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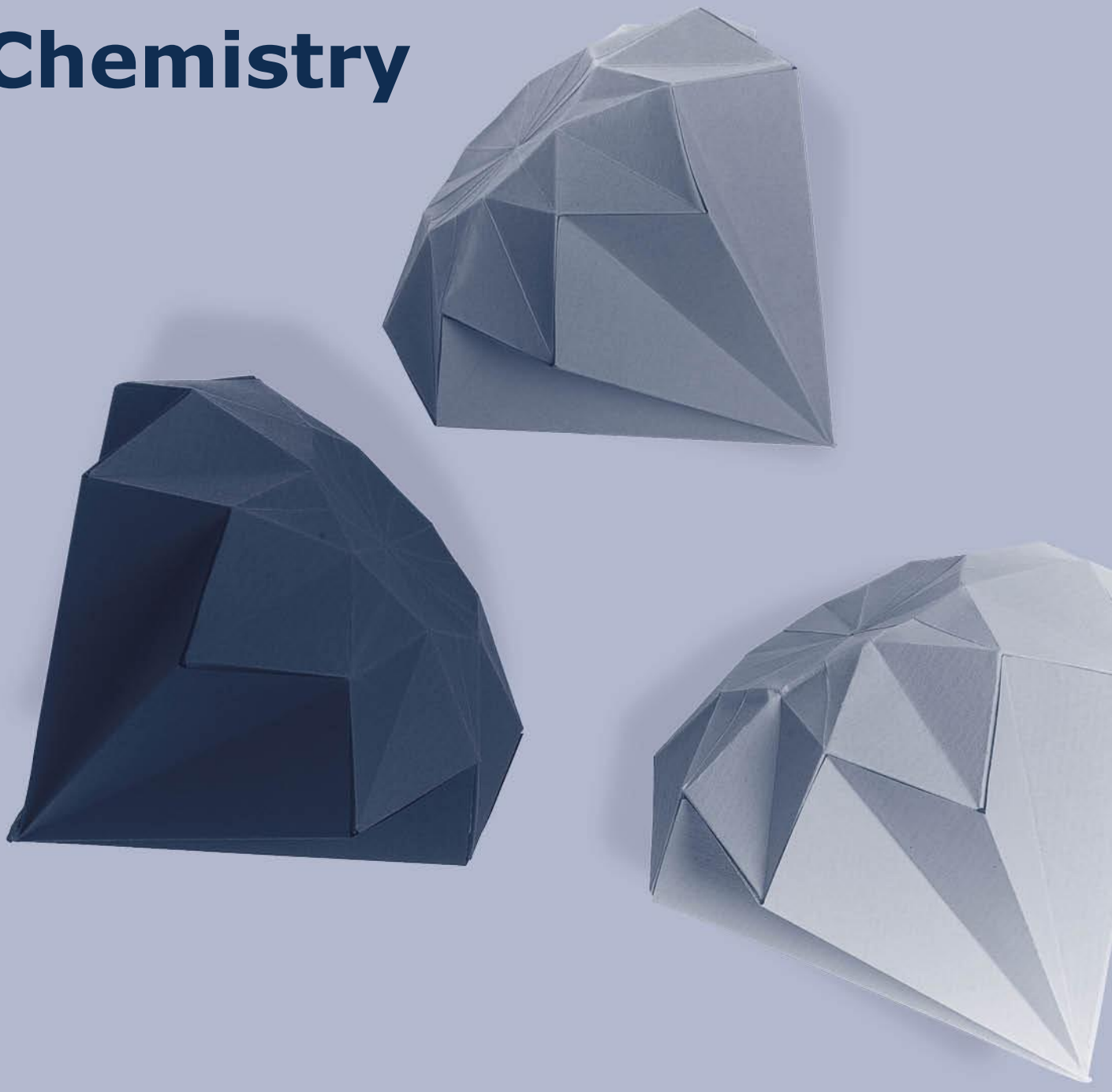
AS and A Level Chemistry



STUDENT GUIDE

ALWAYS LEARNING

PEARSON

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Introduction

Why is practical work important in Chemistry?

In your science or chemistry course at GCSE you have carried out practical work in the laboratory. Although the experiments you carried out were relatively simple you will have used your measurements and observations to make inferences and draw conclusions about the laws and principles of Chemistry.

You may have investigated the reaction between marble chips and hydrochloric acid and found that the rate of the reaction increases as the size of the chips decreases. From this observation it is reasonable to make the inference that the rate of a reaction increases as the surface area of one of the reacting substances increases.

All the laws and principles of Chemistry have been made by following a scientific method similar to that in the marble chips and acid investigation.

In your A level Chemistry course you will continue to carry out experiments making observations and measurements. From your results you will make inferences and analyse results, sometimes by drawing a graph and often by performing calculations using your quantitative results. You will prepare some compounds using practical techniques such as recrystallisation then check the purity of your product and calculate the percentage yield.

By carrying out a programme of practical work you will develop the skills that a student must have if he or she is to be regarded as a competent practical chemist. You will have to be able to work safely in the laboratory and to manage your time so as to complete your work in the time allowed. Normally you will be given written instructions so you must be able to follow these carefully.

Using a burette to deliver accurate volumes is a skill that a competent chemist must develop

Over the two years of your A level course your teacher will assess your competence as a practical chemist by letting you carry out core practicals. At the end of the course your teacher will list some of the practical work you have carried out and decide if you have passed as a competent practical chemist. If you pass, then this will be recorded on your A level certificate.

On the written papers that you sit there will be questions on practical work. By completing a programme of core practicals you should be well prepared to answer these questions.

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Developing practical skills in Chemistry

The specification includes 16 core practicals that you will be expected to complete during the two-year A level course. These are included in some, but not all, topics. For example Topic 1 *Atomic Structure and the Periodic Table* does not include a core practical but Topic 5 *Formulae, Equations and Amounts of Substance* has three. One of these is Core Practical 2: *Prepare a standard solution from a solid acid, and using it to find the concentration of a solution of sodium hydroxide*.

To carry out this core practical you will need to develop and use the following skills.

- Weigh a sample of the solid acid, record the mass, and dissolve this in water
- Transfer the solution to a volumetric flask (as shown below) and carefully add water until the solution reaches the graduation mark.

- Shake the flask carefully so that the solution is well mixed.
- Use a pipette to measure a fixed volume of the solution of acid and transfer it to a conical flask.
- Rinse out then fill a burette with a dilute solution of sodium hydroxide.
- Carry out a titration by running the solution from the burette into the conical flask until the end-point is reached. You will know this when the acid-base indicator added to the flask just changes colour-
- Read the volume from the burette and record it in a suitable table.
- Repeat the titration until you record concordant titres.

By carrying out this single core practical you will use four of the practical techniques listed in *Appendix 5c* of the specification. You will also use and apply some scientific methods including:

- following written instructions,
- making and recording observations,
- keeping appropriate records,
- presenting information and data in a scientific way using practical equipment and techniques.

Other core practicals and experiments that you carry out will allow you to gain and practice the full range of practical skills that an A level Chemistry student is expected to possess on completion of the course.

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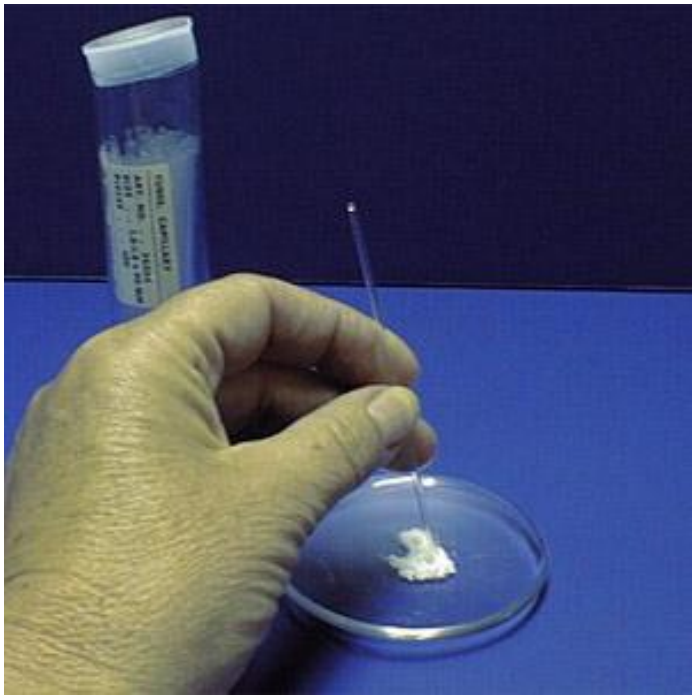
How will practical skills be tested?

There are two ways in which your practical skills will be assessed as part of your A level course.

By your teacher during your A level

When you carry out a core practical your teacher or teachers will observe you working in the laboratory. For example, if you are carrying out the preparation of aspirin you will need to heat the reagents under reflux. Your teacher will inspect your apparatus to check that it is assembled properly and is safe to use. When you have obtained some impure aspirin you will purify it by recrystallisation then dry the product and measure its melting point. Your teacher will examine your aspirin crystals and decide if you have achieved competency in the practical skills used in the preparation.



Filling a capillary tube with a solid compound ready for use in a melting point apparatus

You will be expected to keep a written record of your core practicals and your teacher will wish to see this from time to time to check your results.

At the end of the course your teacher will complete a Practical Competency Authentication Sheet listing the core practicals you have carried out. You will be assessed against common criteria agreed by all the examining boards and you will have to demonstrate that you are competent in all of the practical techniques listed in the specification. The skills which are included in this assessment are:

1. Following written procedures
2. Applying an investigative approach
3. Using a range of apparatus and equipment safely
4. Making and recording accurate observations
5. Researching, referencing and reporting

If you have shown competence in all of the techniques then you will be awarded a Pass in Chemistry Practical Endorsement and this will be recorded on your A level certificate.

No marks are awarded for the Practical Endorsement.

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By questions on exam papers at the end of your A level

There are likely to be questions in any of the three A level papers (and on the AS papers, if you are taking AS Chemistry) that test your knowledge and understanding of practical work.

At A level, there are three question papers:

Paper 1: 1h 45mins. This paper tests your knowledge of inorganic chemistry, energetics and equilibria.

Paper 2: 1h 45mins. This paper tests your knowledge of organic chemistry and kinetics.

Both Paper 1 and Paper 2 can assess your knowledge of chemical bonding and quantitative chemistry (calculations).

Paper 3: 2h 30mins. This paper tests content from all topics and questions may draw on material from two or more different topics. 50% of the marks in this paper will test your knowledge and understanding of experimental methods.

This means the following.

- Paper 3 will have lots of questions testing your practical skills and knowledge of the core practicals.
- This will include mathematical skills, as they are applied to practical work. 20% of the total marks available on all papers will be allocated to mathematical skills.
- Papers 1 & 2 will contain questions which test your knowledge and understanding of the topics listed above but they can be presented in many different forms which are linked to your practical skills e.g. topic questions may well contain data or graphs which you might be asked to interpret and explain – in which case, you will need to use the skills you have developed throughout the whole course.

An example of this type of question is:

> An experiment was carried out involving the addition of aqueous silver nitrate, followed by aqueous ammonia, to distinguish between aqueous solutions containing chloride, bromide or iodide ions.
>
> State the observations that would be made in this experiment that would show which ion is in each solution.
>
> *(6 marks)*

In order to be awarded the marks for this question you must give the colours of the precipitates formed when silver nitrate solution is added to the three halide solutions then describe what is observed when aqueous ammonia is added to the precipitates. You must include in your answer whether you add dilute or concentrated ammonia to the silver halides to test their solubility.

If you have investigated these reactions in the laboratory for yourself then you should be well prepared to answer this question.

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Paper 3 at A level will also include questions that assess your understanding of experimental methods. These will draw on your experience of experimental methods and, in particular, will draw on your experience of the core practicals.

An example of this type of question is:

Cinnamon is a spice that is extracted from the bark of the cinnamon tree.

It is extracted by crushing the soft bark with water, and then steam distilling the mixture to produce cinnamon oil.

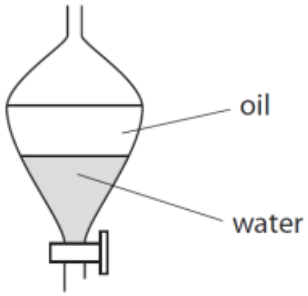
The steam distillate is washed with saturated sodium chloride solution and the oil separated and dried.

(i) Draw a diagram of the apparatus you would use to separate the oil, labelling the oil.

(2 marks)

[Densities: cinnamon oil 1.050 g cm^{-3}; sodium chloride solution 1.122 g cm^{-3}]

As part of your practical course you will have separated non-miscible liquids, so you should be able to draw a diagram of a separating funnel. From your practical experience you will understand that the liquid with the lower density forms the upper layer. In this experiment the cinnamon oil will be the upper layer.



(ii) Give the name of a chemical that could be used to dry the oil.

(1 mark)

One of the techniques used in organic synthesis that you should understand is that of drying liquids. If you have carried out the preparation of an organic liquid then you will have used a drying agent. Acceptable answers are: addition of (anhydrous) sodium sulfate / calcium chloride / magnesium sulfate.

(iii) State the change in appearance of the oil when it is being dried.

(1 mark)

In Core Practical 6: *Chlorination of 2-methylpropan-2-ol* you will have dried the liquid product by shaking it with a drying agent. As it dried you should have observed that the liquid changes its appearance from **cloudy to clear**, and this is the answer required in the question.

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Chemistry practical skills: recording data

As part of your practical activities you will need to keep a written record of your experimental results. This may be in the form of a laboratory notebook or a loose leaf file. Usually your teacher will provide a set of instructions for each experiment often with tables and spaces for your results. Sometimes you will be expected to design and plan your own results tables.

Making and recording observations, keeping appropriate records and presenting information and data are part of the scientific method and are skills that you will be expected to develop during the course.

Recording observations

In Core Practical 15: *Analysis of some inorganic and organic unknowns,* you will carry out reactions, on a test tube scale, to test for the presence of inorganic anions and cations and also organic functional groups.

It is important when recording your observations that you use the correct scientific language. Some changes that you may observe during tests are listed in the table below with recording of the change using the correct chemical language.

Test	Example of correct recording of observation
A colour change in solution	Orange solution turns green
On mixing two solutions a solid forms	Cream precipitate forms
A precipitate dissolves in excess reagent	Green precipitate dissolves in excess to form a green solution
A gas is given off	Bubbles of gas or effervescence
Tests on a gas	The gas turned damp red litmus paper blue
A coloured flame test	Lilac flame
A solid is added to water	Solid dissolves to form a colourless solution
A reaction is exothermic	The mixture feels warm / hot
A reaction is endothermic	The mixture feels cold
A coloured solution loses its colour	The yellow solution turns colourless

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Things to avoid when recording observations.

- Referring to 'layers'. Almost certainly if layers are present in a test tube this is due to inadequate mixing of the reagents if both are in aqueous solution.
- Recording that a 'gas is evolved'. The recording should be that bubbles or effervescence are observed.
- Describing a solution as 'clear instead of 'colourless'. All solutions are clear but not all are colourless.
- Referring to a solid 'dissolving' when it is actually reacting to form a soluble substance e.g. adding a solid carbonate to an acid.

Exercises

1. A series of tests was carried out on **X**, a white solid, which is known to contain one cation and one anion.

   (a) **X** gave a pale green colour in a flame test. Give the name or formula of the cation in **X**.

   (1)

   (b) Dilute nitric acid was added to an aqueous solution of **X**, and then aqueous silver nitrate was added to the mixture. A white precipitate formed, which dissolved in dilute aqueous ammonia.
   Give the name or formula of the anion in **X**.

   (1)

   (c) **Dilute** sulfuric acid was added to an aqueous solution of **X**.

   (i) What change would be observed?

   (1)

   (ii) Write an ionic equation for the reaction in (c)(i). Include state symbols.

   (2)

   (d) A few drops of **concentrated** sulfuric acid were added to a small portion of **solid X** in a test tube. Misty fumes, but no other vapours, were seen. Identify these fumes by name or formula.

   (1)

2. A colourless liquid, compound **Y**, was extracted from raspberries. **Y** has the molecular formula $C_{10}H_{12}O_2$ and contains a benzene ring.

   (a) What would you expect to see if a sample of compound **Y** was burned in air?

   (1)

   (b) A series of tests was carried out on compound **Y**.
   In each test, state what you can deduce about the structure of compound **Y** from the results described.

   (i) **Y** forms a white precipitate with bromine water.

   (1)

   (ii) **Y** forms an orange precipitate with 2,4-dinitrophenylhydrazine.

   (1)

   (iii) Fehling's solution remains blue when warmed with compound **Y**.

   (1)

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Recording measurements

In many of the core practicals you will use apparatus to make measurements. You should read off a measurement from a particular piece of apparatus and record the measurement as accurately as the precision of the apparatus allows.

Measurement	Apparatus	Recording measurement
Mass	Balance	To 0.01 g on a 2-place balance To 0.001 g on a 3-place balance
Time	Timer	Normally to the nearest second
Volume of liquids	Pipette Burette Measuring cylinder	One volume only of 25.0 cm^3 Each volume to the nearest 0.05 cm^3 100 cm^3 capacity to 1.0 cm^3 10 cm^3 capacity to 0.1 cm^3
Volume of gas	Gas syringe	100 cm^3 capacity to 1.0 cm^3
Temperature	Thermometer	0–110°C range to 1.0°C 0–50°C range to 0.5°C
pH	pH meter	To nearest 0.1 e.g. pH 6.8

Titrations

You will do several titrations during your A level course, in different areas of chemistry. It is important that you learn not only the correct technique, but also the standard way of recording titration data.

- You should always record burette volumes to the nearest 0.05 cm^3. If a volume is exactly 24 cm^3 you should record it as 24.00 cm^3.
- When you carry out a titration you should try to obtain at least two titres within 0.20 cm^3 of each other and average these to obtain a mean.
- You should also record a mean titre to the nearest 0.05 cm^3.

Exercise

3. A student's burette readings are shown in the table below.

Titration	1	2	3	4
Burette reading (final) / cm^3	25.00	24.40	24.40	25.70
Burette reading (initial) / cm^3	1.00	2.10	1.60	3.30
Titre / cm^3				

Complete the table then, by choosing suitable titres, calculate a mean titre.

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Chemistry practical skills: use of mathematics

Significant figures

The final result of a calculation using your data from a quantitative practical should only be given to the number of significant figures as is suggested by the measurements made in the exercise. The number of significant figures in the final calculated value should be the same as the number of significant figures in the least accurate measurement.

Example: An experiment to determine ΔH of an exothermic reaction involves a weighing of 1.395 g and a temperature rise of 8.7°C. A student calculates a value of ΔH from these measurements and records it as 87.834 kJ mol^{-1}.

Since the temperature was measured to only two significant figures the student should have recorded the value as ΔH = -88 kJ mol^{-1}.

Exercise

4. The table below shows examples of the measurements made in some practicals and a student's calculated value of the quantity to be determined.

   Complete the final column to give the value to an appropriate number of significant figures based on the measurements shown.

Practical	Examples of measurements	Student's calculated value	Value to appropriate number of SFs
To determine the activation energy of a reaction	15.0 s 53°C	+62.351 kJ mol^{-1}	
Titration to determine the concentration of an acid	1.351 g 24.75 cm^3	0.10345 mol dm^{-3}	
To determine the *K*a of a weak acid	pH 3.7 25.75 cm^3	5.774 × 10^{-4} mol dm^{-3}	
To find the RMM of a volatile liquid	0.284 g 80 cm^3	85.885 g mol^{-1}	

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Errors and uncertainties

You should understand that any piece of apparatus (burette, thermometer, balance, pH meter etc) used in a quantitative practical has a **measurement uncertainty** associated with its use.

Even if the apparatus is used carefully and properly the uncertainty leads to an **error** in the reading and in the final value calculated from the readings.

The manufacturer of the apparatus supplies the **measurement uncertainty**. A piece of apparatus with a low measurement uncertainty is said to be **precise**.

The percentage uncertainty due to the uncertainty in the apparatus may be calculated:

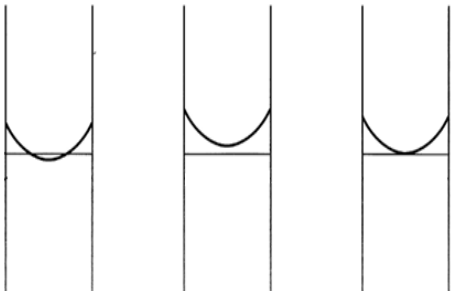
$$\% \text{ uncertainty} = \frac{\text{uncertainty in the equipment}}{\text{reading}} \times 100\%$$

Errors in measurement uncertainty of the apparatus are **systematic errors.**

Errors resulting from careless use of the apparatus or poor technique are **random errors**.

Random errors can be reduced if you repeat the experiment several times and take the **mean.** This should cause them to cancel out and give you a more accurate result

However if there is a systematic error, then the final result will always be inaccurate, however many times it is repeated.



A skilled chemist will top up a volumetric flask until the bottom of the meniscus is exactly on the graduation mark so minimising the random error.

Example

A two decimal place balance has a measurement uncertainty of ±0.005 g.

When the balance is used to weigh 0.56 g the percentage uncertainty is

$$\frac{2 \times \pm 0.005}{0.56} \times 100\% = \pm 1.8\%$$

The percentage uncertainty decreases as the reading increases. When the same balance is used to weigh 4.56 g the percentage uncertainty is

$$\frac{2 \times \pm 0.005}{4.56} \times 100\% = \pm 0.22\%$$

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Exercise

5. Complete the table below.

Apparatus	Uncertainty	Measurement	Number of readings taken	% Uncertainty
Pipette	±0.06 cm^3	25.0 cm^3	1	
Burette	±0.05 cm^3	23.45 cm^3	2	
Thermometer	±1.0°C	62.0 °C	1	

Combining uncertainties

Calculating a value from the results of a practical will involve using measurements from more than one piece of apparatus, each having its own uncertainty. The uncertainties for the different types of apparatus may be combined to find an approximate value for the overall uncertainty in the final calculated value.

Example

The results of a titration are used to calculate a molar mass of a compound as 278 g mol^{-1}.

The percentage uncertainties in the balance, pipette and burette readings used in the calculation are 0.12%, 0.24% and 0.38%.

Total percentage uncertainty = 0.12 + 0.24 + 0.38 = 0.74%

Total uncertainty in the molar mass is

$$\frac{0.74 \times 278}{100} \approx 2.1$$

Molar mass = 278 ±2.1 g mol^{-1}

Exercise

6. The pK_a of a weak acid is calculated as 4.40 from the results of an experiment.

   The percentage uncertainty in the pH meter, pipette and burette readings used in the calculation are 2.5%, 0.34% and 0.26%.
   Calculate the uncertainty in the pK_a value.

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Calculations

In order to develop your skills, knowledge and understanding of the subject you will have to gain competence in area of mathematics relevant to Chemistry. Appendix 6 of the Specification lists the mathematical skills that you need to develop during your course.

Some of the mathematical skills required in order to be able to process the results of practical work are listed below.

Titrations

Mathematical skills: Select appropriate titration data and calculate an arithmetic mean.

Exercise

7. A student's burette readings are shown in the table below.

Titration	1	2	3	4
Burette reading (final) / cm^3	27.00	27.50	26.60	26.65
Burette reading (initial) / cm^3	0.00	1.00	0.00	0.00
Titre / cm^3				

Complete the table then, by choosing suitable titres, calculate a mean titre.

pH and pK_a

Mathematical skills: Use logarithmic functions on your calculator.

$pH = -\log_{10}[H^+]$ $[H^+] = 10^{-pH}$ $pK_a = -\log_{10}[K_a]$

Exercise

8. A solution of a weak acid, HA, is found by a titration to have a concentration of 0.120 mol dm^{-3}. The pH of the acid is 2.88.

(a) Calculate the hydrogen ion, $[H^+]$, in this solution of HA.

(b) Use the expression $K_a = \dfrac{[H^+]^2}{[HA]}$ to calculate K_a

(c) Calculate pK_a.

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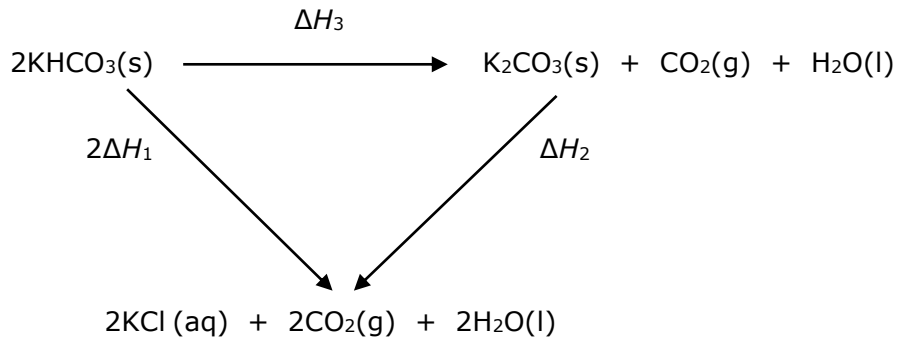
Energetics – Hess's Law

Mathematical skills: Solve algebraic equations.

Exercise

9. A student uses a Hess's Law experiment to investigate the enthalpy change for the decomposition of potassium hydrogencarbonate to potassium carbonate.



The following enthalpy changes are measured:

ΔH_1 = +28.5 kJ mol^{-1} ΔH_2 = -27.6 kJ mol^{-1}

Calculate ΔH_3.

Rates

Mathematical skills: Use exponential functions on your calculator.

Exercise

10. The following times are recorded in a rates investigation.

82s, 52s, 36s, 27s and 18s

Calculate the natural logarithm (ln) of the reciprocal of each time.

Mathematical skills: Substitute numerical values into algebraic equations using appropriate units for physical quantities.

Exercise

11. Using the results of an experiment, a rate expression for a reaction between compounds A and B is worked out as:

$$\text{rate} = k[A][B]^2$$

In one of the experiments, the following results were obtained:

Initial concentration of A / mol dm^{-3}	Initial concentration of B / mol dm^{-3}	Initial rate / mol dm^{-3} s^{-1}
0.150	0.100	2.90 x 10^{-5}

Calculate the value, with units, of the rate constant, *k*.

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Graphs

For some practicals you will have to process your results by drawing and then interpreting a graph.

Some useful points to note when drawing graphs include:

- Plot the pre-determined quantity (e.g. volume of solution in a rates practical) on the *x*-axis
- Plot the dependent variable (e.g. time for a colour to appear in a rates experiment) on the *y*-axis
- Chose the scales so that the results are spread out as far apart as the size of the grid allows. However, do not do this if it causes you to use a scale this is difficult to read e.g. using 1 cm on the axis to represent 3 or 4 units might spread the readings better than using 1 cm to represent 5 units but the scale would be hard to read
- The origin (0, 0) does not need to be included on either scale if it is not relevant e.g. in an energetics practical your temperature readings may be between 15°C and 30°C, but you may need to allow for extrapolation to read off the maximum temperature. A scale from 15°C to 35°C will be appropriate
- Always label each axis with the quantity being plotted and its units
- Join the points you have plotted with a continuous straight line of best fit or a smooth curve of best fit as appropriate. Since the readings are subject to experimental error your line need not necessarily pass through every point.
- Points should never be joined by a series of short, straight lines

Exercises

12. In an experiment sodium hydroxide solution is added to hydrochloric acid. The temperature of the solution is measured after each portion of sodium hydroxide has been added.

Total volume of NaOH added / cm^3	0.00	5.00	10.00	15.00	20.00	25.00	30.00	35.00	40.00
Temperature / °C	21.0	22.6	24.4	26.2	28.0	28.4	27.3	26.2	25.0

Draw a graph of the results in the table.

Draw a straight line of best fit through the points as the temperature rises and a second line of best fit as the temperature falls.

Extend the lines until they meet then read off the highest temperature reached and the volume of sodium hydroxide added at the maximum temperature.

13. In an experiment to follow the rate of a reaction, the concentration of one of the reagents, X, is measured at different times.

Time / s	0	20	40	60	80	100	120	140	160
[X] / mol dm^{-3}	0.200	0.160	0.134	0.113	0.096	0.078	0.066	0.054	0.045

Draw a graph of the results in the table, joining the points with a smooth curve of best fit.

Draw a tangent to the curve at 80 seconds and calculate the slope of the curve at this time. Include units in your answer.

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Chemistry practical skills: preparations

The 16 core practicals include four **preparations**.

Three of these are the preparations of organic compounds and one is for the preparation of a transition metal complex salt.

To complete these preparations successfully you will have to follow written instructions and use, safely and correctly, a range of practical equipment and materials.

In particular you will have to use the practical techniques listed below:

- Use a water bath or electric heater or sand bath for heating
- Set up and use apparatus for heating under reflux and distillation
- Filtration, including the use of fluted filter paper and reduced pressure filtration
- Purify a solid product by recrystallisation and a liquid product using a separating funnel
- Use melting point apparatus
- Safely and carefully handle solids and liquids, including corrosive, irritant, flammable and toxic substances.

Assessing your practical skills carrying out preparations

In order to assess your practical competence in the laboratory your teacher will provide you with written instructions and the equipment and materials for the preparation.

You may be allowed to work in pairs with another student to carry out the preparation.

During the preparation your teacher will check that you are assembling and using the apparatus correctly and safely.

If the product of an organic preparation is crystals you will have to dry and weigh them, and then measure and record their melting point.

You may have to calculate and record the percentage yield of the product.

Your teacher will decide if you have achieved competency in the skills needed to successfully complete the preparation.

Assessing preparation skills on written exam papers

Although all of the written papers may include some questions that relate to Chemistry practical work, Paper 3 will include questions that test your knowledge and understanding of the practical work involved in preparations.

These questions will draw on your experience of the preparations in the core practicals.

So that you may answer these questions successfully make sure that you can do the following:

- Draw labelled diagrams of the apparatus used to carry out:
    - distillation including steam distillation
    - heating under reflux
    - filtration under reduced pressure (Buchner funnel)
    - separation of two liquids using a separating funnel
- Calculate percentage yields

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Exercise

14. The procedure below can be used to make 1-bromobutane.

1. Place a mixture of water, sodium bromide and butan-1-ol in a round-bottomed flask.
2. Slowly add a suitable volume of concentrated sulfuric acid to this mixture whilst it is also shaken and cooled.
3. When this addition is complete, heat the mixture under reflux for about 45 minutes.
4. Rearrange the apparatus for distillation and distil off the crude 1-bromobutane, collecting the distillate between 95°C and 105°C.
5. Shake the 1-bromobutane first with water, then with dilute sodium carbonate solution.
6. Separate the 1-bromobutane from the aqueous layer, add some anhydrous calcium chloride and leave the mixture to stand.
7. Decant the 1-bromobutane from the calcium chloride.

(a) (i) Explain why sodium bromide and sulfuric acid are required in step 2.

(1)

(ii) What would be the effect on this preparation if concentrated sulfuric acid was added in step 2 without water having been added in step 1? Justify your answer.

(2)

(b) Explain why the acid must be added slowly and with cooling in step 2.

(1)

(c) Draw a labelled diagram of the apparatus that could be used to carry out the distillation in step 4.

(4)

(d) Explain why the 1-bromobutane is shaken with sodium carbonate solution in step 5.

(1)

(e) What is the purpose of the calcium chloride in step 6?

(1)

(f) Suggest how you would obtain pure 1-bromobutane after step 7.

(1)

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Core practicals

The core practicals are an integral part of your course. They are not there to get you to demonstrate some text book 'fact' or recall some simple information. They are there to help you develop the whole range of practical and mathematical skills which are essential to chemists and which will be tested in the written assessments.

List of core practicals

1. Measuring the molar volume of a gas
2. Preparation of a standard solution from a solid acid
3. Finding the concentration of a solution of hydrochloric acid
4. Investigation of the rates of hydrolysis of halogenoalkanes
5. The oxidation of ethanol
6. Chlorination of 2-methylpropan-2-ol with concentrated hydrochloric acid
7. Analysis of some inorganic and organic unknowns
8. To determine the enthalpy change of a reaction using Hess's Law
9. Finding the K_a value for a weak acid
10. Investigating some electrochemical cells
11. Redox titration
12. The preparation of a transition metal complex
13. Following the rate of the iodine-propanone reaction by a titrimetric method and investigating a 'clock reaction' (Harcourt-Esson, iodine clock)
14. Finding the activation energy of a reaction
15. Analysis of some inorganic and organic unknowns
16. The preparation of aspirin

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Questions on core practicals

1. Measuring the molar volume of a gas

Greg is doing an experiment to find the molar volume of gas.

He puts 50 cm^3 of 1.0 mol dm^{-3} hydrochloric acid in a flask. The flask has a bung, with a delivery tube connected to a gas syringe.

Greg plans to add 0.40 g of calcium carbonate to the hydrochloric acid in the flask, quickly insert the bung, and measure the volume of gas collected.

(a) Write a chemical equation, including state symbols, for the reaction that takes place.

(b) Show, by calculation, that the HCl is in excess.

(c) Which piece of apparatus should Greg use to measure out the hydrochloric acid? Give a reason for your answer.

(d) Comment on the mass of $CaCO_3$ that the student plans to use.

(e) Greg does not place the bung tightly into the flask as the reaction takes place and some gas is lost. What affect will this have on his calculation of the molar mass of the gas?

2. Preparation of a standard solution from a solid acid

Kathryn prepares a standard solution of sodium hydrogensulfate by weighing 0.900 g of the solid into a beaker. She adds approximately 25 cm^3 of distilled water and stirs with a glass rod until the solid dissolves. She uses a funnel to transfer this solution into a 250 cm^3 volumetric flask.

Finally, she rinses the beaker and the glass rod with distilled water, adds this to the flask, and makes up to the mark with distilled water.

(a) Describe the process of 'making up to the mark'.

(b) What next step should Kathryn take before using this standard solution for a titration?

(c) Calculate the concentration of this standard solution of sodium hydrogensulfate. Give your answer to an appropriate number of significant figures.

(d) Another student repeats Kathryn's method, but does not rinse the beaker and glass rod. Their teacher estimates that 0.5 cm^3 of solution remains in the beaker and on the glass rod. Use this information to estimate the percentage error introduced into this experiment by failing to rinse the apparatus.

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3. Finding the concentration of a solution of hydrochloric acid

Ahmed wants to check the concentration of a sample of concentrated hydrochloric acid.

He uses a 10.0 cm^3 pipette to transfer a sample of the concentrated acid to a 250 cm^3 volumetric flask. He makes up the solution to the mark with distilled water and mixes thoroughly.

He then fills a clean burette with a standardised solution of 0.350 mol dm^{-3} sodium hydroxide solution.

Ahmed uses a suitably rinsed pipette to transfer 25.0 cm^3 of the diluted hydrochloric acid to a conical flask. He titrates this with the sodium hydroxide solution, using an indicator.

The table shows his data.

	1st titration	**2nd titration**	**3rd titration**
Final burette reading / cm^3	24.60	48.85	24.35
Initial burette reading / cm^3	0.00	24.60	0.00

(a) Ahmed uses a 'suitably rinsed' pipette. What does this mean?

(b) Ahmed wants to choose an indicator to use in this titration.

(i) Give a reason why UI is not used in titrations.

(ii) Give the name of an indicator that Ahmed could use, stating the colour change he would see at the end point.

(c) Ahmed calculates that his average titre value is 24.30 cm^3. Show how he arrived at this answer.

(d) Use Ahmed's data to calculate the original concentration of the concentrated hydrochloric acid.

[Hint: you will need to work out the amount, in moles, of HCl using the mean titre value, and therefore the amount present in the 100 cm^3 volumetric flask. Lastly, you need to relate this answer to the original concentration of the HCl]

4. Investigation of the rates of hydrolysis of halogenoalkanes

Rosemary investigates the rate of hydrolysis of 1-chlorobutane, 1-bromobutane and 1-iodobutane.

She takes 1.0 cm^3 of each halogenoalkane, dissolves this in ethanol, and adds 2 cm^3 of silver nitrate solution. She times the time taken for a precipitate to form.

(a) Does taking equal volumes of the three halogenoalkanes increase the validity of Rosemary's experiment? Explain your answer.

(b) What colour precipitate will Rosemary record in each experiment?

(c) Rosemary's results table is shown below.

1-chlorobutane	5mins 12 s
1-bromobutane	104 s
1-iodopropane	14.65 s

Criticise the way in which this data is recorded.

(d) Rosemary suggested extending her investigation by using 1-chlorobutane, 2-bromobutane and 2-iodo-2-methylpropane. Comment on this suggestion.

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5. The oxidation of ethanol

Jason prepares ethanal by adding ethanol, slowly, to a hot solution of acidified potassium dichromate(VI) solution. He distils off the ethanal as it forms.

(a) Why is it important that the ethanol is added slowly to the hot oxidising agent?

(b) Jason distils off the ethanal as it forms, rather than heating the ethanol and the oxidising agent together under reflux. Explain why.

(c) Ethanal boils at 21°C. How does this information influence the way in which ethanal is collected in this experiment?

(d) Describe how Jason could show that the product:

(i) contained ethanal

(ii) was pure.

6. Chlorination of 2-methylpropan-2-ol with concentrated hydrochloric acid

Hazel has been asked to convert butan-2-ol into 2-chlorobutane. She places 10 cm^3 of butan-2-ol into a separating funnel, and adds an equal volume of concentrated hydrochloric acid. She puts a stopper in the separating funnel and shakes it for 5 minutes.

She leaves the mixture to settle, then uses the separating funnel to separate the aqueous layer, containing the hydrochloric acid, from the organic layer, containing the 2-chlorobutane.

(a) Explain one safety precaution that Hazel should take in this experiment.

(b) Draw a labelled diagram of the separating funnel after the shaken mixture has settled, indicating the nature of the layers.
[densities in g cm^{-3}: 2-chlorobutane = 1.397, butan-2-ol = 0.808, concentrated HCl = 1.18]

(c) Hazel leaves the stopper in the separating funnel when she tries to separate the layers. Describe what will happen.

(d) Describe how a pure sample of 2-chlorobutane can be extracted from the organic layer.

(e) Hazel collects 2.64g of 2-chlorobutane. Calculate the percentage yield.

7. Analysis of some inorganic and organic unknowns

Cliff has four bottles labelled A, B, C and D. He knows that the substances are NaCl, NaI, K_2CO_3 and K_2SO_4.

(a) Describe chemical tests, with their results, that Cliff can do to distinguish between these solids.

(b) When testing for halide ions, a little nitric acid is added to the test solution before the silver nitrate solution. Explain why.

(c) An organic liquid has the formula C_3H_6O. The liquid decolourises bromine water; and reacts with PCl_5 to produce misty fumes. Identify the functional groups present and hence draw a possible structural formula for the liquid.

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8. To determine the enthalpy change of a reaction using Hess's Law

Assie wants to find $\Delta_r H$ for the reaction: $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$

She decides to react solid calcium carbonate and solid calcium oxide, separately, with dilute hydrochloric acid; and find $\Delta_r H$ using Hess' Law.

(a) Draw a suitable Hess's Law cycle. Use ΔH_1 to show the enthalpy change for the reaction of calcium carbonate with dilute HCl(aq) and ΔH_2 to show the enthalpy change for the reaction of calcium oxide with dilute HCl(aq).

(b) Many enthalpy change reactions are performed in polystyrene cup. How does this help the accuracy of the experiment?

(c) In the reaction between calcium carbonate and dilute HCl(aq), Assie uses 1.00 g of $CaCO_3$, and 25.0 cm^3 of 1.0 mol dm^{-3} HCl (an excess). She measures a temperature change of +5.6°C.

(i) Calculate the heat energy released in this experiment.

(ii) Hence, calculate the enthalpy change, in kJ mol^{-1} of calcium carbonate.

(iii) Estimate the overall measurement uncertainty in Assie's experiment, stating your assumptions.

9. Finding the K_a value for a weak acid

Bosun is investigating the weak acid, propanoic acid. He places 25.0 cm^3 of propanoic acid into a conical flask and measures its pH using a pH meter.

He adds 2.0 cm^3 of sodium hydroxide solution, of a similar concentration, from a burette, swirls the flask and measures the pH again. He repeats this procedure, plotting the pH against the volume of sodium hydroxide added on a graph as he continues.

Bosun wants to use the data to calculate the K_a value of propanoic acid.

(a) Describe how Bosun should use his results to find K_a.

(b) Bosun's method has a major flaw. Identify this flaw and explain how Bosun can alter this experiment to avoid it.

(c) The electrode of a pH meter is usually stored in a buffer solution. Explain why this is important in an experiment such as this.

(d) Bosun finds that $K_a = 1.349 \times 10^{-5}$ mol dm^{-3}. The acid has an initial pH of 2.58. Calculate the concentration of the acid.

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10. Investigating some electrochemical cells

Helen sets up an electrochemical cell, consisting of a copper electrode in a 1 mol dm^{-3} solution of copper(II) sulfate and a zinc electrode in a 1 mol dm^{-3} solution of zinc(II) sulfate.

Helen connects the two cells with a salt bridge, made from a strip of filter paper soaked in potassium nitrate solution.

(a) Why is a salt bridge necessary in this experiment?

(b) Why is potassium nitrate solution used in the salt bridge?

(c) When Helen connects her voltmeter to the electrodes, it reads -1.10 V, instead of +1.10V. Give a reason why.

(d) Helen's friend sets up a similar experiment, with the same apparatus, but with a different solution of copper(II) sulfate. She records a value of +1.05 V. Give a reason why this difference has occurred.

11. Redox titration

Rhubarb leaves are toxic, because they contain ethanedioic acid, $(COOH)_2$. Ethanedioic acid can be oxidised to produce carbon dioxide and water:

$$(COOH)_2 \rightarrow 2CO_2 + 2H^+ + 2e$$

Richard wants to find how much ethanedioic acid is in a rhubarb leaf. He chops up a rhubarb leaf and leaves it in hot water for 10 minutes. He transfers the liquid into a 250 cm^3 volumetric flask, with washings, makes up to the mark, and mixes the solution.

He titrates 25.0 cm^3 of the rhubarb leaf solution against 0.00200 mol dm^{-3} potassium manganate(VII) solution. 10.50 cm^3 is needed for complete reaction.

(a) What practical problems are often encountered when using potassium manganate(VII) solution in a burette?

(b) It is common to add the first 1 cm^3 or so of potassium manganate(VII), wait for it to react, and then proceed more quickly with the titration. Give a reason why this procedure is used.

(c) Explain why this titration does not need an indicator.

(d) Calculate the mass of ethanedioic acid in the rhubarb leaf.

[Molar mass of ethanedioic acid = 90 g mol^{-1}]

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12. The preparation of a transition metal complex

Damian has found instructions for the preparation of chrome alum in an old chemistry book. Chrome alum is a double salt of potassium sulfate and chromium sulfate, with the formula $K_2SO_4.Cr_2(SO_4)_3.24H_2O$.

106. Chrome Alum, $K_2SO_4, Cr_2(SO_4)_3, 24H_2O$

This salt may be prepared by the reduction of potassium dichromate in acid solution:—

$$K_2Cr_2O_7 + 4H_2SO_4 + 3C_2H_5OH = K_2SO_4 + Cr_2(SO_4)_3 + 3C_2H_4O + 7H_2O.$$

Pour 12.5 c.c. of concentrated sulphuric acid into 100c.c. of water and dissolve in this solution 15 g. of potassium dichromate. Cool the solution in ice water and add 5 c.c. of absolute alcohol with constant stirring. The temperature should not rise above 60°

Pour the solution into an evaporating basin and set aside for crystallisation. Select a few good crystals from the first formed and allow them to grow in the slightly diluted clear solution, turning them regularly on different faces. Large, perfect crystals of a deep violet colour are obtained.

(a) What is the oxidation state of chromium in chrome alum?

(b) In the first step of the reaction, concentrated sulfuric acid is added to water in order to dilute the acid. Suggest why the dilution of sulfuric acid is not performed by adding water to the acid.

(c) Use information in the passage to describe the heat energy change that occurs when ethanol (*absolute alcohol*) is added to the acidified solution of potassium dichromate.

(d) What is the maximum mass of chrome alum that can be made from 15 g of potassium dichromate?

13. Following the rate of the iodine-propanone reaction by a titrimetric method and investigating a 'clock reaction' (Harcourt-Esson, iodine clock)

John investigates the kinetics of the oxidation of glucose by acidified potassium manganate(VII) solution, $KMnO_4(aq)$. His method is:

1. Measured volumes of glucose solution, sulfuric acid and water are added to a conical flask.
2. A measured volume of potassium manganate(VII) solution is added to the flask. The mixture is gently swirled and a stopwatch started.
3. The time taken for the mixture in the flask to change colour is recorded and the initial rate of the reaction is then calculated.
4. The experiment is repeated using different volumes of the solutions.

continued

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John collects these results:

Exp't	Glucose / cm^3	Sulfuric acid / cm^3	$KMnO_4$(aq) / cm^3	Water / cm^3	Initial rate / mol dm^{-3} s^{-1}
A	20.0	20.0	10.0	0.0	1.0×10^{-5}
B	20.0	20.0	5.0	5.0	5.0×10^{-6}
C	10.0	20.0	10.0	10.0	9.8×10^{-6}
D	10.0	10.0	10.0	20.0	4.9×10^{-6}

(a) Give the name of the piece of apparatus that should be used to measure the volumes used in each experiment. Justify your choice.

(b) What colour change would you see in step 3?

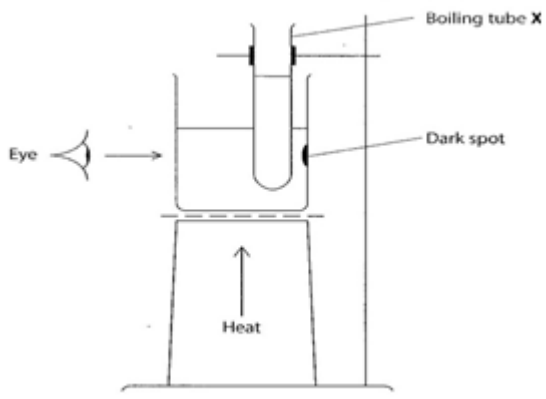
(c) Explain why water is added to the flask in experiments B, C and D.

(d) Suggest a technique that could be used to monitor the change in potassium manganate(VII) concentration during the reaction.

(e) State the order with respect to glucose, sulfuric acid and potassium manganate(VII) and hence write the rate equation for the reaction.

14. Finding the activation energy of a reaction

Henna uses this apparatus to investigate the activation energy of the reaction between sodium thiosulfate and hydrochloric acid.



She does the reaction at four temperatures. Her results table is shown below.

Experiment	Temperature / °C	Temperature / K	1 ÷ T / K^{-1}	ln (rate constant)
A	60.0	333.0		−1.60
B	49.5	322.5		−2.60
C	38.5	311.5		−3.75
D	25.5	298.5		−5.20

(a) How does Henna know when each reaction is complete?

(b) Complete the table.

(c) Plot a graph of ln (rate constant) against 1 ÷ temperature and calculate the gradient of this graph.

(d) Use your answer to (c) to calculate the activation energy, *E*a, for this reaction. Include a sign and units in your answer.

Gradient $= -\underline{Ea} \div R$ where $R = 8.31$ J K^{-1} mol^{-1}

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15. Analysis of some inorganic and organic unknowns

Three compounds **A**, **B** and **C** are subjected to a series of chemical tests. The three compounds are isomers with molecular formula C_3H_6O.

A and **B** contain only one functional group, but **C** contains two separate functional groups. None of the three compounds contains a ring of atoms. In each of the three compounds the oxygen atom is bonded to only one carbon atom.

(a) **A** and **B** are tested separately with 2,4-dinitrophenylhydrazine solution and both give an orange precipitate. When **A** and **B** are heated separately with a mixture of potassium dichromate(VI) and dilute sulfuric acid, the solution containing **B** turns from orange to green. The solution containing **A** remains orange. Use these results and the information at the start of the question to deduce displayed formulae for **A** and **B**.

(b) The melting point of the orange solid formed when A reacts with the solution of 2,4-dinitrophenylhydrazine should be 126 °C. Describe how the melting point of a small sample of this solid can be determined.

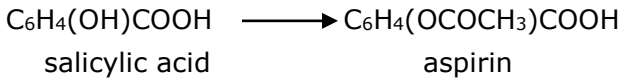
(c) In a different experiment, a student is asked to identify the two cations present in a solution, **D**. The solution is green.

(i) State the inference that the student can make about the solution.

(ii) The student adds dilute ammonia solution to a sample of **D**. He sees a precipitate, which appears to be dark brown. When he adds more ammonia solution, the amount of precipitate decreases and a deep blue solution is formed. He filters the mixture. The remaining precipitate is red-brown. Use this information to identify the two cations present in **D**. Justify your answers.

16. The preparation of aspirin

In an experiment to prepare aspirin, salicylic acid is reacted with excess ethanoic anhydride.



(a) Calculate the maximum mass of aspirin that could be obtained from 2.00 g of salicylic acid.
[molar masses / g mol^{-1}: salicylic acid = 138; aspirin = 180]

(b) In a preparation a student prepares 1.45 g of aspirin from 2.00 g of salicylic acid. Calculate the percentage yield.

(c) Give two reasons why the yield in the preparation is less than 100%.

(d) The sample of aspirin can be purified through the process of recrystallisation. The impure aspirin is dissolved in a minimum quantity of hot solvent. This solution is cooled and the pure aspirin crystals are separated by vacuum filtration and washed with a small amount of cold solvent. The aspirin is then dried.

(i) Explain why the aspirin is dissolved in the minimum quantity of hot solvent.

(ii) Explain what happens to the impurities when the hot solution cools.

(iii) Describe how you could show that the final aspirin sample was pure.

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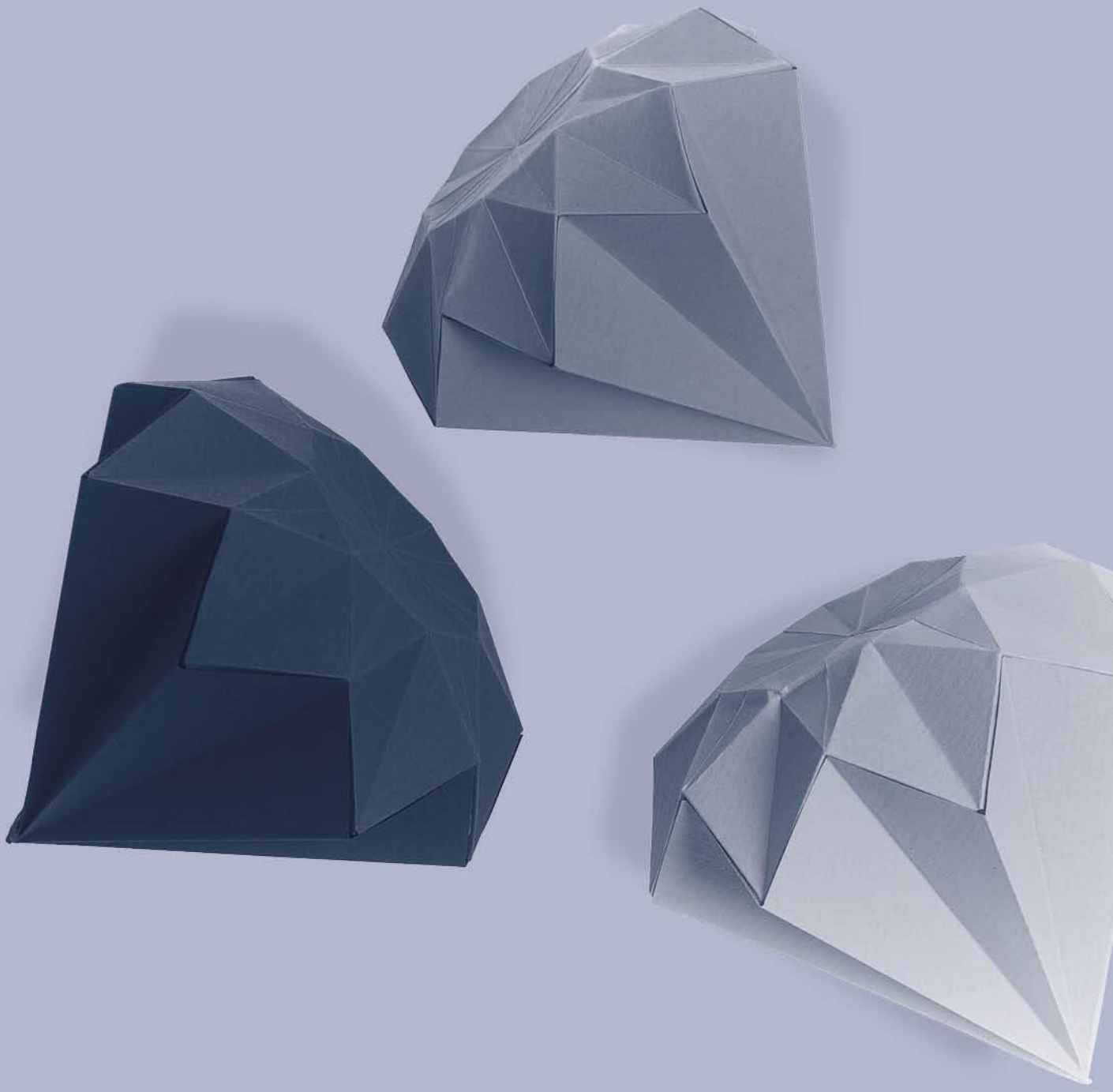

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