Cambridge Assessment
International Education

Practical Booklet 1

Gravimetric analysis

Cambridge International AS & A Level

Chemistry 9701

Version 2.0

Cambridge Pathway

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners’ practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To determine the formula of magnesium oxide by measuring the increase in mass of a piece of magnesium ribbon when heated in air.

Outcomes

Syllabus section 1.4(b) and 1.5(a) and experimental skills 2, 3 and 4.

The further work includes the possibility of supporting experimental skill 1.

Skills included in the practical

AS Level skills		How learners develop the skills
Manipulation, measurement and observation	*Successful collection of data and observations*	use the apparatus successfully to obtain an increase in mass
	Decisions relating to measurements or observations	allow air into the crucible or loss of product as smoke
Presentation of data and observations	*Recording data and observations*	record all balance readings (to same level of precision) and calculated masses with suitable headings and units
	Display of calculation and reasoning	show working in the calculation and use suitable significant figures for the precision of their balance
	Data layout	single table for results and calculated masses
Analysis, conclusions and evaluation	*Interpretation of data or observations and identifying sources of error*	calculate the mole ratio of Mg : O
	Drawing conclusions	suggest the formula of magnesium oxide from their data
	Suggesting improvements	identify significant sources of error in the experiment, suggest a modification to increase accuracy of the results, and suggest how to extend the investigation to answer a new question

Method

Safety

- Learners must wear safety glasses for this investigation.
- The experiment can be very dangerous if magnesium turnings are used, due to the intense heat produced. Ribbon is much safer in this experiment.

- Good practical techniques when crucibles are used for gravimetric experiments should be discussed. Heat is gentle to start with, to avoid loss of some MgO as smoke. Stronger heat, using a ‘cone’ flame, is used later to ensure that the reaction is as complete as possible. Cooling should always be carried out with the crucible lid on, to ensure that the residue does not absorb moisture as it cools.

- Learners should be reminded of the hazards of hot apparatus and that the white smoke, MgO, formed is an irritant. Carrying hot crucibles to the balance is unsafe. Also, hot weighing may introduce balance reading errors owing to buoyancy effects, depending on the precision of the balance.

- It is important that as much of the magnesium as possible is oxidised to magnesium oxide, though it is unavoidable that the oxidation occurs more easily on the surface of the metal. Heating to constant mass, as carried out in this experiment, is one way of trying to increase the accuracy of the results obtained.

- Results in this experiment always show a lower uptake of oxygen than expected. This is mainly due to incomplete oxidation (learners should suggest why this is) and also because some magnesium reacts with nitrogen in the air to give magnesium nitride, Mg_3N_2.

- Learners should be taught how to calculate the empirical formula of magnesium oxide from their data. In this calculation it is easier to consider oxygen as ‘O’ atoms rather than as ‘O_2‘.

- Learners could use a range of lengths of magnesium to find out whether the percentage error increases or decreases with length. Possible relationships can be discussed before collecting data. (Longer reduces % error in mass but makes it less likely that all the Mg will have reacted on heating.)

Results

Learners should draw a table for their balance readings and masses of magnesium and oxygen reacting, which includes unambiguous headings and units shown as / g or (g) (as specified in the syllabus).

Interpretation and evaluation

- The equation $n = m/A_r$ can be introduced or revised.

- The appropriate number of significant figures can be discussed. Most balance readings would be to 2 decimal places so 3 sf is appropriate when dealing with a mass of Mg < 1 g.

- The need for a whole number ratio in a formula can be discussed. Theoretically this is 1:1 for MgO but often it is not (for reasons considered below) and in this case a sensible ratio should be deduced. An example could be if Mg : O = 1 : 0.66 = 3 :2, formula is Mg_3O_2.

- The reasons for Mg : O being 1 : <1 and how to improve this result can be brainstormed. They include (i) not all the magnesium has reacted, (ii) some of the magnesium oxide escaped as smoke.

- It is easy to suggest a modification to minimise the error due to not all the magnesium reacting: steps 4 to 6 of the method should be repeated until no further increase in mass occurs. The modification must be to the *procedure* and so suggestions involving changes in method of measurement are not appropriate. (Extra apparatus may be selected.)

- As well as reacting with oxygen, magnesium also reacts with nitrogen in the air.

$$3Mg(s) + N_2(g) \rightarrow Mg_3N_2(s)$$

This could lead to a discussion of:

(1) how you know magnesium nitride does not contain oxygen (the ending ‘ide’ shows that only the specified elements are present);
(2) how you know it is a solid (ionic bonding);
(3) what effect its formation would have on the experimentally derived formula of magnesium oxide (it would lower the ratio of oxygen in the formula);
(4) whether the magnesium may have reacted with other gases in air and whether any such reaction would be significant in determining the formula.

The presence of magnesium nitride can be detected by adding a few drops of water to the residue in the crucible while it is warm (but not hot) and testing any gas with moist red litmus paper. If any nitride is present, some ammonia gas will be produced, so the litmus will turn blue.

- If the individual results are collected in a table, learners can see whether there is a trend between the length of magnesium ribbon and error in the formula calculated.

- Learners could also plan an experiment to determine the moles of water of crystallisation in one mole of a hydrated salt. ($MgSO_4{\cdot}xH_2O$ or $BaCl_2{\cdot}xH_2O$ **[T]** or $CuSO_4{\cdot}xH_2O$ **[H] [N]** could be used.)

  They should be encouraged to think about:
  (1) the quantity of hydrated solid used (small amount in crucible – large % errors in mass; large amount in crucible – loss of solid by spitting on initial heating as heated without lid);
  (2) how to minimise spitting of solid (heat gently at first);
  (3) how to make sure all the water is driven off (heat to constant mass);
  (4) how to make sure no moisture from the air is absorbed on cooling (cool with lid on, cool in a desiccator, or cool in a closed container with a drying agent such as calcium chloride).

  Care must be taken if hydrated copper(II) sulfate is used, as the anhydrous salt will start to decompose on strong heating to form copper(II) oxide **[H] [N]** and sulfur trioxide **[C]**. However, if water is added to the cold anhydrous copper(II) sulfate, the resulting exothermic reaction and colour change shows that the initial reaction is reversible.

Typical results for the experiment to determine the formula of magnesium oxide:

Mass crucible + lid / g = 22.46
Mass crucible + lid + magnesium / g = 22.66
Final mass crucible + lid + magnesium oxide / g = 22.78

Calculation

Moles of Mg = 0.20 / 24.3 = 8.23×10^{-3} mol
Moles of O = 0.12 / 16.0 = 7.50×10^{-3} mol

Mg : O = 1.1 : 1.0
Formula = MgO

length of Mg ribbon / cm	mole ratio Mg : O
13.5	1.10 : 1.00
etc.	

Information for technicians

Each learner will require:

(a) Eye protection

(b) 1 × porcelain crucible, approximately 15 cm^3, with lid

(c) 1 × pipe-clay triangle

(d) 1 × tongs (suitable to remove crucible lid)

(e) 1 × Bunsen burner (spirit burners are not suitable)

(f) 1 × tripod

(g) 1 × heatproof mat

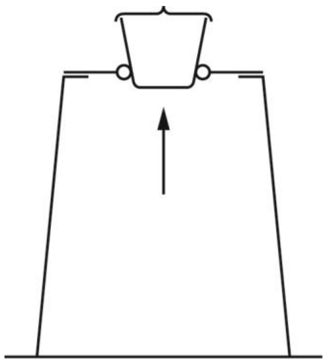
[F] (h) 10–30 cm length of magnesium ribbon (cleaned with sandpaper to remove any surface corrosion)

(i) 1 x crucible tongs – optional

(j) Access to a balance reading to 2 dp.
(If a balance reading to 1 dp is used, the errors in mass will be significantly greater for this procedure.)

(k) Ruler and scissors

Diagram of apparatus



Hazard symbols

GHS02 (flammable **F**)	GHS03 (oxidising **O**)
GHS05 (corrosive **C**)	GHS06 (acutely toxic **T**)
GHS07 (moderate hazard **MH**)	GHS08 (health hazard **HH**)
GHS09 (hazardous to the aquatic environment **N**)	

Worksheet

Aim

To determine the formula of magnesium oxide by measuring the increase in mass of a piece of magnesium ribbon when heated in air.

Method

Safety

- Wear eye protection.
- The tripod, pipe-clay triangle and gauze remain hot for a considerable time. Use tongs for the hot crucible and take care that all apparatus is cool before you touch it.
- Do not inhale the white smoke that can be given off when magnesium burns.

(1) Weigh a crucible with its lid. Record your balance reading in a table of results.

(2) Curl the length of magnesium ribbon loosely so that it fits into the crucible and weigh the crucible + lid + magnesium. Record this mass reading in the table.

(3) Set up the apparatus for heating the crucible + lid and contents.

(4) Heat the crucible + lid and contents, gently at first and then more strongly until eventually you are heating it very strongly with the air hole of the Bunsen burner fully open.

(5) Periodically use tongs to lift the lid to allow air into the crucible. You will see the magnesium burning, with a white flame or glowing red, as it reacts to give magnesium oxide. Stop the oxide escaping as a white smoke by putting the lid back on the crucible.

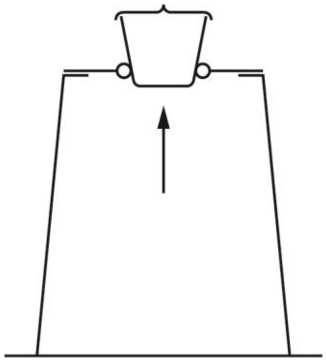
(6) When the magnesium no longer burns when the lid is lifted, allow the crucible to cool. (You may wish to lift the crucible off the pipe-clay triangle with crucible tongs and place it on the heat proof mat to cool.)

(7) When the crucible is cool weigh crucible + lid + magnesium oxide. Record your balance reading in the table.

(8) If there is time, repeat steps 4 to 7 until there is no further increase in the mass of residue. This is called ‘heating to constant mass’.

(9) Record the new balance reading and calculate the mass of magnesium used and the mass of oxygen that reacted in the table.

Diagram of apparatus



Worksheet, *continued*

Results

Record **all** your observations.

Draw a table for your balance readings and masses of magnesium and oxygen reacting, which includes unambiguous headings and units shown as / g or (g) (as specified in the syllabus).

Interpretation and evaluation

Calculation

Use the Periodic Table for any data that you may require.

(1) Calculate the number of moles of magnesium used.
(2) Calculate the number of moles of oxygen atoms that reacted with the magnesium.
(3) Use your answers to 1 and 2 to calculate the formula of magnesium oxide.

Points to consider

(1) To how many significant figures should you calculate the number of moles of each reactant?
(2) Explain how you used the ratio of the number of moles of Mg and O to deduce the formula of magnesium oxide.
(3) Explain the advantage of re-heating the residue to constant mass. (Step 8)
(4) The ratio of Mg : O obtained in this practical nearly always has less oxygen than would be expected.
    a. Suggest **two** sources of error that could account for this fact.
    b. Suggest a modification to the experimental procedure that would lower the error due to **one** of the causes you gave in (i). (You may use additional apparatus or increase the number of steps in the method.)
(5) Magnesium can also react with nitrogen in the air, to give magnesium nitride.
    a. Write the equation for this reaction.
    b. Explain why formation of magnesium nitride (instead of magnesium oxide) reduces the expected increase in mass of the product measured in this experiment.
(6) Others have used different lengths of magnesium ribbon (different masses) from you. Is there any relationship between the length of the magnesium ribbon used and the mole ratio of Mg : O?

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Practical Booklet 2

Gas volume measurement

Cambridge International AS & A Level

Chemistry 9701

Version 2.0

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Guidance for teachers

Aim

To determine the molar gas volume, V_m, at room temperature and pressure by adding magnesium to sulfuric acid then collecting and measuring the final volume of hydrogen given off.

Outcomes

Syllabus section 1.5(a), 1.5(b)(ii), 4.1(b) and (c) as well as experimental skills 2, 3 and 4

Further work: syllabus section 8.1(b), 8.2(b) and 8.3(b)(iii), show in *italics* below.

Skills included in the practical

AS Level skills		How learners develop the skills
Manipulation, measurement and observation	*Successful collection of data and observations*	set up and use the apparatus successfully to obtain a volume of gas
Presentation of data and observations	*Recording data and observations*	record mass of Mg and volume of H_2, with headings and units: *tabulate volume of gas with time*
	Display of calculation and reasoning	show working in the calculation and use suitable significant figures that show the precision of the measuring cylinder / balance
	Data layout	*accurately plot a graph of gas volume against time using suitable scales and drawing a curve of best fit*
Analysis, conclusions and evaluation	*Interpretation of data or observations and identifying sources of error*	calculate molar gas volume, V_m, and percentage error
	Drawing conclusions	*identify the effect of changing concentration or temperature on rate of reaction*
	Suggesting improvements	identify a significant source of error in the experimental procedure and suggest a modification to increase accuracy of the results

Method

Safety

- Learners must wear safety glasses for this investigation.

- Learners should be able to set up and use the apparatus provided (see diagram) to collect and measure the volume of a gas.
- It is necessary that the acid is used in excess, so that the magnesium reacts completely. Learners should be shown how to carry out calculations to demonstrate that this is the case for the quantities used. This will involve revision of the equations needed to calculate the numbers of moles being used ($n = m/A_r$ and $n = cV$).

- Quantities must be chosen so that a suitable volume of gas is collected. The solubility of hydrogen in water is very low, so error caused by solubility of this gas is negligible.

- Measurements should be made to appropriate levels of accuracy, which means to the nearest half a scale division for graduated apparatus. Balances used to weigh the solid should read to two decimal places: a one decimal place balance introduces a very large percentage error.

- Data should be recorded in suitable tables, with appropriate headings, and units. The appropriate number of significant figures can be discussed. In this experiment, answers to 2 or 3 sf are appropriate because of the precision of the measuring instruments and the concentration of acid is only quoted to 2 sf.

- A number of reactions can be studied using this apparatus. Examples include the reaction of magnesium with other dilute acids, of lithium or calcium with water, of metal carbonates with dilute acids and the catalytic decomposition of hydrogen peroxide.

- This method can be used for **further work** when studying reaction kinetics.

- The technique could also be used at A Level as the basis of a planning exercise to determine, for example, the relative formula mass of a metal carbonate. Learners would need to consider issues such as quantities and concentrations of materials to use so that the acid was in excess and a suitable volume of gas was collected. They would also have opportunity to plan and implement improvements to the basic procedure described in this booklet.

Results

Learners should record the mass of magnesium and volume of hydrogen produced with unambiguous headings and units shown as / g or (g), / cm^3 or (cm^3), as specified in the syllabus.

Interpretation and evaluation

- $Mg(s) + 2H^+(aq) \rightarrow Mg^{2+}(aq) + H_2(g)$
  The ionic equation can be used for a discussion of ‘spectator’ ions.

- Errors in the procedure and in the apparatus used can be discussed to determine which is likely to be the most significant. Gas loss before the bung is inserted is the main error. In this procedure, this error has been reduced by using magnesium ribbon instead of magnesium turnings.

  Methods of reducing this error can be discussed. These include use of a divided flask or placing the Mg in a small tube inside the flask, before replacing the bung and mixing the reagents.

- Larger than expected volumes of gas collected may be explained due to some air displaced when the bung is put on. Also, unless the water level in the tub is raised to the level in the measuring cylinder, the volume of gas collected is not measured at atmospheric pressure.

- A planning exercise could be carried out to investigate an “unknown” metal carbonate by reaction with an acid (instead of using magnesium). Learners would aim to determine the number of moles of gas produced and then calculate the M_r of the metal carbonate. Given the formula as either $\mathbf{M}_2CO_3$ or $\mathbf{M}CO_3$, learners could then calculate the A_r of the metal, **M**, and suggest its identity from the Periodic Table.

  There is rapid effervescence of carbon dioxide when a metal carbonate is added to acid, leading to a greater loss of gas before the bung is inserted and also the solubility of CO_2 in water is greater than that of H_2. The effect these factors would have on the volume of gas collected and the percentage error in the value of V_m can be discussed. (Both factors lead to lower volume of gas collected.)

  Subsequently, ways of improving the procedure to gain more accurate results could be discussed. To overcome the error due to solubility, the gas could be collected over hot water, as it is less soluble in hot water. Use of a gas syringe for collection is an alternative. However, most syringes are of 100 cm^3

capacity so this would have the disadvantage that a smaller mass of metal carbonate would need to be used, leading to greater percentage errors in balance readings.

Specimen results		**Calculation**
Mass of magnesium / g	= 0.19	Moles of H_2SO_4 = 2.5×10^{-2} mol
Volume of hydrogen / cm^3	= 198	Moles of Mg = 7.82×10^{-3} mol = moles of H_2
		$V_m = 25.3\ dm^3$
		error = 5.4%

Further work (reaction kinetics)

Method

- Different learners/groups of learners can be given different concentrations of acid or be asked to carry out the experiment at different temperatures.

- Learners should record the volumes of hydrogen obtained at regular intervals during the experiment.

Results

Learners should plot volume of hydrogen against time using over half the available grid area. The points should be plotted accurately with a sharp pencil and the curve of best fit drawn. Any anomalous points should be indicated by ringing or labelling them.

Interpretation and evaluation

- Learners can calculate the initial rate of their reaction by calculating the gradient of the line at/close to the start of their experiment. Discussion can take place about size of the triangle drawn to calculate the initial gradient.

- If several values of concentration or temperature are used within the group of learners, the results can be shared so that the learners can plot a rate against concentration or a rate against temperature graph. Discussion can then take place to decide whether the results show proportionality (straight line), direct proportionality (straight line through the origin) or some other relationship.

Additional further work (catalysis)

- Aqueous hydrogen peroxide can be decomposed to oxygen gas using (a) manganese(IV) oxide, and (b) an aqueous solution of catalase, in order to investigate the effects of heterogeneous and homogeneous catalysts on the rate of decomposition.

Information for technicians

Each learner will require:

(a) Eye protection

(b) 1 × 250 cm^3 plastic or glass measuring cylinder

(c) 1 × stand, clamp and boss (for holding inverted measuring cylinder)

(d) 1 × side-arm conical flask or conical flask with suitable bung and delivery tube (which may be rubber or plastic)

(e) 1 × tub suitable for acting as trough (for collecting gas over water)

(f) 1 × 25 cm^3 measuring cylinder

(g) Paper towel

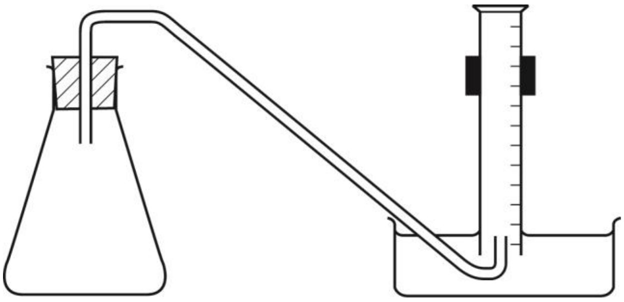
[F] (h) 0.20 ± 0.02 g of magnesium ribbon, cleaned with sandpaper to remove any surface corrosion. (This is approximately a 13.5 cm length of ribbon. Lengths are easier to supply than small masses.)

[C] (i) 30 cm^3 1.0 $mol\,dm^{-3}$ sulfuric acid

(j) Access to a balance reading to 2 dp.
(If a balance reading to 1 dp is used, the errors in mass will be significantly greater for this procedure.)

(k) Access to a tap for filling the tub and large measuring cylinder

Diagram of apparatus



Hazard symbols

GHS02 (flammable **F**)	GHS03 (oxidising **O**)
GHS05 (corrosive **C**)	GHS06 (acutely toxic **T**)
GHS07 (moderate hazard **MH**)	GHS08 (health hazard **HH**)
GHS09 (hazardous to the aquatic environment **N**)	

Worksheet

Aim

To determine the molar gas volume, V_m, at room temperature and pressure by adding magnesium to sulfuric acid then collecting and measuring the final volume of hydrogen given off.

Method

Safety

- Wear eye protection.
- Sulfuric acid is corrosive.

(1) Fill the tub with water to a depth of at least 5 cm.

(2) Fill the 250 cm^3 measuring cylinder completely with water. Hold a piece of paper towel firmly over the top, invert the measuring cylinder and place the open end into the tub.

(3) Remove the paper towel and clamp the inverted measuring cylinder so the open end is just above the base of the tub. There should be no air in the measuring cylinder.

(4) Use the 25 cm^3 measuring cylinder to transfer 25 cm^3 of 1.0 mol dm^{-3} sulfuric acid into the reaction flask.

(5) Set up the apparatus as shown in the diagram below. Check that the bung fits tightly in the neck of the reaction flask, and lower the inverted measuring cylinder (if necessary) to ensure that the end of the delivery tube will stay under it. Remove the bung from the reaction flask.

(6) Weigh the strip of magnesium ribbon and record the mass.

(7) Drop the magnesium into the acid and replace the bung immediately. Swirl the flask to mix the contents.

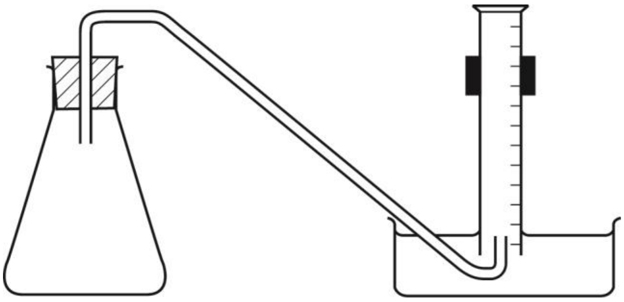
(8) Measure and record the final volume of hydrogen in the measuring cylinder, when the reaction is complete.

(9) Repeat the whole experiment twice.

Extension

(10) Record the temperature of the lab and the atmospheric pressure.

Diagram of apparatus



Worksheet, *continued*

Results

Record **all** your observations.

Record the mass of magnesium and volume of hydrogen produced with unambiguous headings and units shown as / g or (g), / cm^3 or (cm^3), as specified in the syllabus.

Interpretation and evaluation

Calculation

(1) Calculate the number of moles of sulfuric acid added to the reaction flask.
(2) Calculate the number of moles of magnesium weighed out.
(3) Write the equation for the reaction between magnesium and sulfuric acid. Include state symbols. From your answers to 1 and 2, deduce which reagent is in excess.
(4) Deduce the number of moles of hydrogen produced in the reaction.
(5) Use your answer to (4) and your results to calculate the volume occupied by one mole of hydrogen, V_m, under room conditions.
(6) The molar gas volume, V_m, under room conditions, according to the syllabus, is 24 $dm^3 mol^{-1}$. Calculate the percentage error in your value of V_m.

Extension

(7) Use your answer to (5) and the room temperature and pressure to calculate the molar gas volume of hydrogen under standard conditions of 273 K and 101 kPa.

Points to consider

(1) What is the main source of error in this experiment? How could the method be improved to reduce this error?
(2) A metal carbonate could have been used instead of expensive magnesium ribbon. This would lead to a value for the molar volume, V_m, of carbon dioxide. (This value should be the same as that for hydrogen.) Suggest two disadvantages of using a metal carbonate, rather than magnesium ribbon, to determine the molar volume, V_m, of a gas. For one of your disadvantages, suggest a change in the procedure that would improve the accuracy of the experiment.

Further work (reaction kinetics)

Method B

Additional equipment

- Stop clock

Carry out steps (1) to (6) as described in **Method A**.

(7) Drop the magnesium into the acid, replace the bung immediately and start the stop clock as soon as possible. Swirl the flask to mix the contents.

(8) Record the volume of gas in the measuring cylinder every half-minute until the reaction is complete.

Plot a graph of gas volume against time.

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Cambridge Assessment
International Education

Practical Booklet 3
Reacting masses and volumes, a titration exercise

Cambridge International AS & A Level
Chemistry 9701

Version 2.0

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- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To determine the stoichiometric equation for the reaction between amidosulfonic (sulfamic) acid, NH_2SO_3H, and sodium hydroxide, NaOH, by a titration method.

Outcomes

Syllabus section 1.5 (a), (b)(iii) and (c) as well as experimental skills 2 and 3

Further work: syllabus section 6.1

Skills included in the practical

AS Level skills		How learners develop the skills
Manipulation, measurement and observation	*Successful collection of data and observations*	set up and use the apparatus to the level of precision indicated
	Quality of measurements or observations	obtain results that are close to those of an experienced chemist
	Decisions relating to measurements or observations	decide on the end point colour of the indicator decide how many titres are needed
Presentation of data and observations	*Recording data and observations*	record the burette readings with appropriate headings and units
	Display of calculation and reasoning	show the level of precision of their burette readings show working in the calculation and use significant figures appropriate to the precision of measurements
	Data layout	results clearly tabulated
Analysis, conclusions and evaluation	*Interpretation of data or observations and identifying sources of error*	calculate numbers of moles from titration data calculate maximum percentage errors for burettes and pipettes
	Drawing conclusions	write a correct equation for the reaction decide which piece of apparatus gives the greatest maximum percentage error

Method

Safety

- Learners must wear safety glasses for this investigation.

- Learners should be shown how to make up a standard solution by weighing a solid and using a volumetric flask. It is essential that they have opportunity to practise this technique until they carry it out accurately. Standard solutions for use in a titration are sometimes made up by diluting a more concentrated solution, using a pipette and volumetric flask.

- Learners should be shown how to use pipettes (with fillers) and burettes with precision and accuracy. They should know how to run out a pipette in the proper manner. They should also understand and be able to carry out the different approaches needed when a burette is used for a rough titration and when it is used in an accurate titration. These techniques should also be practised. Two accurate titres within 0.1 cm^3 should always be obtained in any experiment.

- The accuracy of the titration technique is due to the precision of the apparatus and to the use of dilute solutions. This means that the one drop of reactant (approximately 0.05 cm^3) needed to obtain an end point contains a very small fraction of a mole so is very sensitive.

- Leaners should appreciate that the amount of indicator used should be reasonably small, but sufficient to allow the colour change to be clearly identifiable. Many end-point colour changes are subtle, so it is important the learners have used a range of indicators in their titration work.

- The advantage of using solids such as amidosulfonic acid or potassium hydrogen phthalate is that they are primary standards which are chemically stable and can be used to find the concentration of the other reagent accurately. However, more common solids are satisfactory for elementary work: anhydrous sodium carbonate can be used as the alkali when an acid is being investigated.

- This method can be used for **further work** when studying redox reactions. Learners should carry out titrations with potassium manganate(VII) to investigate reducing agents, such as iron(II) sulfate or hydrogen peroxide. Titrations in which iodine is produced, and then titrated with sodium thiosulfate, can be used to investigate oxidising agents.

- Types of investigation possible include:
  (a) determination of the stoichiometric equation for a reaction;
  (b) investigation of the change in oxidation number of one of the reactants;
  (c) determination the concentration of one of the reactants;
  (d) determination of the percentage purity of a reactant.

Results

Learners should tabulate the initial and final burette readings and titres (volume used) for the rough and as many accurate titres as deemed necessary with unambiguous headings and units shown as / cm^3 or (cm^3) (as specified in the syllabus). Burette readings for the accurate titrations should always be recorded to the nearest 0.05 cm^3 and titres are considered to be concordant/consistent if they are within 0.10 cm^3.

Interpretation and evaluation

- You can discuss which titres to use in calculating the volume of alkali to be used in the calculations and the number of decimal places to use in the answer. (Selected titres should have a spread of ≤ 0.20 cm^3; answers should be arithmetically correct to 2 dp.)

- The equations $n = m/A_r$ and $n = cV$ can be introduced or revised.

- The appropriate number of significant figures can be discussed. For the number of moles, answers to 3 or 4 sf are appropriate given the precision of the measuring instruments and that the concentration of the alkali is shown to 3 sf.

Extension

- $NaOH + NH_2SO_3H \rightarrow NH_2SO_3Na + H_2O$
  Using the full equation as a starting point, discussion can take place about what constitutes a neutralisation reaction and which are 'spectator' ions.
  $H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$

- The choice of indicator and their colour changes at the end point can be discussed.
  Thymolphthalein is suitable if a strong alkali, such as NaOH, is used in the titration.

Bromophenol blue or methyl orange are suitable if a strong acid, such as HC*l*, is used in the titration

- Errors in measurements made with pipettes, burettes and balances can be discussed such as the effect of the number of readings for one measurement, what a maximum and minimum error would be in each case. (The max % error of the balance will depend on the precision of the balance used.)

Specimen results

Mass of amidosulfonic acid /g = 2.42
Mean titre /cm^3 = 24.83

Calculation

Moles of NH_2SO_3H in each titration = (2.42/97.1) ÷ 10 = 2.49×10^{-3} mol
Moles of NaOH = 2.48×10^{-3} mol
Reacting ratio of moles is 1:1
Max % error of pipette = 0.24%

Max % error of burette = 0.40%

Further work

- Other acid-base titrations can be carried out, using different indicators.

- Redox titrations, using potassium manganate(VII) or sodium thiosulfate, can be used to:
  (a) determine the stoichiometric equation for a reaction;
  (b) investigate the change in oxidation number of one of the reactants;
  (c) determine the concentration of one of the reactants.

Information for technicians

Each learner will require:

(a) Eye protection
(b) 1 × 50 cm^3 burette
(c) 1 × burette stand and clamp
(d) 1 × filter funnel (for filling burette)
(e) 1 × 25 cm^3 pipette
(f) 1 × pipette filler
(g) 1 × 100 cm^3 beaker
(h) 1 × 250 cm^3 volumetric (graduated) flask
(i) 1 × filter funnel (for transferring solution)
(j) 2 × 150 cm^3 or 250 cm^3 conical flask
(k) 1 × white tile
(l) 1 × glass rod
(m) 1 × spatula
(n) paper towel
(o) Access to a balance reading to **at least** 1 dp.
[HH] (p) approximately 2.5 g amidosulfonic acid (supplied in a stoppered container)
[MH] (q) 140 cm^3 0.100 mol dm^{-3} sodium hydroxide
(r) 300 cm^3 distilled water

Hazard symbols

GHS02 (flammable **F**)	GHS03 (oxidising **O**)
GHS05 (corrosive **C**)	GHS06 (acutely toxic **T**)
GHS07 (moderate hazard **MH**)	GHS08 (health hazard **HH**)
GHS09 (hazardous to the aquatic environment **N**)	

Worksheet

Aim

To determine the stoichiometric equation for the reaction between amidosulfonic (sulfamic) acid, NH_2SO_3H, and sodium hydroxide, NaOH, by a titration method.

Method

Safety

- Wear eye protection.
- Amidosulfonic acid (solid) is a health hazard.
- Sodium hydroxide 0.100 mol dm^{-3} is a moderate hazard.

(1) Weigh a small beaker. Record the balance reading.

(2) Add between 2.40 g and 2.45 g of amidosulfonic acid and re-weigh. Record the new reading.

(3) Add approximately 40 cm^3 of distilled water to the beaker and stir to dissolve most of the acid.

(4) Transfer the **solution** to a 250 cm^3 volumetric flask. Do not transfer any remaining solid.

(5) Add approximately 25 cm^3 of distilled water to the beaker and stir to dissolve any remaining acid. Transfer this solution to the volumetric flask and repeat until all the acid has dissolved.

(6) Add distilled water to the beaker to wash out any remaining acid solution and transfer the washings to the volumetric flask.

(7) Make the solution up to the mark. Stopper the flask and shake it to mix the solution thoroughly.

Titration

(8) Clamp the burette carefully so that it is held vertically. Wash the burette with a little aqueous sodium hydroxide and discard the washings. Then fill the burette (through the funnel inserted at the top). Make sure that the region under the tap is full and the alkali level is on the scale. Remove the funnel.

(9) Take a reading at eye level of the position of the bottom of the meniscus on the scale. Burettes have scale markings every 0.1 cm^3 so are read to the nearest 0.05 cm^3. Record this initial burette reading in a suitable table of results.

(10) Use a pipette filler to introduce a small volume of solution of the acid into the 25 cm^3 pipette, wash the pipette and discard the washings. Using a pipette filler, fill the pipette until the bottom of the meniscus is on the marker line when the pipette is held vertically at eye level. Transfer the 25.0 cm^3 of your acid solution into a conical flask. Touch the bottom of the pipette against the wall of the flask or onto the surface of the solution to deliver the correct volume.

(11) Place the conical flask on the white tile under the burette.

(12) Add sufficient drops of bromophenol blue indicator to be able to see the yellow colour.
(If you are using methyl orange indicator the colour will be red at this stage.)

(13) Carry out a 'rough' titration. Determine the approximate volume of alkali needed to neutralise the acid in the conical flask. Swirl the flask between additions of alkaline solution from the burette. Add about 5 cm^3 of alkali at a time until the indicator colour starts to change colour as more alkali is added. Then add about 1 cm^3 of alkali at a time until the indicator turns blue (yellow if methyl orange is used).

Worksheet, *continued*

(14) Read the new level of the alkali in the burette. (Remember, the meniscus should be at eye level.) Record this final burette reading in your result table.

(15) Discard the contents of the conical flask, wash it with water and discard the washings.

(16) Pipette 25.0 cm^3 of acid solution into the conical flask and add indicator.

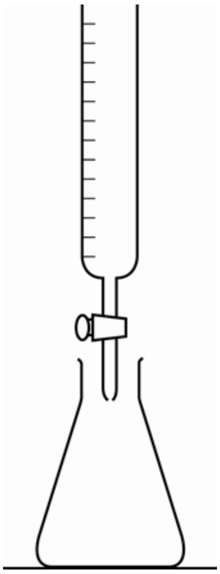
(17) If needed, top up the burette with the aqueous sodium hydroxide, take a reading of the level and record it. This is the initial burette reading

(18) Carry out an ‘accurate’ titration. Add aqueous sodium hydroxide from the burette to the aqueous amidosulfonic acid until the indicator just stays blue (or just stays yellow with methyl orange) when the solution is swirled. You should add the alkali a drop at a time, when close to the end point, until the colour of the indicator changes.

(19) Take the new reading of the alkali level and record it. This is the final burette reading.

(20) Repeat steps 15 – 19 until you have 2 concordant ‘accurate’ titres, that is, titres no more than 0.10 cm^3 apart.

Diagram of apparatus



Results

Record **all** your observations.

Tabulate the initial and final burette readings and titres (volume used) for the rough titration and as many accurate titres as deemed necessary with unambiguous headings and units shown as / cm^3 or (cm^3) (as specified in the syllabus).
Burette readings for the accurate titrations should always be recorded to the nearest 0.05 cm^3 and titres are considered to be concordant/consistent if they are within 0.10 cm^3.

Worksheet, *continued*

Interpretation and evaluation

Calculation

Use the Periodic Table for any data required.

(1) Calculate the number of moles of amidosulfonic acid, NH_2SO_3H, weighed out.
(2) Use your answer to (1) to calculate the number of moles of amidosulfonic acid present in the 25.0 cm^3 pipetted into your conical flask.
(3) Calculate the mean volume of sodium hydroxide that will be used in your calculations.
(4) Use your answer to (3) to calculate the number of moles of sodium hydroxide, 0.100 $mol\,dm^{-3}$ NaOH, required to neutralise the acid in the conical flask.
(5) Use your answers to (2) and (4) to calculate the mole ratio of alkali : acid.
(6) Write a balanced equation for the reaction between sodium hydroxide and amidosulfonic acid.

Extension

(7) Write an ionic equation, including state symbols, for the reaction.

Points to consider

(1) A pipette is marked as being accurate to ± 0.06 cm^3.
What is the maximum percentage error in the volume of amidosulfonic acid you pipetted into the conical flask?
(2) A single burette reading is accurate to ± 0.05 cm^3. What would be the maximum percentage error if your titre was 24.85 cm^3?
(3) The end point in a titration is when the indicator changes to the desired colour on adding one drop of reagent. One drop has a volume of approximately 0.05 cm^3. Calculate the number of moles of 0.100 $mol\,dm^{-3}$ of sodium hydroxide contained in this volume.
(4) The error in the balance reading is ± half the smallest division. For a 2 dp balance the error would be ± 0.005 g. Calculate the maximum percentage error for the mass of amidosulfonic acid you weighed out.
(5) Which piece of apparatus, the burette, pipette or the balance, caused the greatest percentage error in your experiment?

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Practical Booklet 4

Enthalpy changes

Cambridge International AS & A Level

Chemistry 9701

Version 2.0

Cambridge Pathway

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To determine the enthalpy change for a metal displacement reaction by adding a known mass of zinc powder to excess of aqueous copper (II) sulfate solution and recording the rise in temperature.

Outcomes

Syllabus section 5.1(a), 5.1(b)(i), 1.5(b)(i) and (iii), 5.1(c), 5.2(a) and 6.1(a)(ii) as well as experimental skills 2, 3 and 4

Further work: syllabus section 7.2(b) and experimental skill 1

Skills included in the practical

AS Level skills		How learners develop the skills
Manipulation, measurement and observation	*Successful collection of data and observations*	set up and use the apparatus to the level of precision indicated
	Quality of measurements or observations	obtain results that are close to those of an experienced chemist
Presentation of data and observations	*Recording data and observations*	record the times and temperatures with appropriate headings and units
	Display of calculation and reasoning	show the level of precision of their thermometer readings show working in the calculation and use significant figures appropriate to the precision of measurements
	Data layout	results clearly tabulated
Analysis, conclusions and evaluation	*Interpretation of data or observations and identifying sources of error*	calculate numbers of moles of reactants and the enthalpy change of the reaction
	Drawing conclusions	determine whether an exo- or endothermic reaction has taken place
	Suggesting improvements	suggest ways to improve the accuracy of the procedure suggest ways in which to extend the investigation to answer a new question

Method

Safety

- Learners must wear safety glasses for this investigation.

- This technique can be used to determine enthalpies of reaction for a variety of reactions involving solutions. The reaction must be fast enough to be complete within a few minutes (otherwise there is too much heat loss) and it is normal that one of the reactants is used in excess.

- Temperatures are always measured to half of the smallest graduation on the thermometer. In examinations, thermometers reading to 1 ºC are specified, so learners should be trained to read them to the nearest 0.5 ºC. Solutions must always be stirred thoroughly before any temperature is recorded and the bulb of the thermometer must be totally immersed in the solution.

- Learners should be taught how to carry out calculations of ΔH from the temperatures they measure. They should understand why it important to identify which reagent is in excess. They should remember that, for exothermic reactions, ΔH is negative and that ΔH is positive when the reaction proceeds with a decrease in temperature.

- Foamed plastic (polystyrene) cups provide a reasonable level of insulation against heat loss. Plotting a cooling curve provides a correction for heat that is inevitably lost while a reaction is taking place. Method B (see Worksheet) therefore provides more accurate values of ΔH.

- Learners should become familiar with using the apparatus provided so they can perform experiments accurately.

- **Extension involving Hess' Law**: learners could react magnesium turnings **[F]** with excess $CuSO_4$(aq), and determine the enthalpy change for this reaction. Using the ΔH value for the reaction of zinc with $CuSO_4$(aq), Hess' Law can be used to determine the ΔH value for the reaction of magnesium turnings with zinc sulfate solution.

- These techniques can be used for **further work** when studying strong and weak acids and alkalis. Learners can plan a suitable method and concentrations of reactants to use, in order to investigate whether the overall enthalpy change of reaction differs for strong and weak acids and alkalis when mixed in different combinations in neutralisation reactions.

Results

Learners should tabulate results showing a consistent number of decimal places in the balance readings and all thermometer readings recorded to either #.0 or #.5 °C. Headings should be unambiguous with units shown as / g or (g) and / °C or (°C), as specified in the syllabus.

Interpretation and evaluation

- The equations $n = m/A_r$ and $n = cV$ can be introduced or revised.

- The appropriate number of significant figures can be discussed. Answers to 2 – 4 sf are appropriate given the precision of the measuring instruments. The accuracy of the procedure is not as great as for a titration exercise, and in some experiments ΔT may be < 10.0 °C, so answers to 2 sf are acceptable, especially if a 1 dp balance is used.

- Errors in measurements made with balances, measuring cylinders and thermometers can be discussed. The difference between needing one reading (as in a measuring cylinder) and two readings (for the mass of zinc or the change in temperature) can be linked to maximum percentage errors. Heat energy losses can be discussed.

- Ways of reducing the inaccuracies can be discussed.

  (i) A lid with a hole for the thermometer can be used to reduce heat energy losses by convection. (The plastic cup should be of foamed plastic so would be a very poor conductor of heat. The supporting beaker further reduces heat losses by conduction.)

  (ii) A thermometer calibrated to 0.2 °C can be used so that the maximum error is ± 0.1 °C (max % error for ΔT = 37 ºC is 2.7% when using a thermometer accurate to ± 0.5 °C).

  (iii) The volume of $CuSO_4$(aq) can be measured with a burette (more precisely calibrated than a measuring cylinder) so that the volume of solution heated would be more accurate.

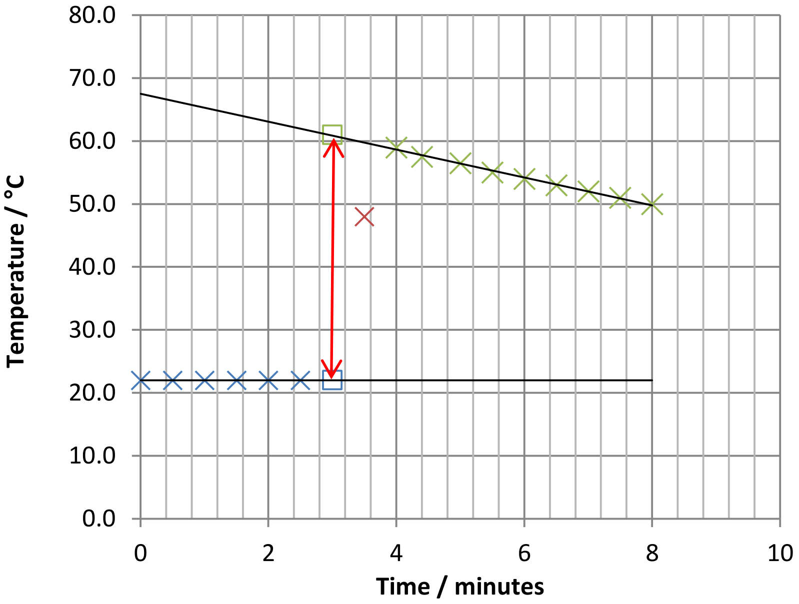
(iv) The amounts of reactants can be scaled up to decrease errors in volume and mass measurements. In any proposed scaling the $CuSO_4$ must remain in excess. (The use of a balance accurate to more dp is not significant unless the balance used in the experiment was to 1 dp.)

Typical results
Initial mass of weighing boat / g = 2.54
Mass of weighing boat and zinc / g = 4.57
Final mass of weighing boat / g = 2.55
Mass of zinc added / g = 2.02
Volume of aqueous $CuSO_4$ used = 40 cm^3
Initial temperature reading / °C = 22.0
Final temperature reading / °C = 59.0
Temperature change / °C = 37.0

Calculation (unrounded values may be carried in the calculator)
Heat energy given out $= mc\Delta T = 40 \times 4.18 \times 37 = 6186 = 6.19 \times 10^3$ J

Moles of Zn = 2.02 / 65.4 = 3.09×10^{-2} mol
$\Delta H = -6186 / (3.09 \times 10^{-2} \times 10^3) = -200$ kJ mol

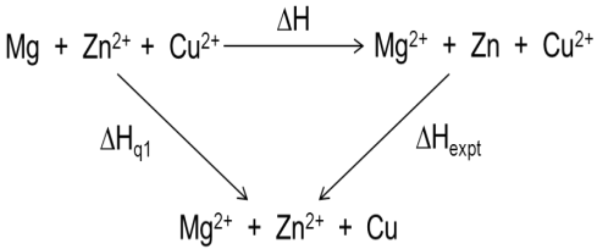
Graph of results for **Method B**



Temperature rise at 3 minutes = 61.0 – 22.0 = 39.0 °C; $\Delta H = -211$ kJ mol^{-1}

Questions

Discussion of which atoms / ions are taking part in the reactions leads to a suitable Hess' Law diagram.


$$\begin{tikzcd} Mg + Zn^{2+} + Cu^{2+} \arrow[rr, "\Delta H"] \arrow[dr, "\Delta H_{q1}"'] & & Mg^{2+} + Zn + Cu^{2+} \arrow[dl, "\Delta H_{expt}"] \\ & Mg^{2+} + Zn^{2+} + Cu & \end{tikzcd}$$

(Using the results and data given in the learner section,
ΔH = –320 – (–200) = –120 kJ mol^{-1};
theoretical value = – (–153.89) + (–466.85) = –312.96 = –313 kJ mol^{-1}.)

Extension

If the extension practical exercise is carried out then possible reasons for the larger difference in ΔH for the experiment with magnesium turnings with copper sulfate solution from the theoretical value using $\Delta H_f^{\ominus}$ compared with that for zinc powder can be discussed. Learners may note some bubbling when magnesium is added to copper sulfate solution showing a competing reaction (releasing hydrogen) is occurring.

Further work

The students can plan an experiment to determine the enthalpy change of neutralisation reactions. This can be carried out in groups with each using a different combination of strong and weak acids and alkalis. This supports learning objective 5.1(b)(ii) and links up with learning objective 7.2(b) in addition to those listed above. Discussion can help learners decide what volumes and concentrations would give them a sensible temperature rise.

Information for technicians

Each learner will require:

(a) Eye protection
(b) 1 × 250 cm^3 beaker
(c) 1 × foamed plastic (polystyrene) cup approximately 150 cm^3
(d) 1 × thermometer (–10 ºC to +110 ºC at 1 ºC)
(e) 1 × weighing boat or 100 cm^3 beaker
(f) 1 × 50 cm^3 measuring cylinder
(g) access to a balance reading to **at least** 1 dp
[MH] [N] (h) 50.0 cm^3 1.0 mol dm^{-3} copper(II) sulfate
[F] [N] (i) 2.0 ± 0.1 g zinc powder (supplied in a stoppered container)

Extension:
Apparatus and 1.0 mol dm^{-3} copper(II) sulfate as above

[F] (j) 0.8 ± 0.1 g magnesium turnings (supplied in a stoppered container)

Hazard symbols

 GHS02 (flammable **F**)	 GHS03 (oxidising **O**)
 GHS05 (corrosive **C**)	 GHS06 (acutely toxic **T**)
 GHS07 (moderate hazard **MH**)	 GHS08 (health hazard **HH**)
 GHS09 (hazardous to the aquatic environment **N**)	

Worksheet

Aim

To determine the enthalpy change for a metal displacement reaction by adding a known mass of zinc powder to excess aqueous copper(II) sulfate solution and recording the rise in temperature.

Method A

Safety
• Wear eye protection. • 1.0 mol dm^{-3} copper(II) sulfate **[MH] [N]** • zinc powder **[F] [N]**

(1) Weigh the container with zinc.

(2) Support the plastic cup in a 250 cm^3 beaker.

(3) Using a 50 cm^3 measuring cylinder, pour 40 cm^3 of the copper(II) sulfate solution into the plastic cup.

(4) Measure the temperature of the copper(II) sulfate solution. Record your thermometer reading.

(5) Add all the zinc from the container to the copper(II) sulfate solution in the plastic cup.

(6) Use the thermometer to stir the mixture gently while the reaction takes place. (It is usually poor practice to stir a mixture with a thermometer. However, it is acceptable here because it is less likely that the thermometer will break with a plastic cup being used.)

(7) Measure and record the highest temperature reached.

(8) Reweigh the empty container (with any traces of zinc remaining). Record this mass. Calculate and record the mass of zinc used.

(9) Record the change in temperature and the mass of zinc powder added.

(10) If there is time, repeat the whole experiment.

Method B

Carry out steps (1)–(3) as in **Method A**

Draw a table for your results. You will be taking thermometer readings almost every ½ minute for 8 minutes.

Measure the temperature of the copper(II) sulfate solution. Record your thermometer reading. This is the temperature at time = 0.

Start the stop clock and take the temperature of the copper(II) sulfate solution every ½ minute for 2½ minutes. Record all your thermometer readings.

At 3 minutes add all the zinc to the copper(II) sulfate solution in the plastic cup.

Use the thermometer to stir the mixture gently while the reaction takes place.

Measure and record the temperature of the mixture every ½ minute from 3½ minutes to 8 minutes.

Reweigh the empty container (with any traces of zinc remaining). Record this mass. Calculate and record the mass of zinc used.

Plot a graph of temperature / °C (*y*-axis) against time / minutes (*x*-axis).

Worksheet, *continued*

(11) Read the new level of the alkali in the burette. (Remember, the meniscus should be at eye level.) Record this final burette reading in your result table.

(12) Discard the contents of the conical flask, wash it with water and discard the washings.

(13) Pipette 25.0 cm^3 of acid solution into the conical flask and add indicator.

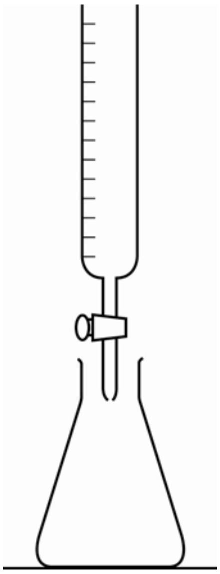
(14) If needed, top up the burette with the aqueous sodium hydroxide, take a reading of the level and record it. This is the initial burette reading

(15) Carry out an ‘accurate’ titration. Add aqueous sodium hydroxide from the burette to the aqueous amidosulfonic acid until the indicator just stays blue (or just stays yellow with methyl orange) when the solution is swirled. You should add the alkali a drop at a time, when close to the end point, until the colour of the indicator changes.

(16) Take the new reading of the alkali level and record it. This is the final burette reading.

(17) Repeat steps 15 – 19 until you have 2 concordant ‘accurate’ titres, that is, titres no more than 0.10 cm^3 apart.

Diagram of apparatus



Results

Record **all** your observations.

Tabulate the initial and final burette readings and titres (volume used) for the rough titration and as many accurate titres as deemed necessary with unambiguous headings and units shown as / cm^3 or (cm^3) (as specified in the syllabus).
Burette readings for the accurate titrations should always be recorded to the nearest 0.05 cm^3 and titres are considered to be concordant/consistent if they are within 0.10 cm^3.

Worksheet, *continued*

Interpretation and evaluation

Calculation

Use the Periodic Table for any data required.

(1) Calculate the number of moles of amidosulfonic acid, NH_2SO_3H, weighed out.
(2) Use your answer to (1) to calculate the number of moles of amidosulfonic acid present in the 25.0 cm^3 pipetted into your conical flask.
(3) Calculate the mean volume of sodium hydroxide that will be used in your calculations.
(4) Use your answer to (3) to calculate the number of moles of sodium hydroxide, 0.100 $mol\,dm^{-3}$ NaOH, required to neutralise the acid in the conical flask.
(5) Use your answers to (2) and (4) to calculate the mole ratio of alkali : acid.
(6) Write a balanced equation for the reaction between sodium hydroxide and amidosulfonic acid.

Extension

(7) Write an ionic equation, including state symbols, for the reaction.

Points to consider

(1) A pipette is marked as being accurate to ± 0.06 cm^3.
What is the maximum percentage error in the volume of amidosulfonic acid you pipetted into the conical flask?
(2) A single burette reading is accurate to ± 0.05 cm^3. What would be the maximum percentage error if your titre was 24.85 cm^3?
(3) The end point in a titration is when the indicator changes to the desired colour on adding one drop of reagent. One drop has a volume of approximately 0.05 cm^3. Calculate the number of moles of 0.100 $mol\,dm^{-3}$ of sodium hydroxide contained in this volume.
(4) The error in the balance reading is ± half the smallest division. For a 2 dp balance the error would be ± 0.005 g. Calculate the maximum percentage error for the mass of amidosulfonic acid you weighed out.
(5) Which piece of apparatus, the burette, pipette or the balance, caused the greatest percentage error in your experiment?

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Cambridge Assessment
International Education

Practical Booklet 5

Reaction kinetics

Cambridge International AS & A Level
Chemistry 9701

Version 2.0

Cambridge Pathway

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To investigate the effect of change in concentration on the rate of the reaction between potassium iodate(V) and sodium sulfite in acidic solution.

Outcomes

Syllabus section 1.5 (a), 6.1 (a)(iii), 8.1 (b) as well as experimental skills 1, 2, 3 and 4.
Further work: AL syllabus section 8.2 (d), 8.1 (d)(ii) and (f), shown in *italics* below.

Skills included in the practical

AS level skills	How learners develop the skills
MMO collection	set up and use the apparatus to the level of precision indicated
MMO quality	obtain results that are close to those of an experienced chemist
MMO decisions	decide whether any experiment should be repeated *decide how many different concentrations/temperatures to use*
PDO recording	record the volumes and times with appropriate headings and units
PDO display	show working in the calculation and use significant figures appropriate to the precision of measurements
PDO layout	results clearly tabulated graph axes correctly labelled and majority of grid used points correctly plotted and the line of best fit drawn
ACE analysis	calculate relative rates identifying errors
ACE conclusions	calculate relative rates identifying errors
ACE improvements	suggest ways in which to extend the investigation
PLAN method	*plan which variables should be controlled* *plan how many experiments are needed and what range of values would be appropriate*

Method

- **Learners must wear eye protection for this investigation**.

- Many experiments to investigate rates of reaction involve redox reactions that produce iodine, which is detected by turning starch blue. The concentrations of chemicals used in these reactions have to be chosen such that the reaction takes a suitable length of time that is accurately measureable.

- Experiments of this kind give learners good practise at following what can be quite complex step-by-step instructions as to what to do. They require good levels of organisation by the learners and accurate use of measuring instruments.

- In kinetic experiments that investigate effect of concentration on the rate of reaction, it is important to keep the total volume of solutions used constant, so that there is only one variable. This is done by “topping up” with distilled water. The concentration of the reactant being investigated is varied, by altering the volume of it used. The concentrations (or volumes) of all other reactants must be kept constant.

- Stop watches should normally be read to the nearest second in examinations. Learners should become familiar with using the apparatus so they can perform such experiments to a high level of accuracy. They need to practice the skills necessary to mix the appropriate solutions and start the stop clock as soon as this is done.

- It is always advisable to dispose of reaction mixtures containing sulfites and acid as soon as possible after the end-point of the reaction, using plenty of water. However, the solutions used in this experiment are very dilute so the formation of sulfur dioxide is minimal.

- This method can be used for **further work** when studying orders of reaction at A Level.

Results

- Discussion can take place before the learners start the practical to decide what data should be tabulated, whether some can simply be recorded, and whether to include a column/row for relative rates. Headings should be unambiguous and units shown as specified in the syllabus. The time taken should be recorded to the nearest second. The unit for relative ‘rate’ will depend on the equation used for its calculation but is likely to be / s^{-1}.

Interpretation and evaluation

- You can discuss the plotting of the graph to use for the learners results, whether to include (0,0) and how to deal with anomalous results. (Learners should indicate anomalies on the graph paper or repeat the experiment to check results.)

- You can discuss any relationship shown by the shape of the best-fit line on the graph, whether concentration and rate are proportional (straight line) or directly proportional (straight line through the origin) or there is some other relationship.

- You can discuss why the use of the volume of iodate solution instead of concentration is valid.

- The equation for the reaction is,
  $$2IO_3^- + 5SO_3^{2-} + 2H^+ \rightarrow I_2 + 5SO_4^{2-} + H_2O$$

- Learners should discuss which of the timings you have made is the least accurate. (The quickest experiment will have the greatest percentage error in the timing. It is more difficult to judge when the blue colour appears in the slowest experiment.)

- The smallest volume measured will have the greatest percentage error (5.0%) using the burette whereas the greatest percentage error with the measuring cylinder is 1.25%.

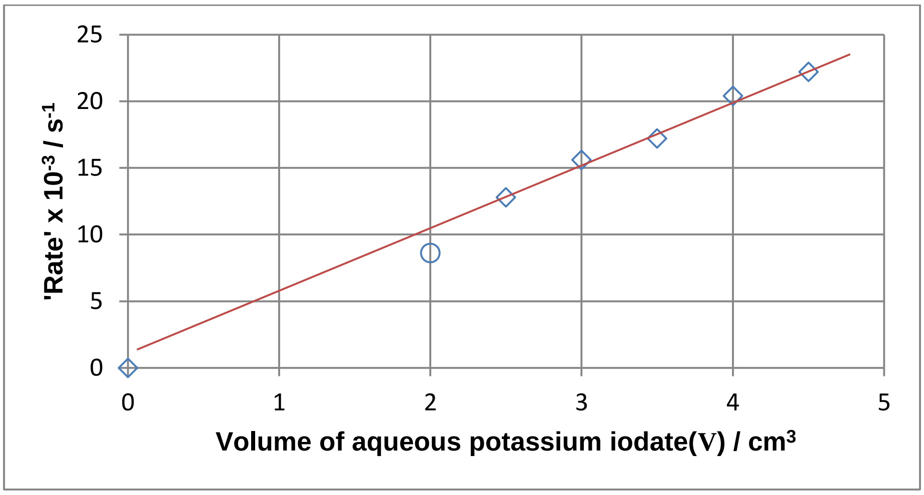
Extension

You can brainstorm ideas:

(i) how to find the temperature of each experiment (e.g. use of thermostatically controlled water baths; measuring the temperature when mixing the solutions and when the blue colour appears then calculate a mean);
(ii) deciding on a suitable range of temperatures to use (using ice as well as heating);
(iii) deciding on suitable volumes and concentrations of reactants so the rate is not too high.

Typical results

Volume IO_3^- / cm^3	Volume H_2O / cm^3	time / s	'rate' x 10^{-3} / s^{-1}
2.0	48.0	116	8.6
2.5	47.5	78	12.8
3.0	47.0	64	15.6
3.5	46.5	58	17.2
4.0	46.0	49	20.4
4.5	45.5	45	22.2



Further work

Learners can investigate the effect of changing the concentration of (i) sulfite ions, (ii) acid on the rate of reaction. **They can use the shapes of the graphs of their results to determine the order of reaction with respect to the concentrations of the ions and hence suggest a rate expression for the reaction.**

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Information for technicians

Each learner will require:

(a) Eye protection
(b) 250 cm^3 beaker
(c) 1 x foamed plastic (polystyrene) cup approximately 150 cm^3
(d) 1 x thermometer (–10 ºC to +110 ºC at 1 ºC)
(e) 1 x weighing boat or 100 cm^3 beaker
(f) 1 x 50 cm^3 measuring cylinder
(g) access to a balance reading to **at least** 1 dp
[MH] [N] (h) 50.0 cm^3 1.0 mol dm^{-3} copper(II) sulfate
[F] [N] (i) 2.0 ± 0.1 g zinc powder (supplied in a stoppered container)

Extension:
Apparatus and 1.0 mol dm^{-3} copper(II) sulfate as above
[F] (j) 0.8 ± 0.1 g magnesium turnings (supplied in a stoppered container)

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To investigate the effect of change in concentration on the rate of the reaction between potassium iodate(V) and sodium sulfite in acidic solution.

Method

Safety:

- Wear eye protection.

1. Set up 4 burettes labelled iodate, sulfite, acid and starch. Fill the burettes with the solution matching the label. Make sure that the region under each tap is full of solution.

First experiment

Beaker A (100 cm^3 capacity)

2. Run 2.00 cm^3 of aqueous potassium iodate(V) from the burette into the beaker. Use the 50 cm^3 measuring cylinder to add 48.0 cm^3 of water (use a dropping pipette to adjust the level of the meniscus).

Beaker B (250 cm^3 capacity)

3. Run 5.00 cm^3 of aqueous sodium sulfite from the burette and add 40.0 cm^3 of water from the measuring cylinder into the beaker. Add 2.50 cm^3 of dilute sulfuric acid and 2.50 cm^3 of starch indicator into the same beaker.

4. Stir the solution to mix the reactants and place beaker **B** on a white tile.

5. Tip all the solution from beaker **A** into beaker **B** as quickly as possible and simultaneously start the stop clock. Stir the contents of the beaker.

6. Stop the clock as soon as you see a blue colouration in the solution. Record the time taken for the blue coloration to appear.

7. Pour away the mixture and rinse both beakers with water. Shake them to get rid of excess water then wipe them dry with paper towel.

Second experiment

8. Run 2.50 cm^3 of aqueous potassium iodate(V) from the burette into beaker **A**. Add 47.5 cm^3 of water.

9. Use exactly the same combination of volumes in beaker **B** as in step 3.

10. Repeat steps 4 – 7.

Worksheet, continued

Third to fifth experiments

11. For these experiments, increase the volume of potassium iodate(V) by 0.50 cm^3 and reduce the volume of water by 0.5 cm^3 each time but keep the volumes of solutions and water in beaker **B** the same.

Results

Record **all** your observations.

When drawing a table for your results it is worth adding an extra line or column for the 'rate' of each reaction.

Before the start of the practical decide what data should be tabulated, whether some can simply be recorded, and whether to include a column/row for relative rates. Headings should be unambiguous and units shown as specified in the syllabus. The time taken should be recorded to the nearest second. The unit for relative 'rate' will depend on the equation used for its calculation but is likely to be / s^{-1}.

Interpretation and evaluation

Calculation

Use the following equation to calculate 'rate':

$$\text{'rate'} = \frac{1000}{\text{time (s)}}$$

1. Calculate the 'rate' of each of the 5 experiments.

2. Plot a graph of 'rate' against volume of aqueous potassium iodate(V).

3. The volume of potassium iodate used is directly proportional to its concentration.
   Use your graph to suggest a relationship between 'rate' and the concentration of potassium iodate.

4. The ions reacting in this experiment are IO_3^-, SO_3^{2-} and H^+. The overall reaction is to form I_2, SO_4^{2-} and H_2O.
   (i) What is the change in oxidation number of the iodine?
   (ii) What is the change in oxidation number of the sulfur?
   (iii) Use your answers to (i) and (ii) to write a balanced ionic equation for the reaction that takes place.

5. Which of your five experiments do you consider to have the greatest error in the time recorded? Explain your answer.

6. Why is it not necessary to use a burette to measure the volume of water added?

Worksheet, continued

Extension

7. How could you adapt this method to investigate the effect of temperature on the rate of reaction? Give practical details of your procedure.

Points to consider

1. Why is the volume of potassium iodate(V) directly proportional to its concentration?

2. A burette is accurate to ± 0.05 cm^3. A 50 cm^3 measuring cylinder is accurate to ± 0.5 cm^3. Why was a measuring cylinder suitable for measuring the volumes of water to be added?

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Cambridge Assessment International Education

Practical Booklet 6

The chemical properties of ethanol

Cambridge International AS & A Level Chemistry 9701

Version 2.0

Cambridge Pathway

Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

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The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To investigate the reactions of ethanol by carrying out a range of different experiments.

Outcomes

Syllabus section 17.1 (a) (c); 15.2 (a)(ii) (b); 18.1 (d); 19.1 (a) (b)(i)(ii), as well as experimental skills 2 and 3

Skills included in the practical

AS Level skills	How learners develop the skills
MMO collection	set up apparatus according to instructions and use it to obtain the expected results
PDO layout	observations clearly recorded
ACE analysis	describe and summarise the key points of a set of observations
ACE conclusions	draw conclusions from interpretations of observations make scientific explanations of the observations they have described

Method

- Learners must wear eye protection for these investigations. Gloves should also be available.

- A number of experiments are described in this booklet:
  Set 1. Test tube reactions of ethanol;
  Set 2. Dehydration of ethanol: collection of and tests on the ethane produced;
  Set 3. Oxidation of ethanol: collection of and tests on the aqueous ethanal produced;
  Set 4. Synthesis of ethanoic acid from ethanol.

- Learners should become familiar with using the type of glass-jointed apparatus often used for organic synthesis; see **Method** (**Set 4**). Centres may not have enough apparatus for all learners to carry out the same experiment at the same time, in which case a ‘circus’ of experiments should be devised, where each experiment is set up in a different part of the lab so that all learners have experience of all the types of apparatus. Considerable manual dexterity and team-work in pairs will be needed to set up some of the apparatus used.

- Learners should become confident in handling hazardous chemicals, such as sodium and concentrated sulfuric acid.

  - **Set 1.** While learners are using these hazardous materials they should be closely supervised by a teacher.

  - **Set 2.** In this experiment there is a risk of cold water being sucked back into a very hot test tube. Learners should be warned to be alert for this, and to remove the delivery tube from the beaker of water immediately if there is any sign of this happening. They should not prolong the heating, once all the ethanol has reacted.

  - **Set 3.** In this experiment, the ethanol produced is very volatile and flammable, so it is essential that iced water is used to surround the test tube in which the ethanol is collected.

  - **Set 4.** In some Centres, it may be necessary for this experiment to be demonstrated by the teacher. While the reflux stage is taking place, other aspects of the experiments can be discussed.

- All observations during tests should be clearly recorded. Learners should note any colours changes, the state of the products and the vigour of reactions taking place. Precipitates should be identified as such, when appropriate. In general, if gases are produced (such as carbon dioxide) they should be identified by their normal tests.

Interpretation and evaluation

- The main reactions of ethanol are covered in this practical work. The reasons for the slower reactions – and why heating is needed – of organic compounds compared with inorganic compounds can be discussed. This may be used to revise bond types, bond energies and reaction kinetics.

Set 1

  - Discussion can take place to suggest why the flame of burning ethanol is blue and non-smoky.
  - The reason for not testing the gas evolved directly with a lighted splint can be discussed. Learners can suggest how to collect the gas for safe testing.
  - Learners can be reminded of the reaction of metal carbonates with acid, and the significance of negative results.
  - The reason for the low solubility of triiodomethane in water can be discussed.
  - The esterification reaction can be extended to prepare other esters.

Set 2

  - The reason for using the safety flask and the reason for removing the delivery tube from the water before stopping the heating can be discussed. (Both prevent the risk of suck-back of cold water onto the hot glass boiling tube.)
  - The addition reactions of alkenes can be introduced or revised.

Set 3

  - The reason for using anti-bumping granules can be discussed. (These help form small bubbles on boiling and reduce risk of reactants ‘bumping’ over into delivery tube.)
  - The oxidation reactions of aldehydes can be introduced. (Sandell’s reagent may be used instead of Fehling’s.) The two tests can also be used with a ketone such as propanone.

Set 4

  - The reasons for refluxing the reactants can be discussed.
  - The use of sodium carbonate to test the product can be discussed. (The product could also be tested with more acidified potassium manganate(VII) or Fehling’s solutions or Tollens’ reagent to show it cannot be oxidised further.)

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Information for technicians

Set 1

Each learner will require:

	(a)	Eye protection and gloves
	(b)	Bunsen burner
	(c)	heat proof mat
	(d)	small evaporating dish
	(e)	3 x test-tube
	(f)	1 x boiling tube
	(g)	1 x teat / dropping pipette
	(h)	1 x 100 cm^3 beaker (or smaller)
	(i)	1 x white tile
	(j)	1 x test-tube holder
	(k)	1 x forceps
	(l)	1 x knife or scalpel
	(m)	filter paper
	(n)	1 x test-tube rack
[F] [C]	(o)	Sodium, stored in oil
[C]	(p)	1 cm^3 glacial ethanoic acid
[F] [MH] [HH]	(q)	5 cm^3 ethanol
[MH]	(r)	50 cm^3 0.2 mol dm^{-3} sodium carbonate

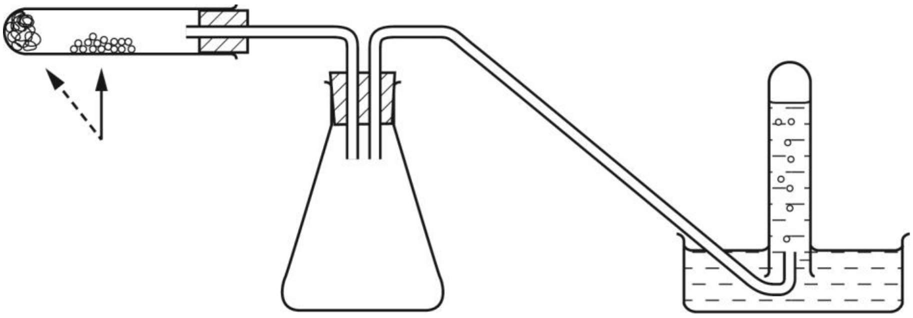
Technician's notes, *continued*

Set 2

Each learner will require:

(a) Eye protection and gloves
(b) Bunsen burner
(c) heat proof mat
(d) 1 x boiling tube
(e) 4 x test-tube with cork or rubber bung
(f) 1 x 250 cm^3 beaker (or larger beaker or small tub)
(g) 2 x teat / dropping pipette
(h) 1 x stand and clamp
(i) 1 x conical flask (to act as safety trap) - *optional as it makes successful collection of ethene gas far more difficult.*
(j) bungs and delivery tubes (as shown in the diagram)
(the delivery tube from the conical flask to the beaker may be glass, plastic or rubber)
(k) 1 x forceps
(l) broken pumice (or unglazed pottery) or aluminium oxide
[F] [MH] [HH] (m) 2 cm^3 ethanol
[MH] (n) 1 cm^3 aqueous bromine (0.005 - 0.01 mol dm^{-3})
[MH] (o) 1 cm^3 acidified aqueous potassium manganate(VII)
(p) mineral or ceramic wool

Equipment set-up



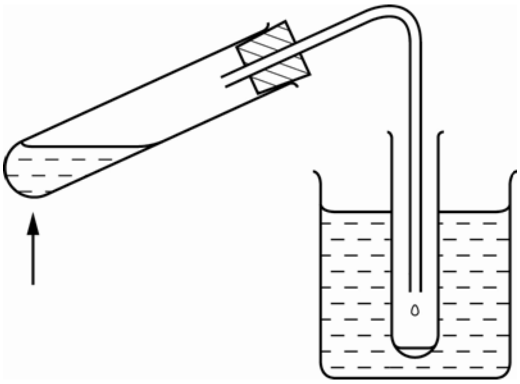
Technician's notes, *continued*

Set 3

Each learner will require:

(a) Eye protection and gloves
(b) 1 x Bunsen burner
(c) 1 x heat proof mat
(d) 1 x 25 cm^3 measuring cylinder
(e) access to balance weighing to at least 1 dp
(f) 1 x weighing boat or small beaker
(g) 1 x boiling tube
(h) 1 x stand and clamp
(i) 1 x 250 cm^3 beaker
(j) bung and delivery tube (as shown in the diagram)
(k) 3 x test-tube
[F] [MH] [N] (l) 5 cm^3 ethanol
(m) access to Fehling's A and Fehling's B solution (**or** Sandell's reagent **[MH]**)
[MH] (n) 1 cm^3 aqueous silver nitrate
[MH] [N] (o) 1 cm^3 aqueous sodium hydroxide
[MH] [N] (p) 1 cm^3 aqueous ammonia
[C] (q) 10 cm^3 dilute sulfuric acid
[O] [MH] [N] (r) 2.5 g potassium manganate (VII)

Equipment set-up



Technician’s notes, *continued*

Set 4

Each learner will require:

	(a)	Eye protection and gloves
	(b)	1 x Bunsen burner
	(c)	1 x heat proof mat
	(d)	1 x 25 cm^3 measuring cylinder
	(e)	access to balance weighing to at least 1 dp
	(f)	1 x weighing boat or small beaker
	(g)	1 x tripod and gauze
	(h)	2 x stand and 3 x clamp
	(i)	1 x 50 cm^3 or 100 cm^3 pear-shaped (or round-bottomed) flask
	(j)	1 x Liebig condenser with suitable tubes from tap and to sink, and bung if needed
	(k)	1 x adaptor to connect flask and condenser for distillation
	(l)	1 x thermometer holder (or bung to hold thermometer)
	(m)	1 x thermometer (–10 ºC to +110 ºC at 1 ºC)
[MH]	(n)	1 x small beaker
[F] [MH] [N]	(o)	1 cm^3 ethanol
[O] [MH] [N]	(p)	2.5 g potassium manganate (VII)
[C]	(q)	10 cm^3 dilute sulfuric acid
[C]	(r)	2 cm^3 concentrated sulfuric acid
[MH]	(s)	2 cm^3 0.2 $mol\ dm^{-3}$ sodium carbonate

Equipment set-up

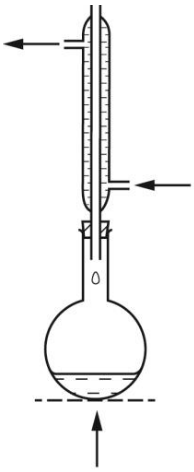


Diagram A

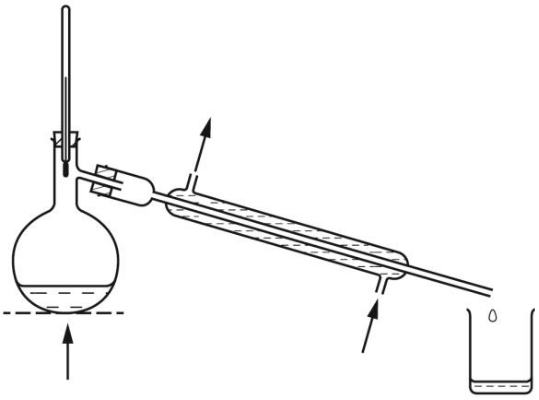


Diagram B

Technician’s notes, *continued*

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To investigate the reactions of ethanol by carrying out a range of different experiments.

Set 1. Test-tube experiments with ethanol.

Method

Safety:

- Wear eye protection
- Gloves may be worn
- Ethanol **[F] [H]**
- Sodium **[F] [C]**
- Glacial ethanoic acid **[C]**
- Concentrated sulfuric acid **[C]**
- 2 mol dm^{-3} sodium hydroxide **[C]**

Hazard symbols

C = corrosive substance

F = highly flammable substance

H = harmful or irritating substance

Experiment 1

1. Use a dropper to place 5 drops of ethanol in a small evaporating dish on a heat proof mat.
2. Use a lighted splint to ignite the ethanol.
3. Observe the flame and any residue.

Experiment 2

1. Pour about 1 cm^3 of ethanol into a test-tube. Place it in a test-tube rack.

2. **With teacher supervision**, use forceps to place a small lump of sodium on a piece of filter paper on a white tile. Use a knife to cut (approximately) a 1 mm cube of sodium and replace the rest of the lump in its bottle (under oil).

3. Use filter paper to wipe any excess oil off the small piece of sodium. **Do not touch the sodium with your fingers.**

4. Use forceps to transfer the small piece of sodium into the test-tube containing ethanol. (If any sodium remains after the reaction is complete, add more ethanol until all the sodium has reacted.)

Worksheet, *continued*

Experiment 3

1. Pour a 1 cm depth of ethanol into a test-tube.
2. Add an equal volume of aqueous sodium carbonate.

Experiment 4

1. Pour a 1 cm depth of ethanol into a test-tube.

2 Add about 1 cm^3 of aqueous iodine.

3. Add aqueous sodium hydroxide drop by drop, with shaking, until the brown colour of the iodine just disappears. Record your observations and cautiously smell the product. (Dispose of the product down the sink with plenty of water.)

Experiment 5

1. Pour about 1 cm^3 of ethanol into a boiling tube.
2. Add several drops of ethanoic acid **[C]** followed by 2 or 3 drops of concentrated sulfuric acid **[C]**. Carefully, shake the tube to mix the reactants.
3. Half fill a small beaker with aqueous sodium carbonate.
4. Use a holder for the boiling tube. Warm its contents gently and carefully over a low Bunsen flame whilst shaking gently.
5. When the mixture starts to boil, tip the contents of the boiling tube into the beaker.
6. Cautiously smell the product.

Results

Record **all** your observations.

Interpretation

Explain what is occurring in each experiment.

Worksheet, *continued*

Set 2. Dehydration of ethanol.

Method

Safety:

- Wear eye protection
- Ethanol **[F] [H]**
- Acidified aqueous potassium manganate(VII) **[H]**
- aqueous bromine **[H]**

Hazard symbols

H = harmful or irritating substance **F** = highly flammable substance

1. Use forceps to push mineral wool down to the bottom of the boiling tube as shown in the diagram. Add about 2 cm^3 of ethanol and add more mineral wool if not all the ethanol has soaked in.

2. Clamp the boiling tube horizontally and place broken pumice (or aluminium oxide) into the middle of tube as shown in the diagram.

3. Half fill a large beaker or small tub with water. Fill 4 test-tubes with water and place them upside down in the beaker. Make sure you have corks or bungs that fit the test-tubes ready to use.

4. Set up the apparatus as shown in the diagram. Clamp the boiling tube at its mouth.

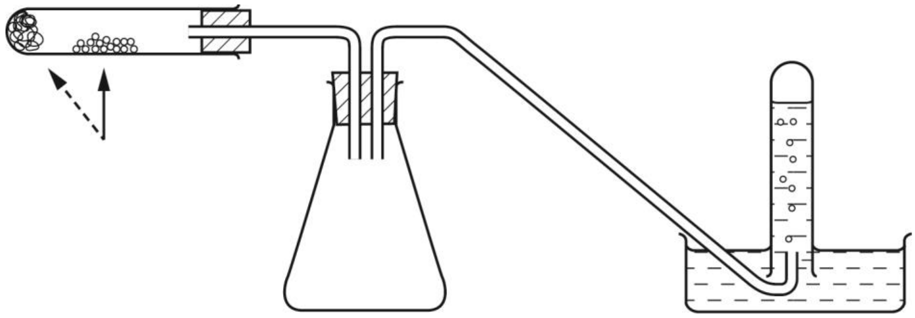
5. Heat the pumice (or aluminium oxide) strongly and occasionally move the flame for a very short period to gently heat the mineral wool to drive the ethanol vapour across the very hot pumice. Do not stop heating the pumice.

6. Collect the gas in the inverted test-tubes. Cork each tube when it fills.

7. When no more gas is produced, remove the delivery tube from the water then stop heating the pumice.

8. Carry out the following tests on the gas collected in three test-tubes.
    - Add a few drops of aqueous bromine, re-cork the tube and shake it.
    - Add a few drops of acidified aqueous potassium manganate(VII), re-cork the tube and shake it.
    - Bring a lighted splint to the mouth of the test tube.
      (Why might the gas collected in the first tube not react?)

Worksheet, *continued*



Results

Record **all** your observations.

Interpretation

Explain what is occurring in each part of the experiment.
Give equations for all reactions.

Worksheet, *continued*

Set 3. Partial oxidation of ethanol.

Method

Safety:

- Wear eye protection
- Ethanol **[F] [H]**
- Potassium manganate(VII) **[H] [O] [N]**
- 1 mol dm^{-3} sulfuric acid **[H]**

Hazard symbols

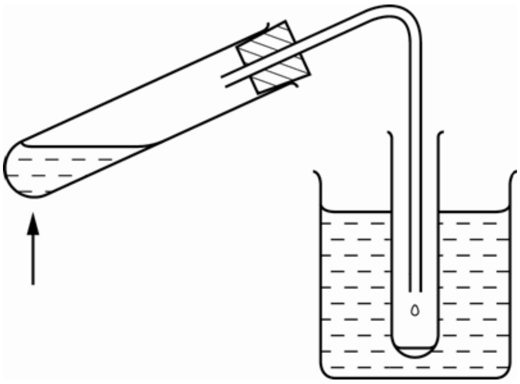
H = harmful or irritating substance

F = highly flammable substance

N = harmful to the environment

O = oxidising substance

1. Use a measuring cylinder to transfer 10 cm^3 of dilute sulfuric acid into a boiling tube.
2. Use the weighing boat or small beaker to weigh out about 2.3 g of potassium manganate(VII) and add the solid to the boiling tube.
3. Swirl the tube to dissolve the potassium manganate(VII) in the acid. Add 2 or 3 anti-bumping granules.
4. Add about 4 cm^3 of ethanol a little at a time to the boiling tube. Shake the tube carefully after each addition.
5. Half-fill a beaker with iced water. This will act as a condenser.
6. Set up the apparatus as shown in the diagram.



7. Warm the reaction mixture gently and collect the product in the test-tube.
8. Carry out the following tests on portions of the product.
   - Cautiously smell the product and compare the smell with that of ethanol.

Worksheet, *continued*

- Transfer about a 1 cm depth of the product into a test-tube. Add several drops of Fehling's A solution followed by enough Fehling's B solution to make a deep blue solution. Place the tube in a beaker half-full of water and heat the water. (This acts as a hot water bath.)

- To a 1 cm depth of aqueous silver nitrate in a **clean** test-tube add 1 or 2 drops of aqueous sodium hydroxide and shake the tube. A brown precipitate will form. Add aqueous ammonia drop by drop with shaking until the brown precipitate **just** dissolves. You have now made Tollens' reagent. Now add about a 1 cm depth of the product, shake the tube to mix the reactants. Place the test-tube in the hot water bath. (Dispose of the contents of the tube down the sink with plenty of water as soon as the reaction is complete.)

The preparation of the product may be carried out using the apparatus for distillation shown in diagram B of the Set 4 method. However, the test tube used to collect the product must be surrounded by iced water, as shown above.

Results

Record **all** your observations.

Interpretation

Explain what is occurring in each part of the experiment.

Worksheet, *continued*

Set 4. Further oxidation of ethanol.

Method

Safety:

- Wear eye protection
- Gloves may be worn
- Ethanol **[F] [H]**
- Potassium manganate(VII) **[H] [O] [N]**
- 1 mol dm^{-3} sulfuric acid **[H]**
- concentrated sulfuric acid **[C]**

Hazard symbols

C = corrosive substance

H = harmful or irritating substance

N = harmful to the environment

F = highly flammable substance

O = oxidising substance

T = toxic substance

1. Use a measuring cylinder to transfer 10 cm^3 of dilute sulfuric acid into a pear-shaped (or round-bottomed) flask.

2. Use the weighing boat or small beaker and weigh out about 2.3 g of potassium manganate(VII) and add the solid to the flask. Swirl the flask to dissolve the potassium manganate(VII) in the acid.

3. Add 2 or 3 anti-bumping granules. Add 2 cm^3 of concentrated sulfuric acid **[care]** in **small** portions while cooling the mixture under the tap or in a beaker of cold water.

4. Set up the apparatus for refluxing as shown in diagram A.

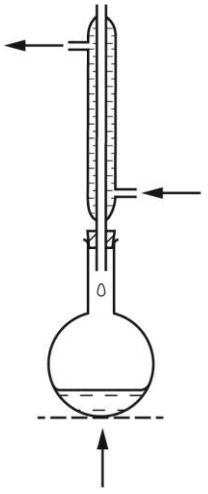


Diagram A

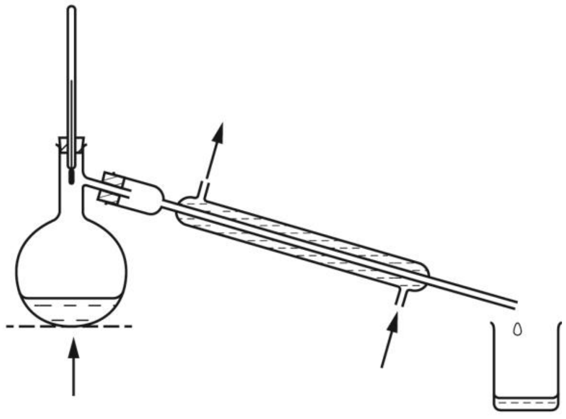


Diagram B

Worksheet, *continued*

5. Run water from the tap through the outer tubing of the condenser. Add 1 cm^3 of ethanol a few drops at a time down the middle of the condenser into the pear-shaped flask.

6. Heat the reaction mixture carefully until it boils. Adjust the flame so that there is a steady drip-back of reactants into the flask from the condenser and vapour does not escape from the top. Reflux the mixture for about 20 minutes then switch off the Bunsen burner and allow the flask to cool.

7. Remove the condenser and set up the apparatus for distillation as shown in diagram B.

8. Distil off a few cm^3 of product.
   - Smell the product cautiously.
   - Add about 1 cm^3 of aqueous sodium carbonate to the product.

Results

Record **all** your observations.

Interpretation

Explain what is occurring in each part of the experiment.

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Practical Booklet 7

Cell potentials and the Nernst equation

Cambridge International AS & A Level

Chemistry 9701

Version 2.0

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To investigate the magnitude of cell potentials using a variety of half cells, and to investigate the effect of change of concentration on the cell potential.

Outcomes

Syllabus section 6.1, 6.3(c), (d), (e), (h) and (i) as well as experimental skills 1, 2, 3, and 4.

Further work: syllabus section 6.2(b)

Syllabus sections 12.1 (h) and 12.2 (m)

Skills included in the practical

A Level skills	How learners develop the skills
Planning	identify a safe and efficient procedure that when followed would lead to a reliable result describe the steps necessary to carry out the procedure suggest appropriate volumes and concentrations describe how to vary the independent variable and how the dependent variable is to be measured
Analysis	calculate concentrations of solutions used in the half cells use the Nernst equation describe the patterns and trends in their data
Evaluation	identify sources of error in the procedure suggest ways to improve the accuracy of the procedure suggest ways in which to extend the investigation to answer a new question
Conclusions	determine electron flow in the external circuit deduce whether the data collected supports the Nernst equation make scientific explanations of the data prediction of relative strengths of oxidising agents (and of reducing agents)

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	set up and use apparatus to an appropriate level of precision
PDO recording	record the pd, half-cell description and sign with appropriate headings and units

Methods A and B

- **Learners must wear eye protection for these investigations.**
- The simplest type of electrode consists of a metal dipping into an aqueous solution of its ions. The metal and ions form an equilibrium: $M(s) \rightleftharpoons M^{2+}(aq) + 2e^-$ (for metals that form 2+ ions).

  If the metal reacts, electrons are produced on the surface of the metal. The more reactive the metal is, the more electrons are produced, so the more negative the metal and the electrode become.
- A simple cell is made by connecting together two metal electrodes (half-cells), using a salt bridge. This makes electrical contact between the two electrodes, with no mixing of the solutions.
- The voltage generated by the cell depends mainly on the difference in reactivity of the two metals. The concentration of the solution of the metal ions into which the metals dip also affects the voltage slightly.
- The equilibrium between a metal and its ions is a redox reaction, so other redox pairs can be used in a half-cell (eg $Fe^{2+}(aq)$ / $Fe^{3+}(aq)$). In this case an inert metal, platinum, is used to provide the pathway for electrons in and out of the half-cell.
- When carrying out the experiments, learners should be aware that a fresh salt bridge is needed for each pair of half-cells. They should also take care that crocodile clips (or other connecters) do not dip into the solutions of ions under test. They should check the connections for corrosion.
- In method B, learners will plan an experiment to investigate changing the concentration of one of the solutions in a cell or half-cell, and determine the effect this has on the pd of the cell formed. In this way they will investigate the Nernst equation. (It may be necessary to prompt learners to alter concentrations by factors of 10 as the Nernst equation includes a $\log_{10}$ term.)

Results

- Learners should tabulate results clearly, showing which is the positive half of the cell and recording the value of the cell pd / V.
- All voltmeter readings should be shown to the same number of decimal places.

Interpretation and evaluation

- Discussion can take place to determine which term is more appropriate: cell emf or cell pd.
- The reasons for differences between the experimentally determined cell potentials and the $E^\ominus$ values can be discussed (concentrations not 1.00 mol dm^{-3}, temperature not 298 K, some current flow).
- The methods to reduce the concentrations accurately can be discussed. A simple method is to use measuring cylinders or more precisely calibrated apparatus can be used instead.
- The effect of changes in concentration on the $M^{n+}(aq) + ne^- \rightleftharpoons M(s)$ equilibrium can be discussed.

Typical results

$Zn(s)\,|\,Zn^{2+}(aq)\ \|\ Cu^{2+}(aq)\,|\,Cu(s)$ = 1.07 V
$Cu(s)\,|\,Cu^{2+}(aq)\ \|\ Ag^{+}(aq)\,|\,Ag(s)$ = 0.45 V
$Zn(s)\,|\,Zn^{2+}(aq)\ \|\ Ag^{+}(aq)\,|\,Ag(s)$ = 1.51 V

A simple demonstration of the effect of changing one concentration in a half-cell of two solutions is to use the $Pt\,|\,I_2(aq), I^-(aq)$ half-cell connected to one of the others and take a reading. Then add solid potassium iodide to the half-cell, stir and take a new voltmeter reading. (The concentration of aqueous iodine was not known.)

$Cu(s)\,|\,Cu^{2+}(aq)\ \|\ I^-(aq), I_2(aq)\,|\,Pt$ = 0.27 V
With an increased concentration of iodide ions,
$Cu(s)\,|\,Cu^{2+}(aq)\ \|\ I^-(aq), I_2(aq)\,|\,Pt$ = 0.21 V

Calculation

If the Cu^{2+} solution is diluted by a factor of 10, the cell potential should decrease (become less positive) by approximately 0.03 V. Hence the voltmeter reading with the original Zn half cell and the new Cu half-cell should be 1.04 V from the results above.

Further work

The connection between cells producing electrical energy from chemical potential energy and electrolysis where electrical energy is used to produce chemical energy can be discussed (reversible reactions). The ability of metals to donate electrons and the ease of reduction of their ions can be linked. Similarly, the oxidising ability of non-metal systems such as the halogens and the ease of producing the non-metal by electrolysis can be linked.

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Information for technicians

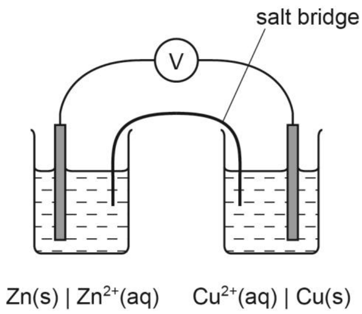
Each learner will require:

(a) Eye protection
(b) 1 x 250 cm^3 beaker
(c) 6 x 100 cm^3 beaker (or smaller)
(d) 1 x 25 cm^3 measuring cylinder
(e) 1 x high resistance d.c. voltmeter or multimeter (reading to **at least** 0.1 V)
(f) 2 x crocodile clips
(g) 2 x connecting wires
(h) filter paper, scissors and forceps
[O] (i) 25 cm^3 saturated aqueous potassium nitrate(V)
[MH] [N] (j) 50.0 cm^3 1.0 mol dm^{-3} copper(II) sulfate
[MH] [N] [C] (k) 50.0 cm^3 1.0 mol dm^{-3} zinc sulfate
[N] [HH] (l) 50.0 cm^3 1.0 mol dm^{-3} magnesium sulfate
[MH] (m) 50.0 cm^3 1.0 mol dm^{-3} iron(II) sulfate
[C] [MH] (n) 50.0 cm^3 1.0 mol dm^{-3} iron(III) chloride
[C] [O] [N] (o) 50.0 cm^3 0.05 mol dm^{-3} silver nitrate
[N] (p) 50.0 cm^3 0.02 mol dm^{-3} iodine dissolved in approx 0.05 mol dm^{-3} potassium iodide
(q) 1 x copper strip (electrode) with surface cleaned with fine sandpaper
[F] [N] (r) 1 x zinc (electrode) with surface cleaned with fine sandpaper
[F] (s) 1 x magnesium ribbon (electrode) with surface cleaned with fine sandpaper
(t) access to silver electrode or silver wire (a silver ring may be used)
(u) access to platinum electrode
(v) potassium iodide

Additional Instructions

The solutions may be retained and reused provided they have not been mixed or otherwise contaminated and there has been no significant current flow. (The amount of KNO_3 entering the solutions is minimal.)

Equipment set-up



Technician's notes, *continued*

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To investigate the magnitude of cell potentials using a variety of half-cells, and to investigate the effect of change of concentration on the cell potential.

Safety:

- Wear eye protection.
- 1 mol dm^{-3} copper(II) sulfate **[H] [N]**
- 1 mol dm^{-3} zinc sulfate **[H][N]**
- 1 mol dm^{-3} iron(II) sulfate **[H]**
- 1 mol dm^{-3} iron(III) chloride **[H]**
- saturated aqueous potassium nitrate(V) **[O]**

Hazard symbols

H = harmful or irritating substance

O = oxidising substance

N = harmful to the environment

Method A

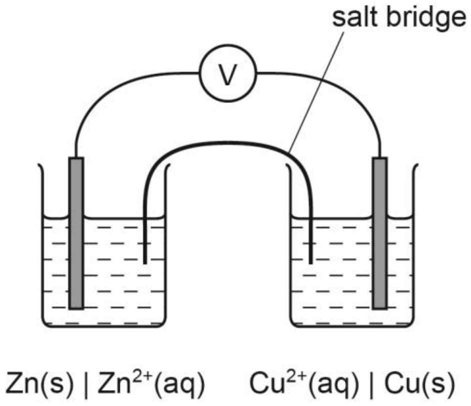
1. Pour a small volume of saturated aqueous potassium nitrate(V) into the 250 cm^3 beaker. Cut strips of filter paper and soak them in the potassium nitrate. These will be your salt bridges.

2. Use a crocodile clip to grip a strip of clean copper metal in a 100 cm^3 (or smaller) beaker. Add 1 mol dm^{-3} copper(II) sulfate to the beaker until the solution covers some of the copper metal. This is your $Cu(s)|Cu^{2+}(aq)$ half-cell.

3. Use a crocodile clip to grip a strip of clean zinc metal in a small beaker. Add 1 mol dm^{-3} zinc sulfate to the beaker until the solution covers some of the zinc metal. This is your $Zn(s)|Zn^{2+}(aq)$ half-cell.

4. Place the two beakers from 2 and 3 close together. Use connecting wires to connect the metal strips to either side of a high resistance d.c. voltmeter or a multimeter adjusted to the appropriate range.

5. Use forceps to remove a strip of filter paper from the saturated aqueous potassium nitrate and position it so one end is in the aqueous copper(II) sulfate and the other end is in the aqueous zinc sulfate.



Worksheet, *continued*

6. Take a voltmeter reading as soon as the circuit is complete. Record your reading. Record which half-cell is attached to the positive terminal of the voltmeter.

7. Disconnect the voltmeter and discard the salt bridge. Do not dismantle your half-cells.

8. Use a crocodile clip to grip a silver electrode (a silver wire or a volunteer's ring are suitable) in a small beaker. Add 0.05 mol dm^{-3} silver nitrate to the beaker until the solution covers some of the silver metal. This is your $Ag(s)|Ag^+(aq)$ half-cell. Place it close to the $Cu(s)|Cu^{2+}(aq)$ half-cell.
(If a volunteer's ring is used, make sure it is washed well with water before it is worn again.)

9. Use the connecting wires to connect the metal strips to either side of the high resistance d.c. voltmeter or multimeter.

10. Use forceps to remove a strip of filter paper from the saturated aqueous potassium nitrate(V) and position it so one end is in the aqueous copper(II) sulfate and the other end is in the aqueous silver nitrate.

11. Take a voltmeter reading as soon as the circuit is complete. Record your reading. Record which half-cell is attached to the positive terminal of the voltmeter.

12. Disconnect the voltmeter and discard the salt bridge.

13. Use your voltmeter readings to **predict** the cell potential if the half-cells used are $Ag(s)|Ag^+(aq)$ and $Zn(s)|Zn^{2+}(aq)$. Which half-cell should be connected to the positive terminal?

14. Set up the cell using a fresh salt bridge. Record the new voltmeter reading and compare it with your predicted value.

15. Use a 25 cm^3 measuring cylinder to place equal volumes of aqueous iron(II) sulfate and aqueous iron(III) chloride in a small beaker. Dip a platinum electrode into the solution. This is the $Pt|Fe^{2+}(aq), Fe^{3+}(aq)$ half-cell.

16. Connect the $Pt|Fe^{2+}(aq), Fe^{3+}(aq)$ half cell with one of the others. Record the new voltmeter reading and which half-cell is connected to the positive terminal.

17. Use other available solutions with the appropriate metal strips or electrodes to investigate which half-cell is the best provider of electrons to the external circuit. (The substances available may include magnesium ribbon, 1 mol dm^{-3} magnesium sulfate, 0.02 mol dm^{-3} iodine in approx. 0.05 mol dm^{-3} potassium iodide.)

Results

Record **all** your observations.

Tabulate your results clearly, showing which is the positive half of the cell and recording the value of the cell pd / V.
All voltmeter readings should be shown to the same number of decimal places.

Worksheet, *continued*

Interpretation and evaluation

1. Comment on the accuracy of your predicted value compared with the experimental value you obtained for the $Zn(s)|Zn^{2+}(aq)||Ag^{+}(aq)|Ag(s)$ cell.

2. The data book value for this combination of half-cells is 1.56 V. Suggest why your experimental value is different from the data book value.

3. Which substance of those you tested has the greatest ability to donate electrons to the external circuit?

4. Which substance of those you tested would you expect to be the best oxidising agent?

Method B

The Nernst equation is given as:

$$E = E^{\ominus} + (0.059/z)\log\frac{[\text{reduced species}]}{[\text{oxidised species}]}$$

where *E* is the cell emf, $E^{\ominus}$ is the standard cell emf and z is the difference in charge between the reduced and oxidised species.

You should write a plan for experiments you could carry out that will test the Nernst equation.

- You may use any of the apparatus, half-cells and results from method A. You may also use distilled water, solid potassium iodide and other normal laboratory apparatus you may require.
- Make sure that your plan includes the concentrations of the solutions you will use and how you will prepare solutions of these concentrations.
- You should make it clear in your plan what you are keeping the same (controlled variables) and what you are changing (independent variable).

Carry out your planned experiments.

Results

Record and tabulate the results of your experiments.

Tabulate your results clearly, showing which is the positive half of the cell and recording the value of the cell pd / V.
All voltmeter readings should be shown to the same number of decimal places.

Interpretation and evaluation

1. Calculate the theoretical E_{cell} values for your experiments.

2. Compare your experimental values for E_{cell} with those you calculated.
   Do your results support the Nernst equation?

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Practical Booklet 8

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Chemistry 9701

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The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To investigate the changes in pH during titrations using strong and weak acids and alkalis and to understand the nature of buffer solutions.

Outcomes

Syllabus section 7.2(c), (e) and (f) as well as experimental skills 2, 3, and 4

Further work: syllabus section 7.2(h) and (k) as well as experimental skill 1

Skills included in the practical

A Level skills	How learners develop the skills
Planning	plan how to extend an investigation to answer a new question identify the steps necessary to carry out the procedure show an understanding of how and why the procedure suggested will be effective
Analysis	use graphs to draw attention to the key points in quantitative data describe and summarise the key points of a set of observations understanding the nature of buffer solutions
Evaluation	identify where repeated readings are appropriate suggest modifications to the experimental procedure that will improve the accuracy of finding the equivalence point
Conclusions	calculation of pH of solutions (including buffers) determine the equivalence point for each set of observations suggest an appropriate indicator/indicators for acid – alkali combinations

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO quality	set up and use the apparatus to an appropriate level of precision obtain results that are close to those of an experienced chemist
PDO recording	record the pH values and total volumes of alkali added
PDO display	show the level of precision of their readings
PDO layout	plot appropriate variables accurately on appropriate, clearly labelled *x*- and *y*-axes
ACE conclusions	carry out calculations involving moles of solutions

Method

- **Learners must wear eye protection for these investigations.**

- The pH changes during the reaction of sodium hydroxide with hydrochloric acid are investigated. It is important that both solutions have the same concentration. Before learners carry out their work, the relative concentrations should be checked by the teacher (or technician) using a conventional titration with thymolphthalein as indicator: the end point should be within the range 24.8 – 25.2 cm^3.

- It is easier to identify the buffer action that takes place if the ethanoic acid (the weak electrolyte) is pipetted into the beaker and the sodium hydroxide (strong alkali) is added from the burette 5.0 cm^3 at a time.

- Buffer action takes place when about 12.5cm^3 of NaOH are added, that is half of the ethanoic acid has been neutralised. This is because the concentrations of ethanoic acid (unreacted) and sodium ethanoate (produced) are equal at this point in the titration. If time allows, the experiment should be repeated with smaller volumes of sodium hydroxide added at around 12.5 cm^3, so that the buffering effect is more clearly observed.

- In the same way, it is desirable that learners should observe in more detail the nature of pH changes near the end-point. In order to do this, the experiment should be repeated with a smaller volume of sodium hydroxide added at around 25.0cm^3.

- Learners should notice the sudden change in pH that takes place at the end-point. This sudden change allows the use of conventional indicators to show the end-point of a titration, because the indicator goes right through its colour change range while only a couple of drops of titrant are added at the end point.

- It is possible for learners to use narrow range universal indicator papers in place of a pH probe, if the latter is not available in sufficient numbers. In this case it is easier to use a conical flask for the reaction and swirl the solution after each addition of alkali. Then dip a glass rod (stirrer) into the mixture and touch the tip of the rod onto the appropriate indicator paper.

- Each learner/pair of learners should carry out the experiment with ethanoic acid and sodium hydroxide. If time allows, different learners/pairs of learners should carry out the experiment using one other combination of strong/weak acid/alkali (NaOH + HCl, HCl + aq NH_3, NH_3 + CH_3COOH). Class members can then pool their results before plotting graphs.

- Using these graphs, learners can discuss which indicators, if any, would be suitable to determine the end-points of these titrations.

Further work

- Learners investigate the characteristics of buffer solutions. The buffering action breaks down if too much acid or alkali is added to the buffer solution, because one of the chemicals needed in the buffering mixture gets used up.

Results

- Learners should tabulate results clearly with unambiguous headings and correctly displayed units.
- All volumes and pH readings should be shown to a consistent number of decimal places.
- Plotted results should cover at least half of the available grid and be drawn with a sharp pencil. The plotting should be accurate (within ½ a small square and in the correct half of the small square, any points on a line having the centre of the mark on the line).

Interpretation and evaluation

- The definition of pH can be introduced or revised.

- Discussion can take place to determine where the equivalence point lies on each graph.

- Possible reasons for different end-point pH values for the different combinations can be brainstormed. (Incomplete dissociation of the weak acid or alkali should be revised.)

- Possible ways of improving the end-point determination can be discussed. (Add NaOH in much smaller volumes as soon as the pH begins to change more rapidly than previously. This will give more points around the end-point so a more accurate line can be drawn.)

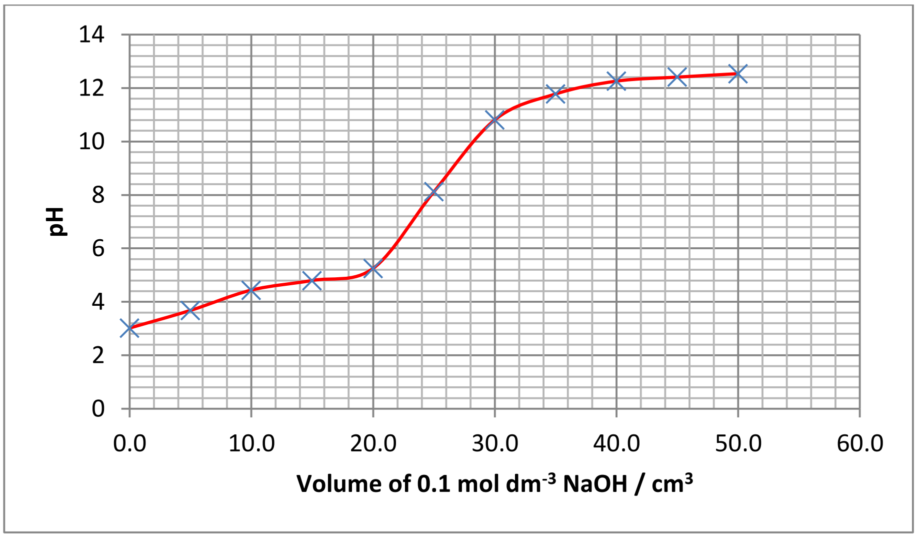
- The suitability of the indicators for each system can be discussed. (The indicator(s) selected should have colour changes in the almost vertical portion of the graph so that a colour change would be apparent on adding one drop (0.05 cm^3) of solution from the burette.)

  The lack of suitable indicator for a weak acid / weak alkali combination can be discussed (lack of significant near-vertical section). (Each weak acid /weak alkali system will have a different pH value at the end-point which will depend on the K_a / pK_a and K_b / pK_b values.

Typical results

0.1 mol dm^{-3} sodium hydroxide titrated with 25.0 cm^3 0.1 mol dm^{-3} ethanoic acid

$pK_a = 4.62$, $K_a = 2.4 \times 10^{-3}$ mol dm^{-3}



Further work

- Before starting further work the nature of buffer solutions can be introduced or revised. The graph of sodium hydroxide versus ethanoic acid can be used to illustrate the partial neutralisation of the weak acid by the strong alkali. Learners should note that the pH changes only slightly until the buffering capacity of the system is exceeded.

- Learners could use their graphs to determine the relative volumes of sodium hydroxide and ethanoic acid which correspond to a pH of 5.0. They can scale these volumes to work out how to prepare 25 cm^3 of buffer solution of pH = 5.

  Learners should suggest how the appropriate volume of ethanoic acid could be measured.

  The reasons for the pH value differing from 5.0 for the solution made can be discussed. (Smaller additions of NaOH beyond 20.0 cm^3 to obtain more points in the region where neutralisation occurs would give a more accurate volume of NaOH to use to prepare the buffer solution.)

- The equations involved in buffer systems can be used and the effects of adding small volumes of strong acid or alkali checked against experimental results.

  $CH_3COOH(aq) \rightleftharpoons H^+(aq) + CH_3COO^-(aq)$ (1)
  $CH_3COONa(aq) \rightarrow Na^+(aq) + CH_3COO^-(aq)$ (2)
  $H_2O(l) \rightleftharpoons H^+(aq) + OH^-(aq)$ (3)

  The common ion effect of the ethanoate ion from (2) on the equilibrium shown in (1) can be discussed.

- Increasing the concentrations of the solutions making the buffer would give it greater buffering capacity.

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Information for technicians

Each learner will require:

(a) Eye protection
(b) 2 x 100 cm^3 beaker
(c) 1 x 50 cm^3 burette
(d) 1 x burette stand and clamp
(e) 1 x filter funnel (for filling burette)
(f) 1 x 25 cm^3 pipette
(g) 1 x pipette filler
(h) 1 x glass rod
(i) 2 x teat / dropping pipettes
(j) 1 x pH probe / pH meter
[MH] (k) 25 cm^3 pH7 buffer solution (ammonium ethanoate)
[MH] (l) 150 cm^3 0.100 mol dm^{-3} sodium hydroxide
[MH] (m) 120 cm^3 0.100 mol dm^{-3} ethanoic acid
[N] [MH] (n) 120 cm^3 0.100 mol dm^{-3} ammonia
[MH] (o) 120 cm^3 0.100 mol dm^{-3} hydrochloric acid
[MH] (p) 20 cm^3 0.100 mol dm^{-3} potassium hydroxide
(q) paper towel

Additional Instructions

If pH probes / pH meters are not available then narrow range universal indicator papers may be used with the chart of colour and pH value for each. If universal indicator papers are used then learners should use conical flasks instead of one of the 100 cm^3 beakers for the reaction mixtures.

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To investigate the changes in pH during titrations using strong and weak acids and alkalis and to understand the nature of buffer solutions.

Method

Safety:

- Wear eye protection.
- 0.1 mol dm^{-3} sodium hydroxide **[H]**
- 0.1 mol dm^{-3} potassium hydroxide **[H]**

Hazard symbols

H = harmful or irritating substance

1. Pour sufficient pH7 buffer solution into a 100 cm^3 beaker (or smaller) so that the bulb of the pH probe is covered by solution. Calibrate the pH meter so it reads 7.0 when the probe is placed in the buffer solution.

2. Using a pipette filler, wash the 25 cm^3 pipette with a little 0.1 mol dm^{-3} ethanoic acid and discard the washings. Transfer 25.0 cm^3 of your acid solution into a 100 cm^3 beaker. (Touch the bottom of the pipette against the wall of the beaker or onto the surface of the solution to deliver the correct volume.)

3. Wash the burette with a little 0.1 mol dm^{-3} sodium hydroxide and discard the washings. Then fill the burette to the 0.00 cm^3 mark, making sure that the region under the tap is full.

4. Remove the pH probe from the buffer solution, rinse it with a little distilled water and shake it gently to remove excess water.
   Place the probe in the solution of acid. Take and record a pH reading. This is your reading with 0.0 cm^3 alkali added.

5. Add 5.0 cm^3 of 0.1 mol dm^{-3} sodium hydroxide to the acid in the beaker. Stir the mixture thoroughly. Measure and record the new pH reading. Record the exact volume of alkali added and the pH value.

6. Add a second 5.0 cm^3 portion of your alkali to the mixture in the beaker. Stir the mixture thoroughly and then take a new pH reading. Record the **total** volume of alkali added and the pH value. It does not matter if the volume added is not exactly 10.0 cm^3 as long as you record the accurate volume of alkali you have run out of the burette so far.

7. Continue adding 5.0 cm^3 portions of alkali to the beaker, stirring, and taking new readings of the total volume and pH until you have added a total volume of 50.0 cm^3 of alkali. (Take care not to spill the reaction mixture as you stir when larger total volumes of alkali are added.) Record all data in a table.

8. Repeat steps 1 - 5 of the procedure. Measure the pH when the following **total** volumes of sodium hydroxide have been added: 10.0, 12.0, 12.2, 12.4, 12.6, 12.8, 13.0, 15.0 cm^3

Worksheet, *continued*

9. Following on from step 9, measure the pH when the following **total** volumes of sodium hydroxide have been added: 20.0, 22.0, 24.0, 24.4, 24.6, 24.8, 25.0, 25.2, 25.4, 25.6, 26.0, 28.0, 30.0 cm^3.

10. Repeat the procedure using different combinations of strong/weak acid/alkali.
    (a) 0.1 $mol\,dm^{-3}$ sodium hydroxide (burette) with 25.0 cm^3 of 0.1 $mol\,dm^{-3}$ hydrochloric acid (pipette).
    (b) 0.1 $mol\,dm^{-3}$ ammonia (burette) with 25.0 cm^3 of 0.1 $mol\,dm^{-3}$ hydrochloric acid (pipette).
    (c) 0.1 $mol\,dm^{-3}$ ammonia (burette) with 25.0 cm^3 of 0.1 $mol\,dm^{-3}$ ethanoic acid (pipette).

11. For each combination of acid and alkali, plot a graph of pH against volume of alkali added.

Results

Record **all** your observations.
Record and tabulate the results of your experiments.

Tabulate your results clearly with unambiguous headings and correctly displayed units.
All volumes and pH readings should be shown to a consistent number of decimal places.
Plotted results should cover at least half of the available grid and be drawn with a sharp pencil. The plotting should be accurate (within ½ a small square and in the correct half of the small square, any points on a line having the centre of the mark on the line.)

Interpretation and evaluation

1. The graph of your experiment with sodium hydroxide and ethanoic acid should be approximately S-shaped. All the ethanoic acid has reacted at the mid-point of the steepest section of the graph.
   From your graph, what volume of aqueous sodium hydroxide is needed to neutralise 25.0 cm^3 of aqueous ethanoic acid? (Are the two solutions of exactly the same concentration?)

2. What is the pH of the mixture at this point?
   Suggest why your answer is not pH = 7.

3. From your graph, find the pH of the mixture when **half** the ethanoic acid has reacted. At this point the concentration of acid equals the concentration of base.
   Use this information and the equation below to calculate the acid dissociation constant for ethanoic acid. (You may convert the equation into a log expression.)

$$K_a = [H^+(aq)]\,[CH_3COO^-(aq)] \,/\, [CH_3COOH(aq)]$$

Worksheet, *continued*

4. The syllabus lists four commonly used indicators. The names and ranges of pH over which their colours change is given below:

Indicator	pH range
methyl orange	2.9 to 4.6
bromophenol blue	3.0 to 4.5
thymol blue	8.0 to 9.6
thymolphthalein	9.3 to 10.5

Which indicator(s) would be suitable for determining the end-point for a titration of sodium hydroxide with ethanoic acid if no pH meter was available?

5. Find the equivalence points and pH at the end-point for the other 3 pairs of acid-alkali.
Suggest, and explain, which of the above indicators would be suitable for determining the end-point for these titrations.
Why would it be difficult to carry out a similar titration involving ammonia and a weak acid?

Further work

- Decide how to use your results with the aqueous sodium hydroxide and aqueous ethanoic acid to make a buffer solution with pH 5.0.

  Make 25 cm^3 of the pH 5.0 buffer using the same solutions as used in the original experiment. (You may wish to use additional apparatus.)

  Test the pH of your buffer solution.

  Is it exactly 5.0? If not, what errors may have occurred?

- Use 5 cm^3 portions of your buffer solution for each of the following experiments.

  - To the first portion, add five drops of 0.1 mol dm^{-3} potassium hydroxide. Record the new pH.
    Add five more drops of KOH and record the new pH.
    Keep adding five drops of KOH each time and record the pH of the solution.
    Keep adding KOH until there is a significant change in the pH.
    Why is there a limit to the volume of potassium hydroxide you are able to add with only a small change in pH?

  - To the second portion, add five drops of 0.1 mol dm^{-3} hydrochloric acid. Record the new pH.
    Add five more drops of the dilute hydrochloric acid to the mixture and record the new pH.. Keep adding five drops of hydrochloric acid each time and note what happens to the pH value after each addition.
    How would you make a buffer solution capable of resisting pH change when larger volumes of hydrochloric acid are added?

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Practical Booklet 9

Transition elements

Cambridge International AS & A Level Chemistry 9701

Version 2.0

Cambridge Pathway

Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To investigate some complexes of the copper(II) ion, Cu^{2+}, and to prepare a complex of copper containing a bidentate ligand.

Outcomes

Syllabus section 12.2(a), (b), (c), 12.3(c), 12.4(a), 12.5 and 5.3(a) (& 20.3(a)) as well as experimental skills 2 and 3

Further work: syllabus section 12.1(e)

Syllabus sections 12.1 (h) and 12.2 (m)

Skills included in the practical

A Level skills	How learners develop the skills
Planning	draw up a simple risk assessment for experiments that have been carried out
Analysis	recognise and understand the significance of observations made from test tube reactions make predictions based on data collected
Conclusions	draw conclusions from observations make scientific explanations of the observations and conclusions that they have described

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	collect an appropriate quantity of observations, including subtle differences in colour or solubility of materials
PDO recording	record observations precisely
ACE analysis	describe and summarise the key points of a set of observations

Method

- **Learners must wear eye protection for these investigations**. Gloves may be provided.

- Copper forms a range of observed complex ions which are easy to produce by test-tube reactions. The complexes absorb visible light at the red end of the spectrum, so most of them are varying shades of blue in colour.

- In the experiments described in Method A, learners will carry out some ligand substitution reactions and will observe a number of complex ions of copper(II). They will work with monodentate and multidentate ligands.

- Learners will consider the equilibria involved in these reactions. They will use Le Chatelier's principle to explain some of their observations and gain an understanding of the significance of stability constants.

- In Method B, learners will prepare a copper(II) complex containing aminoethanoic acid as a bidentate ligand, and use the technique of reduced pressure filtration to separate it from the reaction mixture. It may be necessary for learners (or pairs of learners) to share use of Buchner filtration apparatus.

- Learners should become confident in using small amounts of reagents to obtain all relevant observations. Learners should become confident in handling hazardous chemicals.

If time allows, **further work** may be carried out. Learners will carry out two redox reactions in which the copper(II) ion is reduced to copper(I). Reduction to copper(I) normally only occurs if the copper(I) compound to be produced is insoluble in water. Copper(I) iodide and copper(I) oxide (which is formed in both the Fehling's and Sandell's tests for aldehydes) are precipitated during the reactions described.

Results

- Learners should record all their results using the correct terminology.

Interpretation and evaluation

- The definition of ligand should be revised and the role of the lone pairs on nitrogen and oxygen atoms in the dative bonding of complex ions can be discussed.

- The stages in the formation of a new complex can be discussed ($[Cu(H_2O)_5Cl]^+$, etc).

- Learners should be aware that there are many complex ions containing multidentate ligands. They should understand how the formulae of and charges on such complex ions can be worked out.

- Discussion can take place about the factors affecting colours of complexes (charge on central metal ion, ligand field strength, ligands approaching along (linear, square planar, octahedral) or between (tetrahedral) d-orbital axes).

- Learners should understand that, in the reaction of $[Cu(H_2O)_6]^{2+}$ with ammonia, the ammonia first acts as a base, precipitating insoluble copper(II) hydroxide. With excess ammonia, this is followed by a ligand substitution reaction involving ammonia molecules.

- The idea of zwitterion formation of amino acids can be discussed, after carrying out the preparation in Method B.

$$Cu^{2+} + 2(^+H_3NCH_2COO^-) \rightarrow Cu(NH_2CH_2COO)_2 + 2H^+$$

- The usefulness of increased speed of filtration under reduced pressure can be introduced.

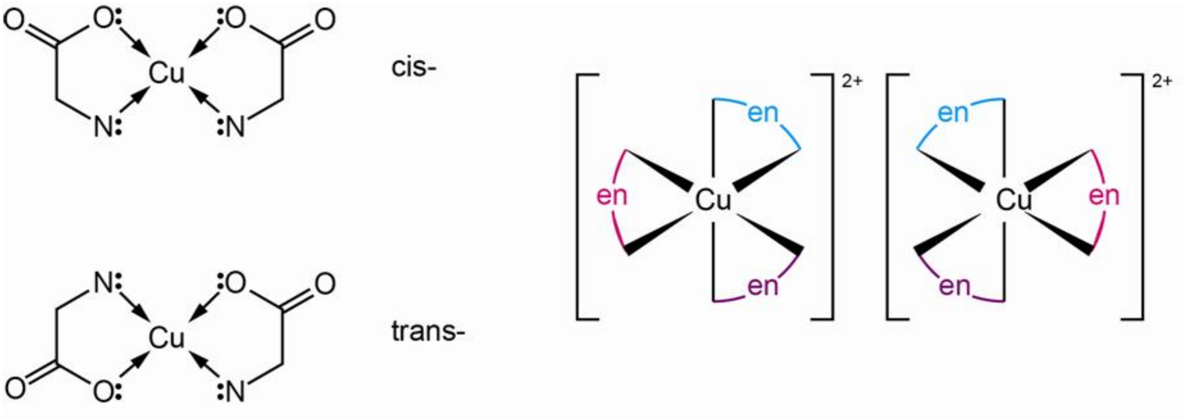
- The effect of stability constants on ligand substitution reactions should be discussed.
  K_{stab}: $[Cu(EDTA)]^{2-} > [Cu(NH_3)_4]^{2+} > [Cu(Cl)_4]^{2-}$

  Reforming of the hexaaquo complex in step 2 of method A can be used to illustrate Le Chatelier's Principle.

- The higher stability of the complexes involving chelating ligands can be discussed and linked to the increase in entropy (more particles on the right); log K_{stab} $[Cu(en)_3]^{2+} \approx 18.1$

$$[Cu(H_2O)_6]^{2+} + EDTA^{4-} \rightleftharpoons [Cu(EDTA)]^{2-} + 6H_2O$$

$$[Cu(H_2O)_6]^{2+} + 3en \rightleftharpoons [Cu(en)_3]^{2+} + 6H_2O$$



bis(glycinato)copper(II)

optical isomers: non-superimposable mirror image
tris(1,2-diaminoethane)copper(II)

Further work

- The effect of the d^{10} configuration on the colour of copper(I) iodide, CuI, can be discussed.

- If excess sodium thiosulfate is added, the CuI precipitate will dissolve.
  (This may be compared to the 'fixing' stage of wet photography:
  $AgBr(s) + 2S_2O_3^{2-}(aq) \rightarrow [Ag(S_2O_3)_2]^{3-}(aq) + Br^-(aq)$.)

- The reduction of Cu^{2+} to Cu_2O by –CHO can be used to revise the reducing property of aldehydes.

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Information for technicians

Each learner will require:

	(a)	Eye protection (gloves may be worn)
	(b)	8 x test-tube and 1 x boiling tube
	(c)	1 x test-tube rack
	(d)	4 x teat/dropping pipette
	(e)	1 x 50 cm^3 measuring cylinder
	(f)	1 x 250 cm^3 beaker
	(g)	2 x 100 cm^3 beaker
	(h)	1 x glass rod
	(i)	2 x spatula
	(j)	1 x Bunsen burner, heat proof mat, tripod and gauze
	(k)	1 x Buchner flask (side arm flask) *
	(l)	1 x Buchner funnel and filter paper to fit the funnel *
	(m)	1 x filter pump *
[N]	(n)	10 cm^3 0.5 mol dm^{-3} copper(II) sulfate
[MH] [C]	(o)	5 cm^3 concentrated hydrochloric acid **
[MH] [C] [N]	(p)	5 cm^3 ‘880’ ammonia **
	(q)	5 cm^3 0.2 mol dm^{-3} EDTA (disodium salt)
[HH] [MH]	(r)	1 g aminoethanoic acid (glycine)
[MH] [N]	(s)	2 g copper(II) carbonate
	(t)	5 cm^3 0.5 mol dm^{-3} potassium iodide
	(u)	1 cm^3 starch indicator
	(v)	5 cm^3 0.2 mol dm^{-3} sodium thiosulfate
[MH] [N]	(w)	0.5 g copper(II) sulfate pentahydrate
	(x)	1 g EDTA (disodium salt)
[C]	(y)	5 cm^3 2 mol dm^{-3} sodium hydroxide
	(z)	0.5 g glucose
	(aa)	70 cm^3 distilled water
	(bb)	paper towel
	(cc)	pen or label for marking glassware

Additional Instructions

* If these items are not available then supply 1 x conical flask, 1 x filter funnel and filter paper.
** These concentrated solutions should be situated in a fume cupboard if one is available.

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 GHS07 (*moderate hazard* MH)	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To investigate some complexes of the copper(II) ion, Cu^{2+}, and to prepare a complex of copper containing a bidentate ligand.

Method

Safety:

- Wear eye protection. Gloves may be worn
- concentrated hydrochloric acid **[C]**
- ‘880’ ammonia **[C] [N]** (concentrated aqueous ammonia)
- copper(II) carbonate **[H]**
- copper(II) sulfate pentahydrate **[H] [N]**
- 2 mol dm^{-3} sodium hydroxide **[C]**

Hazard symbols

C = corrosive substance

N = harmful to the environment

H = harmful or irritating substance

Method A

1. To a 1 cm depth of 0.5 mol dm^{-3} copper(II) sulfate in a test-tube add concentrated hydrochloric acid **[C]** drop by drop with shaking until there is no further change.
   Keep this solution for tests 2 and 3.

2. To a 0.5 cm depth of solution from 1 in a test-tube add water in small portions until the test-tube is about half full. Shake the tube between additions of water.

3. To a 0.5 cm depth of solution from 1 in a test-tube add ‘880’ ammonia **[C] [N]** drop by drop with shaking until there is no further change.
   Keep this solution for test 5.

4. To a 1 cm depth of 0.5 mol dm^{-3} copper(II) sulfate in a test-tube add 0.2 mol dm^{-3} EDTA until there is no further change.

5. Tip half the solution from test 3 into a test-tube and add 0.2 mol dm^{-3} EDTA until there is no further change.

6. Draw up a simple risk assessment for the experiments you have carried out

Worksheet, *continued*

Results

Record **all** your observations, including initial colours and states.

Method B

1. Pour approximately 10 cm^3 of distilled water into a small beaker.
2. Weigh out approximately 1 g aminoethanoic acid (glycine) and dissolve it in the water.
3. Add copper(II) carbonate **[H]** to the solution with stirring until no more solid will dissolve.
4. Filter the mixture (using a Buchner flask and funnel and a filter pump if available). Transfer the filtrate (solution) into a boiling tube and leave it for crystals to form.

Results

Record **all** your observations.

Interpretation and evaluation (A and B)

In Method A you saw four different complexes of Cu^{2+}.
These are $[Cu(H_2O)_6]^{2+}$, $[Cu(Cl)_4]^{2-}$, $[Cu(NH_3)_4]^{2+}$ and $[Cu(EDTA)]^{2-}$. They are (distorted) octahedral, tetrahedral, square planar and octahedral complexes respectively.

1. Why are the four complexes different colours?
2. Why did the colour change gradually in step 1?
3. Explain your observations in step 3.
4. Which complex has the largest stability constant? Give a reason for your answer.
5. $[Cu(H_2O)_6]^{2+} + 4Cl^- \rightleftharpoons [Cu(Cl)_4]^{2-} + 6H_2O \qquad K_{stab} \approx 4 \times 10^5$
   Use this information to explain the change in colour in step 2.
6. What is the coordination number of copper in $[Cu(EDTA)]^{2-}$ and what is the charge on the EDTA ion?
7. The stability constant for EDTA displacing water from around the Cu^{2+} ion is large ($\log K \approx 18.8$). There is little change in enthalpy for the reaction. Suggest a reason for this high stability.
8. In method A most of the ligands are monodentate but EDTA is hexadentate.
   Explain the meaning of the terms *monodentate* and *hexadentate*.

Worksheet, *continued*

9. In method B the Cu^{2+} ion forms a complex with aminoethanoic acid, which is a bidentate ligand. Draw the structure of aminoethanoic acid. Explain how it can act as a bidentate ligand.

   The complex formed has two aminoethanoate ions in a square planar arrangement around the Cu^{2+} ion. As the aminoethanoate ion is not symmetrical there can be two stereoisomers, cis- and trans-. The cis- form exists as pale blue needle-like crystals and the trans- form exists as blue-violet platelets. Which form of the complex did you make in method B?

   Draw the two arrangements of $[Cu(NH_2CH_2COO)_2]$ to show the geometric isomers.

10. Copper(II) ions can also form a complex with another bidentate ion, 1,2-diaminoethane (often simplified in this context to 'en'). The formula of the complex ion is $[Cu(en)_3]^{2+}$ and illustrates a different form of stereoisomerism from the glycine complex.

    Draw the two isomers of $[Cu(en)_3]^{2+}$ so that their 3-D structure is clear.

11. Would you expect the stability constant for $[Cu(en)_3]^{2+}$ to be closer to that of $[Cu(Cl)_4]^{2-}$ or $[Cu(EDTA)]^{2-}$? Give a reason for your answer.

12. What is the advantage of filtering using a Buchner flask, funnel and a filter pump, compared to normal filtration?

Further work

1. To a 1 cm depth of 0.5 mol dm^{-3} copper(II) sulfate in a test-tube add about a 4 cm depth of aqueous potassium iodide. Divide the mixture into two portions in different test-tubes and carry out the following tests.
   - (i) Add a few drops of starch indicator.
   - (ii) Add aqueous sodium thiosulfate a few drops at a time with shaking until the thiosulfate is in large excess and no further change takes place.

2. Weigh out 0.40 g copper(II) sulfate pentahydrate **[H] [N]** in a small beaker.
   Weigh out 0.90 g EDTA (disodium salt) and add the solid to the beaker.
   Add 40 cm^3 of distilled water and stir the contents of the beaker to dissolve the solids.
   Then add 10 cm^3 of 2 mol dm^{-3} sodium hydroxide **[C]**.
   Label this mixture Sandell's solution **[H]**.

   Half-fill a 250 cm^3 beaker with tap water and heat it over a Bunsen burner.
   This will be your hot water bath.

   Dissolve a small spatula measure of glucose in a 2 cm depth of distilled water in a test-tube.
   Add a 2 cm depth of Sandell's solution **[H]** then place the test-tube in the hot water bath and continue heating it until no further change takes place.

Worksheet, *continued*

Results

Record **all** your observations.

Interpretation and evaluation

1. Both experiments involve redox reactions.
   Write an ionic equation for the reaction between Cu^{2+}(aq) and I^-(aq).

   Explain the colour of the precipitate formed when Cu^{2+}(aq) reacted with I^-(aq) in terms of the electron configuration of the metal ion.

2. In some text books glucose is referred to as an aldose (a reducing sugar).

   What group present in glucose reacts with the Cu^{2+} complex in Sandell's solution?

   Identify the copper compound formed in the reaction.

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Practical Booklet 10

Amide hydrolysis

Cambridge International AS & A Level
Chemistry 9701

Version 2.0

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Pathway

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Introduction

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Guidance for teachers

Aim

To hydrolyse an aromatic amide, purify the product, calculate a percentage yield and determine the melting point.

Outcomes

Syllabus section 20.2 as well as experimental skills 2, 3, and 4

Skills included in the practical

A Level skills	How learners develop the skills
Analysis	calculate theoretical yield and percentage yield of product
Evaluation	identify the most significant sources of error in the experiment suggest ways to improve the accuracy of the procedure
Conclusions	draw conclusions from interpretations of observations, data and calculated values make scientific explanations of the data, observations and conclusions that they have described

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	follow instructions given in the form of written instructions or diagrams set up and use apparatus suitable for an organic synthesis
PDO display	show their working in calculations, and the key steps in their reasoning use the correct number of significant figures for calculated quantities

Methods A, B and C

This experiment is suitable for learners to work in pairs or small groups.

- **Learners must wear eye protection for these investigations**.

- In Method A, learners work in pairs or small groups to prepare benzenecarboxylic acid (benzoic acid, C_6H_5COOH) by alkaline hydrolysis of benzenecarboxamide (benzamide, $C_6H_5CONH_2$) under reflux. The first stage of the reaction (steps 7 – 9) produces sodium benezenecarboxylate.

- This salt is then converted into benzenecarboxylic acid by an acid displacement reaction using HCl in step 10. Benzenecarboxylic acid has a low solubility in cold water, due to its benzene ring, so the product precipitated is separated and collected by Buchner (reduced-pressure) filtration.

- In Method B, learners purify their product by recrystallization. It is important to use the minimum volume of hot solvent to dissolve the organic solid, otherwise much more of it than necessary is lost during the purification step. Since the impurities are present in low quantity in the impure product, they do not form a saturated solution, so they remain in solution when the saturated solution is cooled.

- In Method C, learners will determine the melting point of their purified solid. Impurities lower the melting point of a solid and also cause the melting point to take place over a wider range As a result, they will assess the purity of their final sample

- As a result of carrying out these experiments, learners should become familiar with common techniques used in organic synthesis and purification. They should also become confident in assembling and using this type of apparatus.

Results

- Learners should draw up a table for their results which includes the masses of benzenecarboxamide (benzamide) and benzenecarboxylic acid (benzoic acid).

- Their recorded balance readings should be to a consistent number of decimal places.

- They should note the colours of reactant and product and also any change in the colours of the litmus papers.

Interpretation and evaluation

- Other examples of hydrolysis can be revised (esters, acyl chlorides as having a similar group – and their relative reactivity towards hydrolysis).

- The correct way of showing reaction mechanisms can be revised (curly arrow from a lone pair on OH^- to $\delta+$ C of –COO– group).

- If the learners fail to detect the ammonia from the condenser, the likely products of the reaction can be discussed.

- The calculation of percentage yield can be revised. The number of significant figures will depend on the precision of the balance used. (Syllabus: Calculated quantities should be given to the same number of significant figures as (or one more than) the measured quantity of least accuracy.)

- The percentage yield can be collected from each group and compared. This can form the basis for brainstorming the possible reasons why the yield must be less than 100% and discussion of ways to maximise the yield.
  (Yield: the cold solution will be saturated with benzenecarboxamide so 100% yield is impossible. If the water is evaporated to obtain more crystals then any soluble impurity will also solidify and the purity of the product will decrease. Other factors may include insufficient refluxing so not all the benzenecarboxamide is hydrolysed – reflux for longer (until no more ammonia is detected); some benzenecarboxylic acid remains in (alkaline) solution – add more acid; some product will be held in the pores of the filter papers.)

- The melting point range can be collected from each group and compared. This can form the basis for discussing the possible reasons for impurities to be present.
  (Impurities: may include insufficient refluxing so some benzenecarboxamide still present; presence of impurity in benzenecarboxamide which behaves in a similar way to benzenecarboxylic acid.)

Typical results

mass benzenecarboxamide / g = 2.98

mass benzenecarboxylic acid / g = 2.39

% yield = (0.0196/0.0246) x 100 = 79.7%

mp / ºC = 119–121 ºC

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Information for technicians

Each learner will require:

	(a)	Eye protection
	(b)	1 x 250 cm^3 beaker (or larger)
	(c)	3 or 4 x 100 cm^3 beaker *
	(d)	1 x 25 cm^3 measuring cylinder
	(e)	1 x 50 cm^3 or 100 cm^3 pear-shaped or round-bottomed flask
	(f)	1 x Liebig condenser with plastic or rubber tubing to go to the tap and sink
	(g)	1 x stand and 2 x clamp
	(h)	1 x Bunsen burner
	(i)	1 x heat proof mat
	(j)	1 x tripod and gauze
	(k)	access to 1 x Buchner funnel and filter papers **
	(l)	access to 1 x Buchner (side-arm) flask **
	(m)	access to1 x filter pump (water pump) ** with tubing to fit onto the tap
	(n)	1 x sample bottle (or other small container suitable for weighing product)
	(o)	1 x thermometer reading to 250 ºC or 360 ºC
	(p)	1 x melting point tube (glass capillary tube)
	(q)	metal stirrer *** (see diagram C)
	(r)	1 x small piece of rubber tubing or rubber band
	(s)	1 x large watch glass or evaporating dish
[MH] [HH]	(t)	3.5 g benzene carboxamide (benzamide)
[C]	(u)	25 cm^3 2 mol dm^{-3} sodium hydroxide
[C]	(v)	10 cm^3 concentrated hydrochloric acid
[MH] [HH] [F]	(w)	5 cm^3 ethanol
	(x)	50 cm^3 distilled water
	(y)	ice
[F] [HH] [N] [MH]	(z)	40 cm^3 paraffin oil (liquid paraffin) ***
	(aa)	access to balance weighing to at least 1 decimal place
	(bb)	paper towel

Technician’s notes, *continued*

Additional Instructions

* One 100 cm^3 beaker may be replaced with a weighing boat, another with a small conical flask (about 100 cm^3 capacity).

** If this apparatus is not available then each group of learners will require 1 x filter funnel (stemless is preferable) and filter papers, 1 x conical flask.

*** If melting point apparatus is available then it should be used in place of the paraffin oil bath.

Equipment set-up

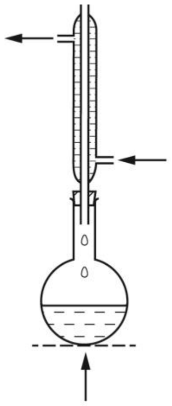

Diagram A
refluxing

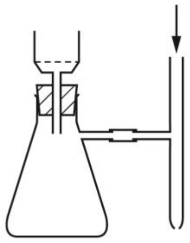

Diagram B
filtering under
reduced pressure

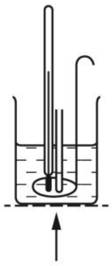

Diagram C
melting point
determination

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To hydrolyse an aromatic amide, purify the product, calculate a percentage yield and determine the melting point.

Method

Safety:

- Wear eye protection.
- benzenecarboxamide **[H]**
- 2 mol dm^{-3} sodium hydroxide **[C]**
- concentrated hydrochloric acid **[C]**
- ethanol **[F] [H]**
- product of reaction **[H]**

Hazard symbols

C = corrosive substance

F = highly flammable substance

H = harmful or irritating substance

Part A. Hydrolysis of the amide

1. Weigh a small beaker or weighing boat. Record the balance reading.

2. Add between 2.9 and 3.1 g of benzenecarboxamide (benzamide) and record the new balance reading.

3. Use a filter funnel to help transfer the benzenecarboxamide into a pear-shaped or round-bottomed flask.

4. Reweigh the small beaker or weighing boat with any residual benzenecarboxamide. Record the balance reading and the mass of benzenecarboxamide transferred to the flask.

5. Use a measuring cylinder to transfer 20 cm^3 of 2.0 mol dm^{-3} sodium hydroxide **[C]** into the flask with the benzenecarboxamide.

6. Clamp the flask above a Bunsen burner and attach a condenser vertically to the flask as shown in diagram A.

7. Run water through the condenser then start heating the flask **gently**. Test for any gas or vapour coming out of the top of the condenser with pieces of damp red and blue litmus paper.

8. Adjust the flame so that there is a steady drip-back of reactants into the flask from the condenser. (This technique is called refluxing.)

9. Reflux the contents of the flask for about 20 minutes then switch off the Bunsen burner and allow the apparatus to cool.

10. Tip the cooled contents of the flask into a 100 cm^3 beaker and use a dropper to add concentrated hydrochloric acid **[C]** until no further reaction is visible.

Worksheet, *continued*

11. Filter the solid product **[H]**. If possible filter the product under reduced pressure using a Buchner funnel, Buchner (side-arm) flask and a filter pump (attached to the cold tap). (See diagram B.)

12. When you set up the Buchner funnel, place one piece of filter paper (it should just fit the funnel over the grid) and dampen it with a little distilled water. Then add a second piece of the filter paper and dampen it again. The funnel is now ready for use. When you filter the product make sure that there is a good seal between the funnel and the flask. Do not turn off the tap operating the filter pump before removing the funnel or, at least, breaking the seal so that water does not enter the flask.

Part B: purifying the product [H]

13. Rinse the solid product with a few cm^3 of **cold** distilled water and then about 2 cm^3 of ethanol. If you are using a filter pump, suck air through the solid to help dry it.

14. Scrape the impure product into a small clean conical flask or beaker with a spatula. Place the container with the impure product in a hot water bath. (This may be a larger beaker heated over a Bunsen burner.)

15. Heat about 40 cm^3 of **distilled** water in a clean 100 cm^3 beaker until it is very hot.

16. Add the very hot **distilled** water in approximately 1 cm^3 portions to the impure product and stir to dissolve it. Use the minimum volume of very hot **distilled** water for the all solid to dissolve.

    If some solid appears to be insoluble in the hot distilled water then you will need to carry out hot filtration to remove the solid impurity. In this case you need to prevent the desired product from crystallising in the funnel. You can do this by running very hot water through the filter paper before filtering. This time the material you will need to keep is in the filtrate so check that there is no water in your filter flask. (Some product is likely to solidify in the cold filter flask.)

17. Cool the container with the hot solution by placing it in an ice bath until crystals form. (If you had to carry out hot filtration then this will be the filter flask.)

18. Filter off the crystals (preferably under reduced pressure) and rinse them with about 2 cm^3 of distilled water followed by 2 cm^3 of ethanol. Place the filter paper with the crystals on a large watch glass or evaporating dish. Allow the crystals to dry, either by leaving them overnight or by heating for a short time in an oven at about 70 ^{o}C.

19. Weigh a sample bottle (or clean weighing boat or small beaker) and record the mass. Carefully tip the (cooled) dry crystals into the weighed container and reweigh. Record the new mass and the mass of purified product.

Part C: checking the purity of the product [H] by melting point determination

20. Seal one end of a melting point tube (glass capillary tube) by rotating the end in the hot part of the Bunsen flame. Let the glass cool.

Worksheet, *continued*

21. Press the open end of the tube carefully into the dry crystals of product. Hold the tube vertically with the sealed end down and tap the outside of the top of the tube so the crystals fall to the bottom. Keep adding crystals until the depth is about 1 cm.

22. Attach the melting point tube to a thermometer reading to either 250 °C or 360 °C with a small piece of rubber tubing or rubber band. The end of the melting point tube containing the crystals should be level with the bulb of the thermometer.

23. Clamp the thermometer in a 100 cm^3 beaker about half full of paraffin oil. Make sure that the open end of your melting point tube is above the oil level. (See diagram C.)

24. Heat the oil and use a stirrer to ensure the temperature is approximately uniform. When the temperature reaches about 100 °C heat the oil gently so the temperature rises by about 2 °C per minute. (Keep stirring the oil.)

25. Note the temperature at which the crystals start to melt and the temperature when all the crystals have melted. This is the melting point range of your product.

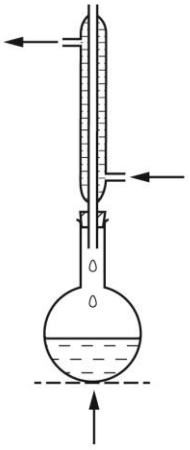

Diagram A
refluxing

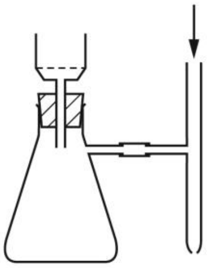

Diagram B
filtering under
reduced pressure

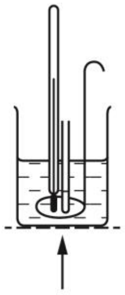

Diagram C
melting point
determination

Results

Record **all** your observations.

Draw a table for your results which includes the masses of benzenecarboxamide (benzamide) and benzenecarboxylic acid (benzoic acid).

Your recorded balance readings should be to a consistent number of decimal places.

Note the colours of reactant and product and also any change in the colours of the litmus papers.

Worksheet, *continued*

Interpretation and evaluation

For data required, refer to the Periodic Table.

1. The formula of benzenecarboxamide is $C_6H_5CONH_2$. Calculate the number of moles of benzenecarboxamide you placed in the flask.

2. Explain the observation you made whilst refluxing and testing for vapours.

3. Sodium hydroxide and benzenecarboxamide react in a 1 : 1 mole ratio.

   (i) Complete the equation for the reaction taking place during the reflux.

   $$C_6H_5CONH_2 + NaOH \rightarrow \qquad\qquad +$$

   (ii) Show by calculation that the sodium hydroxide is in excess

   (iii) Use ‘curly arrows’ to draw the mechanism for the hydrolysis of benzenecarboxamide.

4. A strong acid will displace a weaker one from its salt.
   Name, and give the formula of, the crystalline product of the reactions in step 10 of method A.

5. Calculate the theoretical yield of your crystalline product. (If you are not sure of its identity, ask your teacher for assistance.)
   Calculate the percentage yield of crystalline product you obtained.
   Suggest reasons why this yield is unlikely to be close to 100%.

6. Impurities in the product will reduce its melting point and will cause it to melt over a range of temperatures. This temperature range decreases as the purity of the product increases.
   The melting point of the product when pure is 122 °C.
   Comment on the purity of your crystalline product.

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Practical Booklet 11

Chemical properties of primary amines

Cambridge International AS & A Level Chemistry 9701

Version 2.0

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners' practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To compare and contrast the properties and reactions of ammonia, an alkyl amine and an aromatic amine.

Outcomes

Syllabus section 20.1 and 12.2(a) as well as experimental skills 2 and 3

Skills included in the practical

A Level skills	How learners develop the skills
Evaluation	analyse qualitative data to draw appropriate conclusions understand the significance of observations made in qualitative tests
Conclusions	make scientific explanations of the observations and data that they have described make predictions about the results of further experiments

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	use apparatus to collect an appropriate quality of observations, including subtle difference of colour and solubility
PDO layout	choose a suitable and clear method of presenting the results obtained
PDO recording	record observations to the same level of fine discrimination

Method

- **Learners must wear eye protection for these investigations.**

- Amines show basic properties because of the lone pair on the nitrogen atom in the $–NH_2$ group. The properties of the $-NH_2$ group are affected by the nature of the alkyl or aryl group attached to it. Some of the experiments carried out in this investigation will illustrate how the properties of the $–NH_2$ group are affected. The availability of the lone pair to cause basic properties may either be increased by an alkyl group or reduced by an aryl group. See experiments 1 and 2.

- The presence of an $-NH_2$ joined to a benzene ring enhances the reactivity of the benzene ring towards attack by electrophiles, such as bromine. This is because of the delocalisation of the lone pair on nitrogen into the ring. See experiment 3. Benzene itself does not react with aqueous bromine.

- The $–NH_2$ group can be substituted to form a diazonium salt, with functional group $–N_2Cl$. Diazonium salts derived from alkyl amines are very unstable, but aryl diazonium salts can be prepared and stored at a low temperature of about 5 °C. See experiment 4.

- Aryl diazonium salts can be used in coupling reactions to prepare brightly coloured dyes, called azo dyes. See experiment 5. The discovery of these dyes revolutionised the clothing industry. Certain azo dyes can be used as indicators (e.g. methyl orange) because their colour changes according to whether or not a proton attaches to the molecule

- This experiment is suitable for learners to work in pairs or small groups.

- Learners should become familiar with working safely with hazardous chemicals.

Results

- Learners should note the colours and solubilities of reactants and products.

- They should record their observations using correct terminology (e.g. ‘cloudy solution’ is not acceptable for precipitate/ppt).

Interpretation and evaluation

- The inductive effect of the alkyl group can be revised, and the overlap of the lone pair of electrons on the nitrogen atom with the delocalised system of the benzene ring can be introduced or revised.

- Discussion can take place on whether phenylamine is expected to be more or less basic than ethyl 4-aminobenzenecarboxylate (electron withdrawing effect of the carboxylate group).

- Complex formation can be revised. The availability of the nitrogen lone pair for forming a dative covalent/coordinate bond can be discussed. (Ethyl 4-aminobenzenecarboxylate may only form a complex with Cu^{2+} on heating.)

- Learners can be reminded that only chlorine and bromine (but not iodine) react with benzene and the reactions require a catalyst. The mechanism of the electrophilic substitution reaction can be revised. The reactivity of the benzene ring can be compared to that in phenol or phenylamine, in which the benzene ring is more reactive.

- The possible mechanisms for a reaction with bromine can be brainstormed or revised. The similarity of phenol and aminobenzene in activating the benzene ring can be introduced or revised. (The product of the electrophilic substitution reaction of ethyl 4-aminobenzenecarboxylate with bromine is ethyl 4-amino-3,5- dibromobenzenecarboxylate.)

- When nitrous acid is added to the alkyl amine, the following reaction occurs during which bubbling should be observed.

  $RNH_2 + HNO_2 \rightarrow ROH + H_2O + N_2$

  When the same reaction is carried out with the aryl amine at 5 °C, no nitrogen is given off, but the diazonium salt is produced instead. At higher temperatures the aryl amine behaves in the same way as the alkyl amine in this reaction.

- The stabilising effect of the ring preventing electrophilic substitution in benzene diazonium chloride can be discussed ($-N^+\equiv N$ the positive charge can be delocalised in the ring with the 2, 4 and 6 positions becoming $\delta+$). The reason why the atom closer to the benzene ring carries the positive charge can be discussed (due to dative bonding electrons).

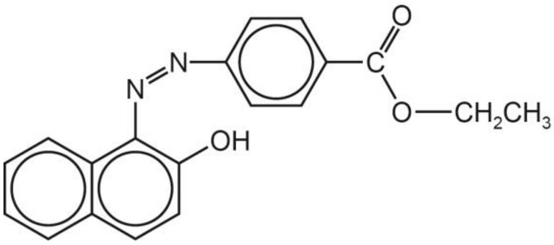
- Diazonium salts readily undergo nucleophilic substitution reactions, due to the positive charge on the nitrogen atom joined to the benzene ring.□□

  ⌬–$N^+{\equiv}N\ Cl^-$ + KI → ⌬–I + KCl + N_2

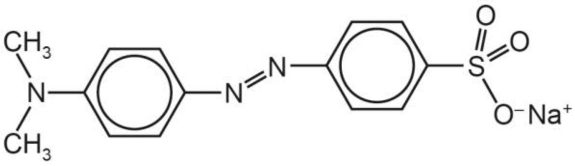
  ⌬–$N^+{\equiv}N$ + H_2O → ⌬–OH + H^+ + N_2

- $C_{10}H_7OH(s) + OH^-(aq) \rightarrow C_{10}H_7O^-(aq) + H_2O(l)$

  The difference in acid behaviour of alcohols, phenols and carboxylic acids can be revised.



ethyl 4-[2-(2-hydroxy-1-naphthyl)hydrazine]
benzenecarboxylate
(systematic names not needed)



methyl orange
sodium 4-[(4-dimethylamine) phenyldiazenyl]
benzenesulfonate

- The reasons why chemicals are coloured can be discussed and colour in complexes may be revised. The red form (acidic) of methyl orange absorbs light of a shorter wavelength (more energetic transition). (The anion is protonated at the –N=N– group allowing a different pattern of delocalisation. The reason why conjugated and delocalised organic systems are chromophores is not required for this syllabus.)

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Information for technicians

Each learner will require:

	(a)	Eye protection
	(b)	Gloves (nitrile) may be worn
	(c)	2 x 250 cm^3 beaker
	(d)	1 x 25 cm^3 measuring cylinder
	(e)	1 x 100 cm^3 beaker
	(f)	8 x test-tube (these may be rinsed and re-used but more tubes should be available)
	(g)	3 x boiling tube
	(h)	1 x Bunsen burner (placed away from the ethylamine or 1-aminobutane)
	(i)	1 x heat proof mat
	(j)	1 x tripod and gauze
	(k)	1 x thermometer (–10 ºC to +110 ºC at 1 ºC)
	(l)	4 x teat/dropping pipette
	(m)	1 x glass rod
	(n)	3 x weighing boat or container for weighing small masses of solid
	(o)	10 cm^3 2.0 mol dm^{-3} ammonia
[F] [C]	(p)	10 cm^3 aqueous ethylamine (70% solution) **or** 1-aminobutane (butylamine) supplied in a stoppered test-tube **or** in the fume cupboard
[MH]	(q)	1 g ethyl 4-aminobenzenecarboxylate (benzocaine)
[N]	(r)	5 cm^3 0.1 mol dm^{-3} copper(II) sulfate
[MH]	(s)	10 cm^3 aqueous bromine (0.02 mol dm^{-3} is a suitable concentration)
[MH]	(t)	15 cm^3 2.0 mol dm^{-3} hydrochloric acid
[O] [T] [N]	(u)	2 g sodium nitrite
[MH]	(v)	0.5 g naphthalen-2-ol (β-naphthol)
[C]	(w)	5 cm^3 2.0 mol dm^{-3} sodium hydroxide
	(x)	15 cm^3 distilled water
	(y)	ice
	(z)	access to balance weighing to at least 1 dp
	(aa)	pen suitable for marking glassware
	(bb)	paper towel

Technician's notes, *continued*

Additional Instructions

A simple ice bath is made by placing (crushed) ice in a 250 cm^3 beaker. If a freezing mixture is required then sodium chloride salt is added to the ice.

Good ventilation of the laboratory or use of a fume cupboard is needed.

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 **GHS07 (*moderate hazard* MH)**	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To compare and contrast the properties and reactions of ammonia, an alkyl amine and an aromatic amine.

Method

Safety:

- Wear eye protection.
- Do not work close to the Bunsen flame in any of these experiments.
- Gloves (nitrile) may be worn.
- aqueous ethylamine or 1-aminobutane **[F] [H]**
- ethyl 4-aminobenzenecarboxylate **[H]**
- aqueous bromine **[H]**
- 2.0 mol dm^{-3} hydrochloric acid **[H]**
- sodium nitrite **[O] [T] [N]**
- naphthalen-2-ol **[H]**
- 2.0 mol dm^{-3} sodium hydroxide **[C]**

Hazard symbols

C = corrosive substance	**F** = highly flammable substance
H = harmful or irritating substance	**O** = oxidising substance
N = harmful to the environment	**T** = toxic substance

- Approximately half-fill a 250 cm^3 beaker with (tap) water and heat it over a Bunsen flame. This will be your hot water bath. When the water starts boiling switch off the Bunsen burner.

Experiment 1: solubility and pH

1. Test a 1 cm depth of aqueous solutions of ammonia and ethylamine (or 1-aminobutane) in separate test-tubes with one drop of universal indicator solution. If universal indicator solution is not available, use a glass rod to transfer 1 drop of solution onto narrow range universal indicator papers.

2. Transfer a **small** spatula measure of ethyl 4-aminobenzenecarboxylate (benzocaine) into a test-tube. Add a 2 cm depth of distilled water and heat the mixture in your hot water bath. Carefully pour about a 1 cm depth of the solution formed into a test-tube and add one drop of universal indicator solution. Cool the remaining solution.

Worksheet, *continued*

Experiment 2: reaction with copper(II) ions .

1. To a 1 cm depth of 0.1 mol dm^{-3} copper(II) sulfate in a test-tube add aqueous ammonia drop by drop with shaking until no further change takes place.

2. Repeat step 1 but use aqueous ethylamine (or 1-aminobutane) in place of aqueous ammonia.

3. To a 1 cm depth of 0.1 mol dm^{-3} copper(II) sulfate in a test-tube add a **small** spatula measure of ethyl 4-aminobenzenecarboxylate and heat the mixture in your hot water bath (re-heat if necessary). Then allow the contents of the tube to cool.

Experiment 3: reaction with aqueous bromine [H]

1. To a 1 cm depth of aqueous ammonia in a test-tube add a 2 cm depth of hydrochloric acid followed by a 2 cm depth of aqueous bromine. Carefully shake the contents of the tube.

2. Repeat step 1 but use aqueous ethylamine (or 1-aminobutane) in place of aqueous ammonia.

3. To a **small** spatula measure of ethyl 4-aminobenzenecarboxylate in a test-tube add a 2 cm depth of hydrochloric acid. Warm the tube in your hot water bath (re-heat if necessary) to dissolve the solid then add a 2 cm depth of aqueous bromine.

Experiment 4: reaction with aqueous sodium nitrite [O] [T] [N]

Gloves may be worn. Switch off the Bunsen burner while carrying out this test

1. Use a measuring cylinder to transfer 10 cm^3 of distilled water into a boiling tube. Add 2 g of sodium nitrite and shake the tube carefully to form a solution. Label the tube $NaNO_2$. Place the tube in an ice bath or freezing mixture.

2. To a 1 cm depth of aqueous ammonia in a test-tube add a 2 cm depth of dilute hydrochloric acid. Label the tube NH_3. Use a dropper to transfer about a 1 cm depth of aqueous sodium nitrite into the tube. Let the test-tube stand at room temperature. If no change is observed, place the tube in the hot water bath.

3. Repeat step 2 but use aqueous ethylamine (or 1-aminobutane) in place of aqueous ammonia. Label the tube RNH_2.

4. Dissolve 0.5 g of ethyl 4-aminobenzenecarboxylate in a 2 cm depth of dilute hydrochloric acid in a boiling tube. Label the tube ϕNH_2 and place it in the ice bath or freezing mixture. Half of this solution will be needed for experiment 5.

5. Use a dropper to transfer a 1 cm depth of solution from step 4 into a test-tube. Add about a 1 cm depth of aqueous sodium nitrite into the tube. Let the test-tube stand at room temperature. If no change is observed, place the tube in the hot water bath.

Worksheet, *continued*

Experiment 5: a diazonium coupling reaction

Gloves may be worn.

1. Dissolve 0.5 g naphthalen-2-ol **[H]** in 5 cm^3 aqueous sodium hydroxide **[C]** in a boiling tube. Place the tube in the ice bath.

2. Add the remaining cold acidified solution of ethyl 4-aminobenzenecarboxylate to the tube containing the aqueous sodium nitrite. Keep the tube in the ice bath.

3. When the temperature of the solution from step 1 is $\leq 5\,^{o}C$ transfer all of it into a 100 cm^3 beaker. Add the mixture from step 2 to the contents of the beaker with stirring. Record what you observe.

   Dispose of the contents **[H]** of the beaker carefully down the sink with plenty of water. This is an azo dye and most azo dyes are irritant, harmful or toxic.

Results

Record **all** your observations.

Interpretation and evaluation

1. List ammonia, ethylamine (or 1-aminobutane) and ethyl 4-aminobenzenecarboxylate in order of their basic strength.

   (i) Explain in terms of structure why they are in this order.

   (ii) Would you expect phenylamine to be a stronger or weaker base than
   ethyl 4-aminobenzenecarboxylate? Explain your answer.

2. Explain fully your observations in experiment 2.

3. Draw the displayed formula of ethyl 4-aminobenzenecarboxylate, $H_2NC_6H_4COOC_2H_5$.
   The product of the reaction between aqueous bromine and ethyl 4-aminobenzenecarboxylate is a dibromo- compound.

   Indicate on your displayed formula where you would expect the bromine atoms to be found.

   Suggest a mechanism for the reaction between aqueous bromine and ethyl 4-aminobenzenecarboxylate.

4. When sodium nitrite reacts with hydrochloric or sulfuric acid the sodium salt of the acid and nitrous (nitric(III)) acid is formed.

   $$NaNO_2(aq) + HCl(aq) \rightarrow NaCl(aq) + HNO_2(aq)$$

   (If the solutions are cold and sufficiently concentrated the resulting solution will be blue.)

Worksheet, *continued*

Nitrous acid can react with amines to form a gas.

(i) Suggest the identities of the products of the reaction between RNH_2 and HNO_2.

$$RNH_2 + HNO_2 \rightarrow$$

(ii) When the solutions are kept below 5 °C any diazonium compound formed is more stable.

Suggest a reason for the lower stability of the diazonium compound formed by an alkyl amine, RNH_2, compared with that formed by an aromatic amine, ⌬NH_2.

Aromatic diazonium compounds are useful in organic syntheses as the diazo group can be replaced by a halogen atom, a nitrile group or a hydrogen atom.

(iii) Using ⌬ to represent a phenyl (C_6H_5–) or substituted phenyl group, write an equation for the likely reaction between an aromatic diazonium chloride and potassium iodide.

5. Naphthalen-2-ol, $C_{10}H_7OH$, reacts in a similar way to phenol with aqueous sodium hydroxide.

(i) Write the ionic equation for the reaction between naphthalen-2-ol and sodium hydroxide. Include state symbols.

(ii) What type of behaviour is naphthalen-2-ol showing in this reaction?

(iii) In the coupling reaction with the diazonium compound (formed in step 2 of experiment 5) the 1 position of naphthalen-2-ol is used. Using to represen⌬e substituted phenyl group, –$C_6H_4COOC_2H_5$, draw the displayed formula of the diazonium compound formed in step 2.

(iv) Using ⌬ to represent the substituted phenyl group, write the equation for the reaction that occurs when the diazonium compound is warmed in aqueous solution.

(v) Draw the structure of the azo dye formed in step 3.

(vi) When 4-aminobenzenesulfonic acid (sulfanilic acid), $H_2NC_6H_4SO_3H$, reacts with nitrous acid and an alkaline solution of naphthalen-2-ol (instead of ethyl 4-aminobenzenecarboxylate) then a water soluble azo dye is formed, called orange II.

If N,N-dimethyphenylamine, $C_6H_5N(CH_3)_2$ (instead of naphthalen-2-ol) is added to 4-aminosulfonic acid, methyl orange indicator is the product.

Draw the structure of methyl orange, which is another azo dye.

(vii) Methyl orange is red in acidic solution and yellow in alkaline solution. Which form absorbs energy of shorter wavelength?

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Introduction

Practical work is an essential part of science. Scientists use evidence gained from prior observations and experiments to build models and theories. Their predictions are tested with practical work to check that they are consistent with the behaviour of the real world. Learners who are well trained and experienced in practical skills will be more confident in their own abilities. The skills developed through practical work provide a good foundation for those wishing to pursue science further, as well as for those entering employment or a non-science career.

The science syllabuses address practical skills that contribute to the overall understanding of scientific methodology. Learners should be able to:

- plan experiments and investigations
- collect, record and present observations, measurements and estimates
- analyse and interpret data to reach conclusions
- evaluate methods and quality of data, and suggest improvements.

The practical skills established at AS Level are extended further in the full A Level. Learners will need to have practised basic skills from the AS Level experiments before using these skills to tackle the more demanding A Level exercises. Although A Level practical skills are assessed by a timetabled written paper, the best preparation for this paper is through extensive hands-on experience in the laboratory.

The example experiments suggested here can form the basis of a well-structured scheme of practical work for the teaching of AS and A Level science. The experiments have been carefully selected to reinforce theory and to develop learners’ practical skills. The syllabus, scheme of work and past papers also provide a useful guide to the type of practical skills that learners might be expected to develop further. About 20% of teaching time should be allocated to practical work (not including the time spent observing teacher demonstrations), so this set of experiments provides only the starting point for a much more extensive scheme of practical work.

Guidance for teachers

Aim

To determine a partition coefficient for iodine between two immiscible solvents and to investigate separation of suitable mixtures by chromatography and electrophoresis.

Outcomes

Syllabus section 7.3, 22.1(a), 20.3(c) and 21.4(d), and a link with 22.2, 22.3, 22.4 and 22.5 as well as experimental skills 1, 2, 3 and 4

Skills included in the practical

A Level skills	How learners develop the skills
Planning	identify a safe and efficient procedure that when followed would lead to a reliable result describe all the steps necessary to carry out the procedure show an understanding of how and why the procedure suggested will be effective
Analysis	calculation from quantitative data obtained
Evaluation	suggest modifications to a procedure that will improve its accuracy estimate the uncertainty in a quantitative experiment and express the uncertainty as a percentage error
Conclusions	carry out appropriate calculations from data obtained make scientific explanations of data obtained

This practical provides an opportunity to build on essential skills introduced at AS Level.

AS Level skills	How learners develop the skills
MMO collection	make measurements using pipettes, burettes and other common laboratory apparatus
MMO quality	obtain results that are close to those of an experienced chemist
MMO decisions	identify where repeated readings are appropriate
PDO recording	record quantitative results appropriately in a table, with appropriate headings and units
ACE conclusions	draw conclusions from interpretations of observations, data and calculated values

Method A, B and C

- **Learners must wear eye protection for these investigations.**
- The laboratory should be well ventilated**.**
- Method A provides an opportunity for learners to revise the various skills required to carry out a titration accurately. In the experiment they titrate an aqueous and organic layer, both of which contain iodine, in order to determine the partition coefficient of iodine between these two solvents.
- Learners should carry out this experiment individually. The teacher should also carry out the experiment at the same time, so that the learners' accuracy can be assessed by comparing their value for the partition coefficient to the teacher's value.
- In a titration involving iodine and sodium thiosulfate, it is normal to not add the starch indicator at the start of the titration. It is better to add the starch when most of the iodine has reacted, shown by the solution becoming yellow. Then the starch is added. By doing this, the blue-black colour (caused by an iodine-starch complex) decomposes more easily, making the end-point sharper to observe.
- Since iodine is much less soluble in water than in *Volasil 244*, the organic solvent chosen, the sodium thiosulfate solution used to titrate the iodine in the organic layer must be diluted before the aqueous layer of iodine is titrated.
- It is important that the aqueous layer does not become saturated with iodine. If this happens there is no longer an equilibrium of iodine partitioning between the two solvents. This error is avoided by limiting the mass of iodine used.
- The experiments involving chromatography and electrophoresis (Methods B and C) are suitable for learners to work in pairs, in which case it may be helpful to have a 'circus' of experiments. If time is short, learners could work in groups, so that each learner or pair of learners should carry out one of the experiments described. The learners should describe to others in the group how they used the apparatus and what results they obtained from each experiment.
- Learners should become familiar with common techniques used in organic extraction and in some forms of analysis and they should gain confidence in handling hazardous materials safely.

Results

- Learners should record their burette readings correct to 0.05 cm^3.
- They should record the distances travelled by solvent front and spots to the nearest 0.1 cm.
- They should note any differences in the order of spots when using different solvents or different buffer solutions.

Interpretation and evaluation A

- The values of *K* obtained by the learners can be collected and reasons for discrepancies discussed (equilibrium not reached; inaccurate end-point; inaccurate dilution; too much starch added so some I_2 locked).

- The reaction mechanism for the reduction of nitrobenzene can be revised.

- Adding strong base to liberate a weaker base from its salt can be compared with the similar acid displacement reaction.

- The reasons for using several small portions of organic solvent (rather than one large portion) to extract the organic compound can be discussed and this may provide an introduction to partition chromatography.

  [5(i) $x/50 \div (4-x)/50 = 30$; $x = 3.87$ g]

  [5(ii) $y/25 \div (4-y)/50 = 30$; $y = 3.75$ g; mass remaining in aqueous solution = 0.25 g;
  $z/25 \div (0.25-z)/50 = 30$; $z = 0.23$ g; total extracted = 3.98 g]

Typical results

Titre: I_2(org) with 0.10 mol dm^{-3} $Na_2S_2O_3$ = 14.15 cm^3

Titre: I_2(aq) with 0.010 mol dm^{-3} $Na_2S_2O_3$ = 5.90 cm^3

$[I_2(org)] / [I_2(aq)]$ = titre (org) / (titre (aq) ÷ 10) = 14.15 / 0.59 = 24.0

Interpretation and evaluation B

- The calculation to find R_f values can be introduced or revised.

- The increase in mass of solute extracted by successive small portions of solvent can be linked to paper chromatography as a process happening very many times as fresh mobile phase solvent passes over the stationary phase.

- The differences in polarity of mobile phase solvents and relative solubilities of solutes in them can be discussed. The suitability of the learners' suggestions for further experiments can be discussed. (Keep everything the same except for the solvent. Use solvents such as ethanol (polar) and petroleum spirit / cyclohexane / *Volasil 244* (non-polar) and compare results.)

- Discussion about greater separation may include smaller pore size (use chromatography grade paper), different polarity of solvent / mixture of solvents and length of strips.

- Differences in speed of solvent front, separation of components and 'tailing' of spots may be discussed for the two types of chromatography (paper and TLC).

- Learners could discuss which technique(s) are suitable to analyse the food colour and what apparatus would be needed. The use of reference spots of pure tartrazine yellow and curcumin and comparison of R_f values with the spot from the orange squash can be discussed. The possibility of cutting out each spot formed from the orange squash, extracting it from the paper / adsorbent (TLC) with a suitable solvent and then analysing it can be mentioned.

- The techniques of analysis and the information each will give may be introduced or revised (mass spec: M_r and m/z for fragments; IR: groups; nmr: number and type of environments.)

Interpretation and evaluation C

- The structures of the α-amino acids used can be discussed and their systematic names can be suggested. (–R groups for gly, lys, asp and glu respectively: –H, $-(CH_2)_4NH_2$, $-CH_2COOH$ and $-(CH_2)_2COOH$)

- The formation of zwitterions can be introduced or revised and the reasons for the different isoelectric points / charges in different pH buffers can be discussed. The direction of travel in an electric field can be discussed.

- Learners might suggest the effect of the size of the ion on the speed of migration (aspartic acid is smaller).

- Other factors affecting the speed of migration can be brainstormed (buffer pH leading to nature of charge on ion, size of charge (lysine +2 in pH 4), shape of ion (–R group with benzene ring or chain), pore size of medium (e.g. amount of cross linking of polyacrylamide)).

- The hydrolysis of proteins can be introduced. The use of gel electrophoresis (often polyacrylamide for peptides, agarose for nucleic acids) in identifying peptides and nucleic acids can be discussed (e.g. genetic fingerprinting).

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Information for technicians

Method A

Each learner will require:

(a) Eye protection

(b) 1 x 100 cm^3 beaker (or larger)

(c) 1 x separating funnel (tap funnel)

(d) 2 x 50 cm^3 measuring cylinder

(e) 1 x burette

(f) 1 x stand and burette clamp

(g) 1 x filter funnel (for filling burette)

(h) 3 x 25 cm^3 pipette

(i) 1 x pipette filler

(j) 1 x 250 cm^3 volumetric (graduated) flask

(k) 2 x 150 cm^3 or 250 cm^3 conical flask

(l) 1 x white tile

(m) 1 x spatula

(n) 1 x weighing boat (or similar)

(o) 1 x stoppered bottle with tightly fitting stopper or bung (≥ 100 cm^3 capacity)

[MH] [N] (p) iodine

[MH] (q) 40 cm^3 *Volasil 244* (or other non polar solvent)

(r) 50 cm^3 0.10 mol dm^{-3} sodium thiosulfate

(s) 2 cm^3 starch indicator

(t) 50 cm^3 distilled water

(u) access to balance weighing to at least 1 dp

(v) paper towel

Technician's notes, *continued*

Method B

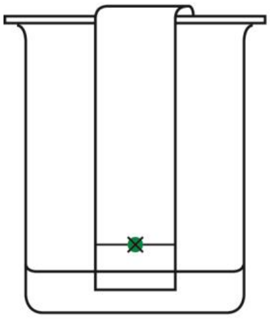
Each learner will require:

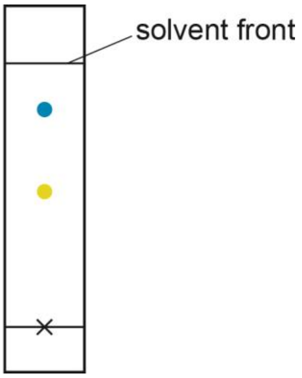
(a) Eye protection
(b) 2 x 100 cm^3 beaker
(c) 2 x chromatography paper or filter paper
(d) 2 x thin layer chromatography plate
(e) 2 x glass rod
(f) 2 x watch glass or plastic wrap (cling film) (to cover beaker)
(g) 1 x melting point tube (glass capillary tube)
(h) access to scissors, pencil and ruler
(i) 1 cm^3 screened methyl orange indicator (or mix of other pH indicators)
[F] [MH] (j) 25 cm^3 propanone
(k) 25 cm^3 distilled water
(l) mineral wool (approx 1 cm cube)
(m) paper towel

Additional Instructions

Optional experiment: glass tube approx. 2 cm diameter, 20 cm length fitted with rubber tubing and gate clip at one end; stand and clamp; aluminium oxide (fine powder); ethanol **[F] [H]**; mineral wool.

Equipment set-up





Technician’s notes, *continued*

Method C

Each learner will require:

(a) Eye protection
(b) 3 x chromatography paper or (fine pore) filter paper
(c) 3 x microscope slide
(d) 3 x test-tube
(e) 3 x melting point tube (glass capillary tube)
(f) 2 x crocodile clips
(g) 1 x 100 V d.c. supply
(h) 2 x connecting wires
(i) 1 x large beaker or tub to protect the slide and crocodile clips
(j) access to scissors, pencil and ruler
(k) glycine – small amount only
(l) lysine – small amount only
(m) aspartic acid – small amount only
(n) glutamic acid – small amount only
(o) 5 cm^3 pH 6 buffer solution
(p) 5 cm^3 pH 4 buffer solution
(q) 5 cm^3 pH 9 buffer solution
[MH] (r) ninhydrin spray
(s) paper towel

Additional Instructions

If gel electrophoresis is available then this would be preferable to paper electrophoresis. Good ventilation of the laboratory or use of a fume cupboard is needed.

Technician’s notes, *continued*

Hazard symbols

 GHS02 (*flammable* **F**)	 GHS03 (*oxidising* **O**)	 GHS05 (*corrosive* **C**)
 GHS06 (*acutely toxic* **T**)	 GHS07 (*moderate hazard* MH)	 GHS08 (*health hazard* **HH**)
	 GHS09 (*hazardous to the aquatic environment* **N**)	

Worksheet

Aim

To determine a partition coefficient for iodine between two immiscible solvents and to investigate separation of suitable mixtures by chromatography and electrophoresis.

Method

Safety:

- Wear eye protection.
- iodine **[H]**
- *Volasil 244* **[H]**
- propanone **[F] [H]**
- ethanol **[F] [H]**
- ninhydrin spray **[H]**

Hazard symbols

H = harmful or irritating substance	**F** = highly flammable substance

Experiment A

The laboratory must be well ventilated.

1. Use a measuring cylinder to transfer 35 cm^3 distilled water into a bottle which has a correctly fitting stopper or bung.

2. Use a second measuring cylinder to transfer 35 cm^3 *Volasil 244* (or other suitable organic solvent) into the same bottle.

3. Weigh out approximately 1 g of iodine and carefully add the solid to the mixture in the bottle.

4. Stopper the bottle and shake it for several minutes to dissolve the iodine.

5. Leave the bottle until the colour intensity in each layer does not change.

6. Transfer both liquid layers from the bottle into a separating funnel. Stopper the funnel and clamp it vertically until you are ready to run off one of the layers.

Titrating the organic layer

You will only be able to carry out **one** titration for each solution so care is needed when approaching the end-points.

7. Wash the burette with a little 0.10 mol dm^{-3} sodium thiosulfate and discard the washings. Then fill the burette. Make sure that the region under the tap is full.

8. Take a reading at eye level of the position of the bottom of the meniscus on the scale. Record the initial burette reading to the nearest 0.05 cm^3.

Worksheet, *continued*

9. Remove the stopper from the separating funnel. Run off the lower (aqueous) layer into a 100 cm^3 beaker. Close the tap as the interface enters it. The solution left in the separating funnel should now have no aqueous layer present. You will use the aqueous solution of iodine in step 18.

10. Using a pipette filler wash a 25 cm^3 pipette with a **small** portion of your solution of iodine in the organic solvent (which is left in the separating funnel) and discard the washings. Pipette 25.0 cm^3 of this solution into a conical flask. Touch the bottom of the pipette against the wall of the flask or onto the surface of the solution to deliver the correct volume. (Put the stopper back in the separating funnel to trap the vapour from any remaining organic solvent.)

11. Place the conical flask on the white tile under the burette. Add about 10 cm^3 of distilled water and swirl the flask to allow some iodine to enter the aqueous layer.

12. Run sodium thiosulfate from the burette in small portions and swirl the flask between additions. As soon as the iodine colour fades to yellow add a few drops of starch indicator so the mixture turns blue-black.

13. Add sodium thiosulfate, a few drops at a time, from the burette until the solution **just** becomes colourless.

14. Measure and record the new volume of the sodium thiosulfate in the burette. Calculate and record the volume of the 0.10 mol dm^{-3} sodium thiosulfate needed to react with the iodine in the organic solvent.

15. Pour the contents of the conical flask into an organic residues bottle together with any remaining organic solvent from the separating funnel. Wash the conical flask with water and discard the washings.

Titrating the aqueous layer

16. Firstly you will need to dilute the solution of sodium thiosulfate. Using a pipette filler wash a second 25 cm^3 pipette with a little of the 0.10 mol dm^{-3} sodium thiosulfate and discard the washings. Pipette 25.0 cm^3 of this solution into a 250 cm^3 volumetric flask and add distilled water up to the mark. Mix the diluted solution thoroughly before use.

17. Rinse the burette with water and then rinse with your diluted sodium thiosulfate. Then fill the burette with this solution and record the initial burette reading to the nearest 0.05 cm^3.

18. Wash a third 25 cm^3 pipette with a **small** portion of your solution of aqueous iodine (in the beaker) and discard it. Transfer 25.0 cm^3 of this solution into a second conical flask and place the flask on the white tile.

19. Repeat steps 12 and 13.

20. Record your final burette reading and discard the contents of the conical flask. Record the volume of your diluted sodium thiosulfate needed to react with the iodine in the aqueous layer.

Worksheet, *continued*

Results – experiment A

Record **all** your observations.

Record your burette readings correct to 0.05 cm^3.

Interpretation and evaluation – experiment A

1. Use your titre and the original concentration of the sodium thiosulfate solution to calculate the number of moles of iodine dissolved in 25.0 cm^3 of the organic solution.

2. Use your titre, the original concentration of the sodium thiosulfate and the dilution factor to calculate the number of moles of iodine dissolved in 25.0 cm^3 of the aqueous solution.

3. The iodine is in the same molecular state in the two solvents.
   The partition coefficient, *K*, is given by

   $$K_{ow} = \frac{[\mathrm{I2(org)}]}{[\mathrm{I2(aq)}]}$$

   Calculate the partition coefficient for iodine between your two solvents at the temperature of your laboratory.

4. What are the errors in single readings for each piece of apparatus that you have used?
   Estimate the maximum percentage error in the value of the partition coefficient you have calculated.

5. The partition of solute between two immiscible solvents is used in extracting organic compounds from an aqueous solution which contains inorganic compounds. For example, one of the stages in preparing phenylamine from nitrobenzene is an ether extraction.

   Nitrobenzene is refluxed with tin and concentrated hydrochloric acid. The salt $C_6H_5NH_3^+Cl^-$ (aq) is formed. What is added to the mixture to displace the phenylamine from its salt?

   To extract the phenylamine from the ionic products of the reaction, the mixture is shaken with ether (ethoxyethane), an organic solvent which is immiscible with water.
   This process is called ether extraction.

   Assume that the partition coefficient for phenylamine between ethoxyethane and water, K_{ew} = 30 at the temperature of the laboratory. Assume there is 4 g of phenylamine present in 50 cm^3 of aqueous solution.

   (i) What mass of phenylamine will be found in the organic layer if an equal volume of ethoxyethane is added to the aqueous solution and the distribution of phenylamine between the two layers is allowed to reach equilibrium?

   (ii) What mass of phenylamine will be extracted if the same volume of ethoxyethane is used but in two successive extractions? That is, 25 cm^3 of ethoxyethane is used initially and then a further 25 cm^3 is shaken with the aqueous layer to remove more of the phenylamine.

Worksheet, *continued*

Experiment B: chromatography

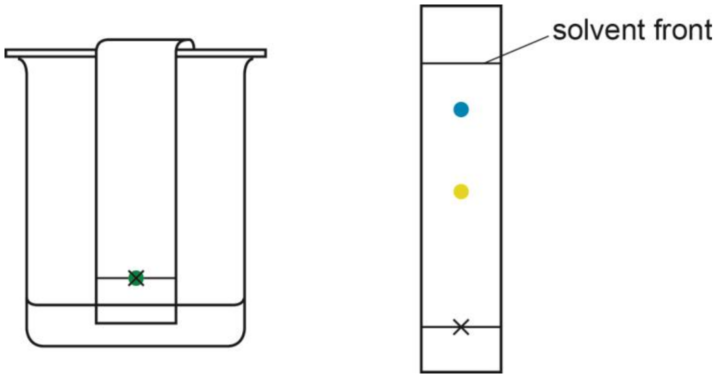
1: paper chromatography

1. Cut two strips of filter paper approximately 2 x 10 cm. Draw a pencil line about 1.5 cm from the bottom of each strip and parallel to the base. Put a pencil cross at the centre of the line.

2. Set up two beakers, one containing about 1 cm depth of water and the other containing about 1 cm depth of propanone. Cover the tops of the beakers with plastic film (or a watch glass) so the vapour in the beaker becomes saturated with solvent.

3. Dip the end of a melting point tube (glass capillary tube) into screened methyl orange indicator (or a mix of pH indicators) and pick up about a 2 mm depth of solution. Touch the tube onto the pencil cross on one strip of filter paper. Repeat the process for the second strip.

4. Fasten the top of each filter paper strip to a glass rod or a wooden spill so that the bottom of the paper enters the solvent in the beaker but the coloured spot will be above the liquid level (see diagram.)



5. Cover the tops of the beakers with plastic film and leave them until the solvent level is close to the fixing point of the strip. (The two solvents are unlikely to travel upwards at the same rate.)

6. Remove the filter paper strips from the beakers and use a pencil to mark the height of each solvent front. Let the paper dry then measure the distance from the base pencil line to (a) the solvent front, (b) the coloured spots.

Worksheet, *continued*

2: thin layer chromatography

1. Set up two beakers to act as chromatography tanks as in experiment 1.

2. Draw a pencil line across the two TLC plates (as experiment 1).

3. Spot the same coloured substance onto a pencil cross on the line.

4. Place the plates in the two beakers and cover both beakers.

5. Remove the plates when the solvent front nears the top of the plate and mark the position of the front in pencil.

6. Measure the distances travelled by the solvent fronts and the coloured spots.

Extension

These experiments may be carried out with the colour from dark green leaves (chlorophyll). Use a pestle to grind up dark green leaves with sand and about 5 cm^3 propanone in a mortar. Extract the colour in a non-polar solvent such as 5 cm^3 petroleum spirit **[F] [H]** using a separating funnel. A suitable mobile phase (solvent) is a 3:17 by volume mixture of propanone and petroleum spirit. If the colour of the spot is not dark enough, dry the paper / plate then add a second portion at the same position. Repeat as necessary. If the resulting colour or position of the different spots is not clear then shine a UV lamp onto the paper / plate.

Other suitable substances include coloured inks or the colouring extracted from the coating of chocolates or sweets, such as Smarties, M&Ms and Skittles. (A wider strip of paper should be used and the different colours spotted onto separate pencil crosses on the pencil line. The order of the colours applied should be recorded. It is then possible to see whether the same dyes are used in more than one ink or sweet.)

3: column chromatography

1. Mix alumina (aluminium oxide) with ethanol to make a slurry (like thin mud).

2. Set up a tube of approximately 2 cm diameter and 20 cm length with rubber tubing and a gate clip at one end. Place a small ball of mineral wool at the bottom end of the tube. (Part of a broken burette is suitable for this experiment, especially if the tap is still intact.)

3. Clamp the tube vertically with the mineral wool at the bottom. Gradually fill the tube with the slurry, tapping it to remove any air bubbles and with the gate clip open for excess ethanol to run off into a beaker to be used again. Leave a 1 – 2 cm gap at the top of the column.

4. Use a dropper to place about 5 drops of screened methyl orange indicator on the top of the alumina column without disturbing the surface and allow them to soak in.

Worksheet, *continued*

5. Add a thin layer of sand to the top of the alumina to protect it. Then add ethanol so there is always at least 0.5 cm depth of liquid above the sand. Keep the tap open.

6. Collect and re-use the ethanol that leaves the column without any colour. Collect and keep ethanol containing each of the colours. (There should be a complete separation of the two components of screened methyl orange.)

Results

Record **all** your observations.

Record the distances travelled by the solvent front and spots to the nearest 0.1 cm.

Note any differences in the order of spots when using different solvents or different buffer solutions.

Interpretation and evaluation

Paper chromatography involves partition coefficients.

The stationary phase is the water which is naturally adsorbed onto the paper fibres and which is immiscible with any mobile phase solvent (including water).

In a mixture, the solute with the greater solubility hence concentration in the mobile phase will travel further than one of lower solubility in that phase.

1. Calculate the retardation factor, R_f, values for each spot in the different solvents used in the (ascending) paper chromatography experiment.

2. Suggest why there are differences in R_f values for the same coloured spot in the different solvents. What further experiment could you carry out to test your suggestion?

3. With some mixtures the coloured components are not completely separated. Suggest how better separation might be achieved.

Thin layer, column and gas chromatography are all examples of adsorption chromatography. The components of a mixture are adsorbed onto the solid or liquid stationary phase. The weaker the adsorption the further the liquid mobile phase will move the component.

4. Calculate R_f values for each spot in the different solvents used in the TLC experiment.

5. Describe differences you noticed in the chromatograms formed using the two techniques, paper and thin layer chromatography.

Worksheet, *continued*

Some azo dyes are used to colour foods. Tartrazine yellow is one of these and has been banned as a food additive in some countries as it may be linked to hyperactivity in children. Some companies use β-carotene (in carrots and sweet potatoes) or curcumin (in turmeric) as a food colour instead.

6. A company producing orange squash is accused of using tartrazine yellow in their product to improve its colour, but the company claims that curcumin is being used.
   Design an experiment to determine which food colouring is being used by the company.

7. An advantage of carrying out column chromatography is that the separate components of the mixture may be run out of from the column and analysed. Suggest techniques that can be used in the analysis and what each technique can add to information about the component. (You may select techniques not available in your school laboratory.)

Experiment C: electrophoresis

Gloves may be worn to prevent fingerprints.

1. Use forceps to hold a piece of chromatography paper (or fine pore filter paper) and cut it to the size of a microscope slide. Place it on the slide and draw a pencil line across the middle (shorter length) and mark the ends positive and negative. Clip the (longer length) ends of the paper onto the glass slide with clean non-corroded crocodile clips.

2. Soak the paper with pH 6 buffer solution.

3. Mix together small amounts of the amino acids glycine, lysine, aspartic acid and glutamic acid in a test-tube. Add a small volume of the pH 6 buffer solution to make a concentrated solution. Use a melting point tube to transfer some of the solution to the line on the chromatography paper. Draw the tube along about 1 cm length of the line.

4. Attach wires to the crocodile clips. Cover the slide and crocodile clips with a large beaker or tub to protect the paper. Connect the wires to a 100 V d.c. supply (positive to +), switch on the supply and allow current to pass for at least 30 minutes. Switch off the d.c. supply and disconnect the crocodile clips. Let the paper dry.

5. Make sure that the laboratory is well ventilated before starting the next stage or use a fume cupboard when spraying and drying the paper.
   Spray the paper with ninhydrin and allow it to dry. The positions of the bands (spots) should gradually become visible. Draw around the blue or brown bands in pencil, because the colours will fade. Record the positions of the bands relative to the original pencil line and in which direction they travelled.

Worksheet, *continued*

6. Repeat the experiment twice using buffer solutions of (a) pH 4, and (b) pH 9. Keep everything else the same.

   (A better separation is achieved using a gel such as polyacrylamide or agarose which has been made up in the required pH buffer solution. A hole is made in the gel and the sample placed in the hole.)

Results

Record **all** your observations.

Record the distances travelled by the solvent front and spots to the nearest 0.1 cm.

Note any differences in the order of spots when using different solvents or different buffer solutions.

Interpretation and evaluation

1. Use books or the internet to find the chemical structures of the four amino acids.
   (If you cannot access this information ask your teacher for help.)

2. Use your results and the structures of the amino acids to identify each spot.

3. Suggest a reason for the different positions of aspartic and glutamic acids with the pH 6 buffer.

4. Suggest two more factors which may influence the movement of the amino acids in an electric field.

5. A practical instruction book suggests using a voltage of 500 V for 1 hour when carrying out gel electrophoresis on a mixture of peptides. Suggest why the instructions are different from the ones given for your experiment.

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