenecarboxylic acid
- other pure organic solids as desired by the centre
- thermometer (0 °C to 250 °C range)
- melting point apparatus to include either: an electrothermal melting point apparatus or oil bath (Thiele tube or small beaker halffilled with mineral oil)
- tripod, gauze and Bunsen burner
- rubber ring to attach melting point tube to thermometer (if needed can be cut from rubber tubing)
- melting point tubes (if open ended, these will need sealing at one end by the technician)
- watch glass
- spatula.

You can choose to use a range of organic solids, but the target should be to ensure that:

- the solid does not decompose on heating
- the melting point of each solid is in the range 100 °C to 200 °C
- the solid gives a precise and sharp melting point temperature.

Providing a range of thermometers at the melting point stage is a good way to assess correct equipment as students will have to pick the one with the correct scale and range required.

As an extension to this practical, thin-layer chromatography can be used to examine the product at the beginning, the crude product and the purified product.

Spare supplies of all reagents specified in these notes should be available for student use (if needed).

### Prepare a pure organic solid and test its purity: To prepare a sample of aspirin.

### STUDENT SHEET

Aspirin is prepared by the acylation of salicylic acid (2-hydroxybenzenecarboxylic acid) using ethanoic anhydride as the acylating agent.

The reaction can be represented as follows.

HOOCC <sub>6</sub> H <sub>4</sub> OH	+	$(CH_3CO)_2O$	$\rightarrow$	$HOOCC_6H_4OCOCH_3$	+	CH₃COOH
salicylic acid		ethanoic anhydride		aspirin		ethanoic acid

Aspirin (2-ethanoylhydroxybenzenecarboxylic acid) is an antipyretic drug (reduces fever by lowering body temperature) and an analgesic (relieves pain).

Aspirin does not react in the acidic conditions in the stomach, but is hydrolysed in the alkaline conditions found in the intestines to produce ethanoate ions and salicylate.

(2-hydroxybenzencarboxylate) ions. Salicylates lower the body temperature of feverish patients and have a mild analgesic effect relieving headaches and other pain. The toxic dose is relatively high, but symptoms of poisoning can occur with quite small quantities.

### REQUIREMENTS

You are provided with the following:

Part 1

- salicylic (2-hydroxybenzenecarboxylic) acid
- 100 cm<sup>3</sup> conical flask
- 10 cm<sup>3</sup> measuring cylinder
- ethanoic anhydride
- concentrated sulfuric acid in a dropping bottle
- 400 cm<sup>3</sup> beaker
- tripod, gauze and Bunsen burner
- thermometer
- 250 cm<sup>3</sup> beaker
- reduced pressure filtration apparatus
- filter paper
- stirring rod
- deionised or distilled water in a wash bottle
- spatula.

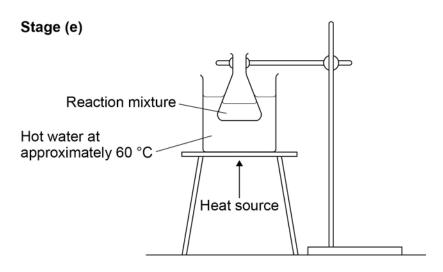
### Part 2

- 25 cm<sup>3</sup> measuring cylinder
- boiling tube
- ethanol
- thermometer
- deionised or distilled water in a wash bottle
- 250 cm<sup>3</sup> beaker
- 100 cm<sup>3</sup> conical flask
- stirring rod
- kettle
- access to a digital mass balance (reading to 2 decimal places).

### SUGGESTED METHOD

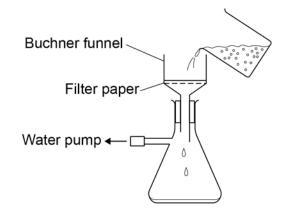
### **Part 1 Preparation**

- a) Weigh out approximately 6.00 g of salicylic acid directly into a 100 cm<sup>3</sup> conical flask.
- b) Record the mass of salicylic acid used.
- c) Using a 10 cm<sup>3</sup> measuring cylinder, add 10 cm<sup>3</sup> of ethanoic anhydride to the flask and swirl the contents.
- d) Add 5 drops of concentrated sulfuric acid to the flask and swirl the mixture in the flask for a few minutes to ensure thorough mixing.
- e) Warm the flask for about 20 minutes in a 400 cm<sup>3</sup> beaker of hot water at approximately 60 °C. The temperature in the flask should **not** be allowed to rise above 65 °C.



- f) Allow the flask to cool and pour its contents into 75 cm<sup>3</sup> of water in a beaker, stirring well to precipitate the solid.
- g) Filter off the aspirin under reduced pressure, avoiding skin contact.

Stage (g)



h) Collect the crude aspirin on a double thickness of filter paper and allow it to dry.

### Part 2 Purification

- a) Using a 25 cm<sup>3</sup> measuring cylinder, measure out 15 cm<sup>3</sup> of ethanol into a boiling tube.
- b) Prepare a beaker half-filled with hot water at a temperature of approximately 75 °C. The safest way to do this is to use a kettle of boiling water and add water from the kettle to cold water in the beaker until the temperature is at approximately 75 °C.
   NB The boiling point of ethanol is 78 °C and the temperature of the water in the beaker should not be allowed to go above this.
- c) Use a spatula to add the crude aspirin to the boiling tube and place the tube in the beaker of hot water. **Do not scrape the filter paper**.
- d) Stir the contents of the boiling tube until all of the aspirin dissolves into the ethanol.
- e) Pour the hot solution containing dissolved aspirin into approximately 40 cm<sup>3</sup> of water in a 100 cm<sup>3</sup> conical flask. If a solid separates at this stage, gently warm the contents of the flask in the water bath until solution is complete. You should avoid prolonged heating, since this will decompose the aspirin.
- f) Allow the conical flask to cool slowly and white needles of aspirin should separate.
- g) If no crystals have formed after the solution has cooled to room temperature, you may need to scratch the insides of the flask with a glass stirring rod to obtain crystals. Cool the whole mixture in an ice bath.
- h) Filter off the purified solid under reduced pressure and allow it to dry on filter paper.
- i) Record the mass of the dry purified solid.

### Analysing the effectiveness of this method of preparation of aspirin

- a) Calculate the theoretical yield of aspirin which should be formed from 6.00 g of salicylic acid.
- b) Calculate the percentage yield of aspirin from your experiment and comment on the reasons for the losses that have occurred during the preparation and the purification of the solid.
- c) Calculate the atom economy for the preparation of aspirin by this method.
- d) Consider the reasons why the alternative preparative method which uses ethanoyl chloride rather than ethanoic anhydride, is not favoured by industry even though this alternative method has a higher atom economy.

### To test the purity of an organic solid by measuring its melting point

The purity of an organic solid can be determined in part by measuring its melting point and comparing the value with the known Data Book value of the melting point for that compound. A pure dry solid will melt at a precise temperature whereas an impure solid will melt over a **range** of temperatures which are **lower** than the melting point of the pure solid.

Melting point apparatus varies in type from the most simple using an oil bath to the more sophisticated electrothermal devices. In every case, the same general principle applies that the heating of a small quantity of the solid in a thin-walled melting point tube should be undertaken slowly and with care. When melting occurs, the solid should collapse into a liquid without any change in temperature and the way in which this occurs can give a clue to the purity of the solid. Repeat measurements should be taken with further samples of the organic solid to verify the reliability of the value obtained.

The method will not work if the solid decomposes on heating.

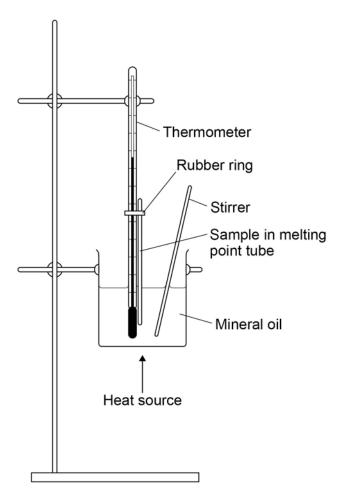
### REQUIREMENTS

You are provided with the following:

- pure benzenecarboxylic acid
- other pure organic solids as desired by the centre
- thermometer (0 °C to 250 °C range)
- melting point apparatus to include either: an electrothermal melting point apparatus or oil bath (Thiele tube or small beaker halffilled with mineral oil)
- tripod, gauze and Bunsen burner
- rubber ring to attach melting point tube to thermometer (if needed)
- melting point tubes
- watch glass
- spatula.

### SUGGESTED METHOD

- a) Powder a sample of the organic solid by crushing it gently with a spatula onto the surface of a filter paper.
- b) Fill three melting point tubes with the organic solid to a depth of approximately 0.5 cm.
- c) Set up the melting point apparatus provided and mount one of the melting point tubes ready for taking a measurement.



- d) Heat the apparatus gently and observe the temperature at which the solid collapses into a liquid. The melting point will be in the range 100 °C to 200 °C.
- e) Allow the melting point apparatus to cool and repeat the measurement of the melting point of the solid with the other two samples. If the first reading is taken as an approximate value, then the subsequent heating of the other two samples can be done much more slowly as this approximate value is approached.
- f) On the basis of the three measurements that you have taken, record the melting point of the organic solid.
- g) Ask your teacher for the Data Book value of the melting point for the solid that you have tested and compare this value with your own.

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