PAG 01

Moles determination

WHAT THIS PAG COVERS

1.1 percentage by mass of copper(II) carbonate in basic copper carbonate, by reacting it with excess sulfuric acid and collecting the CO_2. **1.2** relative atomic mass of magnesium, by reacting it with sulfuric acid and collecting the H_2. **1.3** empirical formula of magnesium oxide, by heating magnesium in air.

KEY TECHNIQUES

Weighing accurately by difference and to constant mass; measuring the volume of a gas with a gas syringe or over water.

GAS-COLLECTION METHOD (1.1, 1.2)

1. Record the mass of the solid or metal accurately, weighing **by difference**.
2. Add excess acid and replace the bung **as fast as possible** so little gas is lost.
3. Collect the gas in a gas syringe, or over water in an inverted measuring cylinder.
4. Record the final volume once the reaction has stopped, with the room temperature and pressure.
5. Convert the volume to moles (24 000 cm^3 mol^{-1} at RTP, or PV = nRT), then find the % by mass or A_r.

HEATING IN A CRUCIBLE (1.3)

1. Weigh clean magnesium in a crucible with a loose-fitting lid.
2. Heat strongly, lifting the lid briefly at intervals to admit oxygen without losing any solid.
3. Reheat to **constant mass**; the mass gained is the mass of oxygen, which gives the formula.

MEASURING GAS VOLUMES

Gas volume depends on temperature and pressure, so record both. Find moles from the molar gas volume (24 000 cm^3 mol^{-1} at RTP) or from PV = nRT. Collect over water only for gases of low solubility; a gas syringe avoids losses, though the plunger can stick.

KEY EQUATIONS

moles of gas = volume in cm^3 ÷ 24 000 (at RTP)

$Mg + H_2SO_4 \rightarrow MgSO_4 + H_2$ (1.2)

$CuCO_3 + H_2SO_4 \rightarrow CuSO_4 + H_2O + CO_2$ (1.1)

WORKED EXAMPLE (GAS SYRINGE)

0.150 g of a volatile liquid gives 80.0 cm^3 of vapour at 100 kPa and 343 K.

n = PV ÷ RT = 100 000 × 80.0×10^{-6} ÷ (8.31 × 343)

= 2.81 × 10^{-3} mol

M_r = 0.150 ÷ 2.81×10^{-3} = **53.4**

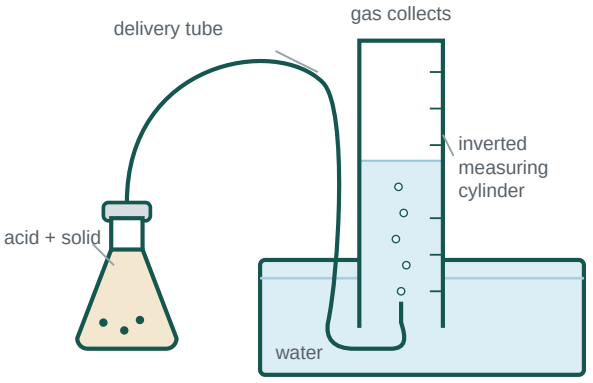
WORKED EXAMPLE (WATER OF CRYSTALLISATION)

2.875 g of hydrated $ZnSO_4$ leaves 1.615 g after heating to constant mass, so the water lost is 1.260 g.

$n(ZnSO_4)$ = 1.615 ÷ 161.5 = 0.0100 mol

$n(H_2O)$ = 1.260 ÷ 18.0 = 0.0700 mol

ratio 1 : 7, so x = 7 and the salt is **$ZnSO_4 \cdot 7H_2O$**



Collecting a gas over water. The reading is slightly low: a little gas escapes before the bung is in, and some CO_2 dissolves in the water. A gas syringe avoids the dissolving problem.

WORKED EXAMPLE (1.3)

0.240 g of magnesium gains 0.160 g of oxygen on heating to constant mass.

n(Mg) = 0.240 ÷ 24 = 0.0100 mol

n(O) = 0.160 ÷ 16 = 0.0100 mol

ratio Mg : O = 1 : 1, so the formula is **MgO**

COMMON EXAM ERRORS

! Gas escaping before the bung is in makes the volume too low, so the % of $CuCO_3$ or the A_r comes out **too small**.

! Forgetting that some CO_2 dissolves in the water over which it is collected.

! In 1.3, not lifting the lid lets in too little oxygen, so the formula shows **too little oxygen**; using a wet crucible makes the mass loss too large.

WHAT TO DRAW

If you draw a gas syringe, show the **graduation markings** so a volume can be read, and leave no gaps where gas could escape. Show the crucible lid slightly open.

REDUCING UNCERTAINTY

Small masses carry the largest percentage uncertainty, so weigh by difference and use a balance reading to more decimal places, or a larger mass.

WATER OF CRYSTALLISATION

The same constant-mass method finds the water in a hydrated salt: heat to constant mass, and the mass lost is the water. For $ZnSO_4 \cdot xH_2O$, the ratio $n(ZnSO_4)$: $n(H_2O)$ gives x.

ONE METHOD, THREE ANSWERS

All three rest on the same step: turn a measurement into moles, then use the equation. 1.1 and 1.2 convert a gas volume to moles; 1.3 works from the masses of magnesium and oxygen. The mole ratio then gives the A_r, the formula, or the percentage by mass.

Safety: Sulfuric acid is an irritant — wear eye protection. In 1.3 the crucible, tripod and tongs stay hot; let them cool before handling.

PAG 02

Acid–base titration

WHAT THIS PAG COVERS

2.1 concentration of hydrochloric acid, using a standard solution of sodium hydrogencarbonate. **2.2** molar mass of an acid (citric acid monohydrate) against standard sodium hydroxide. **2.3** identity of an unknown Group 1 carbonate, by finding its molar mass.

MAKING THE STANDARD SOLUTION

1. Weigh the solid **by difference** on a two- or three-decimal-place balance.
2. Dissolve it in distilled water, stirring with a glass rod; warm gently if slow, then cool.
3. Transfer to a 250 cm^3 volumetric flask through a funnel, rinsing the beaker, rod and funnel in.
4. Make up to the line with distilled water, a dropping pipette for the last drops, the bottom of the meniscus on the line.
5. Stopper and invert several times. Never add a warm solution — the glass is calibrated at a set temperature.

CARRYING OUT THE TITRATION

1. Rinse the burette with the solution it will hold, the pipette with its solution, the flask with distilled water only.
2. Pipette 25.0 cm^3 into the conical flask, touching the last drop off against the surface.
3. Add only a **few drops** of indicator, then fill the burette and the jet space below the tap.
4. Titrate while swirling, dropwise near the end point; record every reading to 0.05 cm^3.
5. Do a rough run, then repeat to **concordant** titres (within 0.10 cm^3) and mean only those.

CHOOSING THE INDICATOR

With a strong base such as **NaOH**, use **phenolphthalein** — pink in alkali, and the end point is where the pink just disappears. With a strong acid such as **HCl**, use **methyl orange** — yellow in alkali, red in acid, orange at the end point. Indicators are themselves weak acids, so add only a few drops.

RECORDING YOUR RESULTS

	Rough	1	2
Final reading / cm^3	23.05	22.55	22.50
Initial reading / cm^3	0.00	0.05	0.00
Titre / cm^3	23.05	22.50	22.50

Mean only the **concordant** titres — those within 0.10 cm^3. Here that is runs 1 and 2, giving a mean of 22.50 cm^3; the rough run is discarded.

KEY EQUATIONS

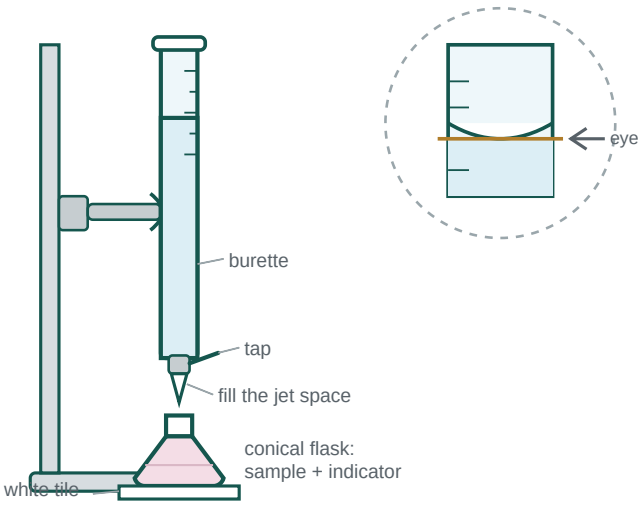
$HCl + NaHCO_3 \rightarrow NaCl + H_2O + CO_2$ (2.1)

$2HCl + M_2CO_3 \rightarrow 2MCl + H_2O + CO_2$ (2.3)

citric acid is triprotic: 1 mol reacts with 3 mol NaOH (2.2)

DILUTING A SAMPLE

If a sample is too concentrated to titrate, pipette 25.0 cm^3 into a 250 cm^3 volumetric flask and make up to the mark — a tenfold dilution. Use the pipette and flask, never a measuring cylinder, to keep the uncertainty low.



Read the burette at eye level from the bottom of the meniscus, to 0.05 cm^3, and keep the jet space below the tap filled.

READING THE BURETTE

Record to **two decimal places**, ending in 0.00 or 0.05. On a line, read .00; between two lines, read .05 — one drop is about 0.05 cm^3.

WORKED EXAMPLE

25.0 cm^3 of NaOH needs a mean titre of 22.50 cm^3 of 0.100 mol dm^{-3} HCl.

$HCl + NaOH \rightarrow NaCl + H_2O$

$n(HCl) = 0.100 \times 22.50 \div 1000 = 2.25 \times 10^{-3}$ mol

ratio 1 : 1, so $n(NaOH) = 2.25 \times 10^{-3}$ mol

$c(NaOH) = 2.25 \times 10^{-3} \div (25.0 \div 1000) =$ **$0.0900\ mol\ dm^{-3}$**

COMMON EXAM ERRORS

- ! Leaving the **funnel** in the burette: drops can run in after you read it, so the titre is unreliable.
- ! Not filling the **jet space** below the tap: it fills during the titration, so the titre reads too high.
- ! Not rinsing the burette with its solution: residual water dilutes the titrant, so the titre is too large.
- ! Adding too much indicator, or reading the wrong meniscus: both shift the apparent end point.

UNCERTAINTY

Each reading is ±0.05 cm^3, so a titre carries ±0.10 cm^3. Percentage uncertainty = 0.10 ÷ titre × 100, so a **larger titre** lowers it.

WASHING DOWN THE FLASK

You may rinse the inside walls of the conical flask with distilled water during the titration to wash splashes back in. It does **not** change the titre, because water adds no extra moles of acid or alkali.

Safety: Acids and alkalis are irritants, and corrosive when concentrated; wear eye protection throughout and rinse any splashes promptly.

PAG 03

Enthalpy determination

WHAT THIS PAG COVERS

3.1 enthalpy change of neutralisation (hydrochloric acid with sodium hydroxide). **3.2** an enthalpy change by Hess' law — K_2CO_3 and $KHCO_3$ with acid, to find the enthalpy of decomposition of $KHCO_3$. **3.3** enthalpy change of combustion of alcohols.

KEY TECHNIQUES

Calorimetry — measuring a temperature change — and processing the data, including extrapolating a temperature–time graph.

METHOD ESSENTIALS

1. Measure known volumes or masses of the reactants and record the starting temperature each minute for a few minutes.
2. Mix, stir, and record the temperature every minute; find the maximum (or minimum) and so ΔT.
3. For combustion (3.3), measure the mass of fuel burned and the mass and temperature rise of the water.
4. When a solid dissolves or reacts into a solution, add the solid and solution masses together for m.

SLOW REACTIONS

If the reaction is slow, cooling overlaps it, so record the temperature at regular intervals before and after mixing and **extrapolate both lines back** to the moment of mixing to read the true ΔT.

KEY EQUATIONS

$q = mc\Delta T$ ($c = 4.18$ J g^{-1} K^{-1} for solutions)

$\Delta H = -q \div n$ ($\div 1000$ for kJ mol^{-1})

Hess' law: $\Delta H_1 = \Delta H_2 + \Delta H_3$

HESS' LAW (3.2)

The decomposition of $KHCO_3$ is too slow to measure directly, so react K_2CO_3 and $KHCO_3$ separately with excess acid, measure each ΔH, and combine them through a Hess cycle to find the enthalpy of decomposition.

WORKED EXAMPLE (3.3)

0.650 g of propan-1-ol raises 150 g of water by 25.4 K.

$q = mc\Delta T = 150 \times 4.18 \times 25.4 = 15\,926$ J

$n = 0.650 \div 60.0 = 0.01083$ mol

$\Delta H = -15\,926 \div 0.01083 =$ **-1470 kJ mol^{-1}**

WORKED EXAMPLE (HESS' LAW, 3.2)

Measured: $KHCO_3$ + HCl gives +21.0 kJ mol^{-1}; K_2CO_3 + 2HCl gives −36.0 kJ mol^{-1}.

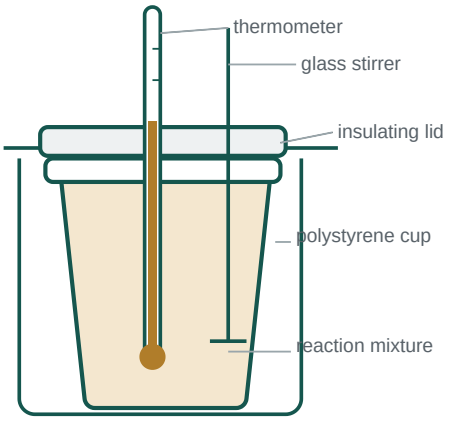
For $2KHCO_3 \rightarrow K_2CO_3 + H_2O + CO_2$, round the cycle:

$\Delta H = 2(+21.0) - (-36.0)$

$=$ **$+78.0$ kJ mol^{-1}**

GETTING N RIGHT

Divide q by the moles the answer is quoted **per**: per mole of water for neutralisation, per mole of fuel for combustion, per mole of the solid for a dissolving reaction. Using the wrong n is the commonest slip here.



A polystyrene cup inside a beaker, with a lid, reduces heat loss. The thermometer bulb is immersed and the stirrer keeps the mixture at one temperature.

WORKED EXAMPLE (3.1)

25.0 cm^3 of 2.0 mol dm^{-3} HCl and 25.0 cm^3 of 2.0 mol dm^{-3} NaOH give $\Delta T = 13.5$ K.

$q = mc\Delta T = 50 \times 4.18 \times 13.5 = 2822$ J

$n(HCl) = 2.0 \times 25.0 \div 1000 = 0.0500$ mol

$\Delta H = -2822 \div 0.0500 =$ **-56.4 kJ mol^{-1}**

COMMON EXAM ERRORS

- ! Heat lost to the surroundings makes ΔT too small — combustion is worst affected, so the value is **less exothermic** than the data book.
- ! Incomplete combustion (3.3) releases less energy than complete combustion.
- ! Assuming the c and density of pure water for solutions, and not being at standard conditions, both add error.

WHAT TO DRAW

Show the cup standing in a beaker for insulation, a **lid**, the thermometer bulb below the liquid surface, and a stirrer.

FLAME CALORIMETRY (3.3)

For combustion, find the mass of fuel burned from the spirit burner before and after, and the temperature rise of a known mass of water. A draught shield and a short distance to the beaker reduce heat loss and incomplete combustion.

IMPROVING ACCURACY

Reduce heat loss with a lid, an insulated cup, and a draught shield; stir to spread the heat evenly; and extrapolate the cooling line back to the moment of mixing. Take the specific heat capacity and density of a dilute solution as those of water. A temperature probe read to 0.1 K lowers the uncertainty.

DEFINITIONS TO QUOTE

Standard enthalpy of neutralisation is the energy released when one mole of water forms from acid and alkali under standard conditions; standard enthalpy of combustion is for one mole of a fuel burned completely. Written papers often ask for these in words.

Safety: Alcohols are flammable — keep the spirit burner away from other flames, and refill it well away from any naked flame.

PAG 04

Qualitative analysis of ions

WHAT THIS PAG COVERS

4.1 identifying the positive and negative ions in a mixture of Group 2 salts. **4.2 & 4.3** identifying a range of unknown compounds, choosing the tests yourself.

ORDER MATTERS

Test for **carbonate, then sulfate, then halide**. Carbonate and sulfate also give precipitates with silver and barium, so interfering ions must be removed or ruled out first, and solutions acidified before the precipitation test.

CATION TESTS — ADD NAOH(AQ)

Ion	Result with NaOH(aq)
Mg^{2+}	white precipitate
Ca^{2+}	white precipitate (needs more NaOH)
Sr^{2+}, Ba^{2+}	no precipitate (very alkaline)
Cu^{2+}	blue precipitate
Fe^{2+}	green precipitate
Fe^{3+}	brown precipitate
Cr^{3+}	green precipitate
NH_4^+	warm: NH_3 turns damp red litmus blue

Group 2 hydroxides become **more soluble** down the group, so the precipitate is harder to form lower down.

GROUP 2 TRENDS BEHIND THE TESTS

Hydroxide solubility **increases** down Group 2, so Sr and Ba give no hydroxide precipitate. Sulfate solubility **decreases** down the group, so Sr and Ba give a sulfate precipitate while Mg and Ca do not.

KEY IONIC EQUATIONS

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2$$

$$Ba^{2+} + SO_4^{2-} \rightarrow BaSO_4$$

$$Ag^+ + Cl^- \rightarrow AgCl$$

FLAME TESTS

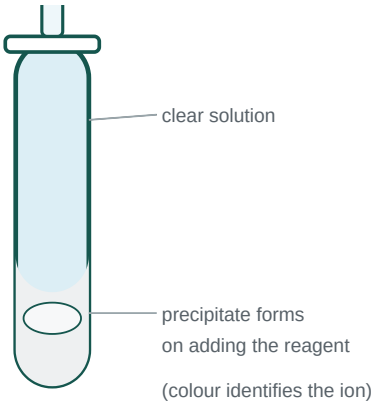
A flame test tells the Group 2 cations apart: Ca^{2+} brick-red, Sr^{2+} crimson, Ba^{2+} pale (apple) green. Clean a nichrome wire in concentrated hydrochloric acid, dip it in the solid, and hold it at the edge of a blue Bunsen flame.

WORKED IDENTIFICATION

A white solid gives a brick-red flame, a white precipitate with NaOH (more needed), and fizzes with dilute acid to turn limewater milky.
Brick-red flame and the hydroxide point to Ca^{2+}.
Fizzing and milky limewater show carbonate.
The compound is **calcium carbonate, $CaCO_3$**.

HYDROXIDES IN EXCESS

Some hydroxide precipitates redissolve in **excess** NaOH because they are amphoteric — $Al(OH)_3$, $Cr(OH)_3$ and $Zn(OH)_2$ dissolve, while Cu, Fe and Mg hydroxides do not. Adding NaOH dropwise then in excess separates them.



A precipitate on adding the reagent is a positive result; its colour identifies the ion.

ANION TESTS

Ion	Reagent	Positive result
Carbonate	dilute acid	fizzing; gas turns limewater milky
Sulfate	acidify (HCl), $BaCl_2$	white precipitate
Halide	acidify (HNO_3), $AgNO_3$	white / cream / yellow precipitate
Hydroxide	red litmus	turns blue

CONFIRMING A HALIDE

AgCl dissolves in **dilute** ammonia; AgBr dissolves only in **concentrated** ammonia; AgI does not dissolve. This separates the three by solubility.

COMMON EXAM ERRORS

- ! Testing for halides **before** removing carbonate or sulfate gives a false precipitate.
- ! Not acidifying first: carbonate gives a white precipitate with both $BaCl_2$ and $AgNO_3$.
- ! Using sulfuric acid in the sulfate test (it contains sulfate), or HCl in the halide test (it adds chloride).

WORKING THROUGH AN UNKNOWN

Keep to the order so each result is reliable: dilute acid for carbonate, then acidify and add $BaCl_2$ for sulfate, then acidify with nitric acid and add $AgNO_3$ for a halide, confirming with ammonia. Use a fresh sample for each test.

TWO GAS TESTS

A carbonate fizzes with dilute acid and the CO_2 turns limewater milky. An ammonium salt warmed with NaOH releases ammonia, which turns damp red litmus blue and smells pungent. Both confirm an ion that the precipitate tests alone would miss.

RECORDING OBSERVATIONS

Note the reagent added, then describe the colour precisely — cream, not just white — and say whether a precipitate forms or dissolves in excess. The halides are told apart by small colour differences, so a vague description loses marks.

Safety: Silver nitrate stains skin and clothing; barium salts are toxic. Wear eye protection and wash your hands afterwards.

PAG 05

Synthesis of an organic liquid

WHAT THIS PAG COVERS

5.1 a haloalkane, from an alcohol and concentrated hydrochloric acid. **5.2** cyclohexene, by dehydrating cyclohexanol. **5.3** oxidation of ethanol to ethanal and to ethanoic acid. **5.4** hydration of hex-1-ene to hexan-2-ol.

KEY TECHNIQUES

Heating under reflux; distillation; purifying a liquid with a separating funnel and a drying agent.

METHOD ESSENTIALS

1. Heat under reflux with anti-bumping granules so it reacts fully without boiling dry.
2. Distil to collect the product over its boiling range, the thermometer bulb at the side-arm.
3. Shake with the separating funnel to wash it; run off and discard the lower aqueous layer.
4. Dry with an anhydrous drying agent until the liquid is clear, then redistil if a purer product is needed.

WASHING THE PRODUCT

Wash with sodium hydrogencarbonate to remove acid (release the CO_2 pressure), or saturated sodium chloride to help the layers separate. The lower-density organic layer is usually on top. The drying agent must be insoluble in and unreactive with the liquid.

KEY REACTIONS

cyclohexanol → cyclohexene + H_2O (conc H_3PO_4, 5.2)

CH_3CH_2OH → CH_3CHO → CH_3COOH ([O], 5.3)

R–OH + HCl → R–Cl + H_2O (5.1)

hex-1-ene + H_2O → hexan-2-ol (H_3PO_4, 5.4)

ALDEHYDE OR CARBOXYLIC ACID (5.3)

Oxidising ethanol with acidified potassium dichromate gives the **aldehyde** (ethanal) if you distil it off as it forms, before it can oxidise further, or the **carboxylic acid** (ethanoic acid) if you heat under reflux with excess oxidant so oxidation goes to completion. The dichromate turns from orange to green either way.

SEPARATING THE LAYERS

Identify the organic layer by adding a little water — the aqueous layer grows. Run the lower layer off through the tap, keep whichever layer is the product, and discard the other. Vent the funnel often when washing with hydrogencarbonate, as CO_2 builds up pressure.

WORKED EXAMPLE (PERCENTAGE YIELD)

10.0 g of cyclohexanol (M_r 100.0) gives cyclohexene (M_r 82.0), 1 : 1.

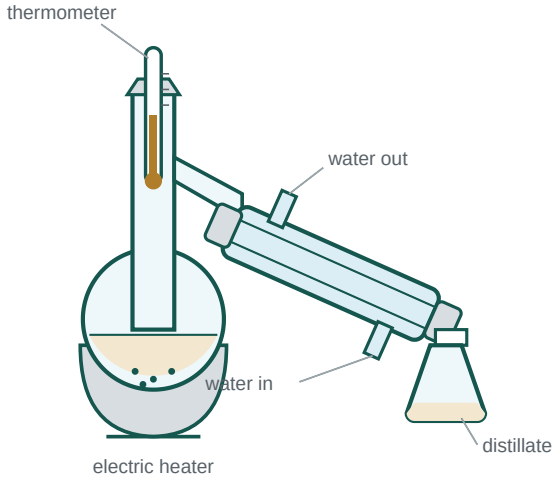
n = 10.0 ÷ 100.0 = 0.100 mol

maximum mass = 0.100 × 82.0 = 8.20 g

5.20 g collected, so yield = 5.20 ÷ 8.20 × 100 = **63.4%**

CHECKING THE PRODUCT

A pure product distils over a narrow range at its boiling point. An infrared spectrum confirms the change: the broad O–H band of the alcohol disappears, and a new absorption appears — C=C for cyclohexene, or C–Cl for the haloalkane.



Distillation. The thermometer bulb sits level with the side-arm to read the vapour temperature; cooling water runs counter-current, in at the lower end and out at the upper; anti-bumping granules give smooth boiling.

DRAWING REFLUX CORRECTLY

- The top of the condenser is **open**, never sealed — a sealed system builds up dangerous pressure.
- Draw the outer water jacket sealed top and bottom, with two open ports for water in and out.
- No thermometer is needed in reflux, and vapour should not reach the top.
- Do not draw lines between the flask and the condenser.

DRAWING DISTILLATION CORRECTLY

- The thermometer **bulb** sits level with the side-arm, so it reads the vapour temperature.
- Cooling water runs **counter-current**: in at the lower end, out at the upper.
- The receiver is **open** to the air — never seal the apparatus.
- Collect over a narrow range around the product's boiling point.

COMMON EXAM ERRORS

! Drawing the reflux condenser **sealed** at the top, or adding a thermometer — both wrong and unsafe.

! Condenser water flowing in at the top gives poor cooling and lost vapour.

! Leaving out anti-bumping granules causes violent, uneven boiling.

CHOOSING THE DRYING AGENT

Use an anhydrous solid that is insoluble in and unreactive with the product, such as anhydrous magnesium sulfate or sodium sulfate. Calcium chloride is unsuitable for alcohols, which it reacts with. Add until the cloudiness clears, then decant off the dry liquid.

REFLUX OR DISTILLATION

The same condenser is used differently: **vertical** in reflux, returning vapour to the flask for prolonged heating; **sloped** (Liebig) in distillation, carrying the product away as it forms over a narrow boiling range.

Safety: Concentrated acids are corrosive and many of these liquids are flammable; heat with an electric mantle or water bath, not a naked flame.

PAG 06

Synthesis of an organic solid

WHAT THIS PAG COVERS

6.1 aspirin, in two stages from 2-hydroxybenzoic acid and ethanoic anhydride. **6.2** benzoic acid, by alkaline hydrolysis of an ester then acidification. **6.3** methyl 3-nitrobenzoate.

KEY TECHNIQUES

Reflux; filtration under reduced pressure; recrystallisation; melting-point measurement; thin-layer chromatography.

RECRYSTALLISATION

1. Dissolve the crude solid in the **minimum** volume of hot solvent to make a saturated solution.
2. Hot-filter through fluted paper to remove insoluble impurities; the heat stops crystals reforming.
3. Cool, ideally in ice, to crystallise — soluble impurities stay in solution.
4. Filter under reduced pressure in a Buchner funnel; wash with a little cold solvent and dry.

MELTING POINT

A pure solid melts **sharply** at the data-book value. Impurities lower the melting point and spread it over a range. Use a melting-point machine, or a capillary tube strapped to a thermometer in heating oil.

THIN-LAYER CHROMATOGRAPHY

- Spot the sample on a **pencil** baseline; keep the solvent below that line.
- Run with a lid on to stop the solvent evaporating; use a UV lamp for colourless spots.
- R_f = distance moved by spot ÷ distance moved by solvent (to the centre of each spot).

TWO-STAGE SYNTHESES

Aspirin (6.1) is made by esterifying the phenol group of 2-hydroxybenzoic acid; benzoic acid (6.2) by hydrolysing an ester under reflux with alkali, then acidifying to precipitate the solid.

WORKED EXAMPLE (PERCENTAGE YIELD)

5.00 g of 2-hydroxybenzoic acid (M_r 138.0) is used; aspirin (M_r 180.0) forms 1 : 1.

n = 5.00 ÷ 138.0 = 0.0362 mol

maximum mass = 0.0362 × 180.0 = 6.52 g

4.80 g obtained, so yield = 4.80 ÷ 6.52 × 100 = **73.6%**

CHOOSING THE SOLVENT

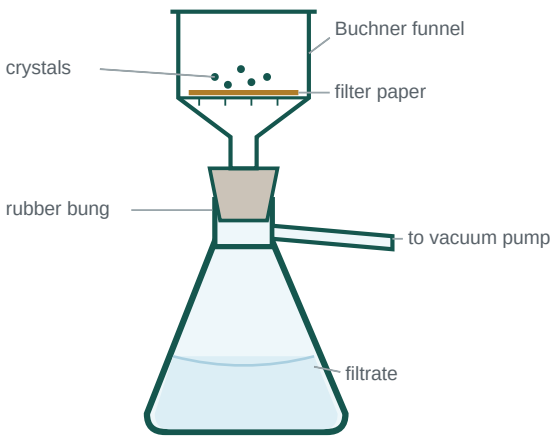
A good recrystallisation solvent dissolves the solid when hot but barely when cold, and does not react with it. Impurities should either stay dissolved in the cold solvent or be insoluble and come out in the hot filtration.

CONDITIONS FOR EACH PREP

Aspirin (6.1) needs a few drops of concentrated acid as catalyst with the ethanoic anhydride. Methyl 3-nitrobenzoate (6.3) is made by nitrating methyl benzoate with concentrated nitric and sulfuric acids, kept cold to favour the single nitro product.

WHY THE ANHYDRIDE

Aspirin is made with ethanoic anhydride rather than ethanoic acid because the anhydride is more reactive and the reaction does not reach an equilibrium, so the yield is higher and the by-product is only ethanoic acid, which washes away.



Filtration under reduced pressure. An airtight rubber bung seals the funnel into the thick-walled flask, so the vacuum pulls the filtrate through and leaves the crystals on the paper. Draw the filter paper in the funnel.

COMMON EXAM ERRORS

- ! Using too much hot solvent in recrystallisation lowers the yield — product stays dissolved.
- ! Solvent above the pencil line in TLC washes the spots off the plate.
- ! Reporting an impure melting point as a single value rather than a range.
- ! Not drying the crystals, so the mass is too high and the yield comes out above 100%.

WHERE YIELD IS LOST

Crystals are lost when filtering and washing, some product stays dissolved after recrystallisation, and side reactions reduce the amount formed.

WHAT TO DRAW

Show the Buchner funnel with **filter paper**, the thick-walled side-arm flask, and the side arm connected to the vacuum pump.

CHECKING PURITY

Compare the melting point of the dried solid with the data-book value: a pure sample melts sharply at that temperature, an impure one lower and over a range. A single TLC spot is further evidence of purity.

MEASURING THE MELTING POINT

Pack a little dry solid into a capillary tube, heat slowly near the expected value, and record the range from the first liquid to fully melted. A pure solid melts within a degree or two; a wider range, starting lower, means an impurity remains.

MIXED MELTING POINT

To confirm identity, mix the product with a known pure sample and take the melting point again. The same compound still melts sharply at the same temperature; a different one melts lower and over a wider range.

Safety: Ethanoic anhydride and concentrated acids are corrosive; work in a fume cupboard where the method directs. Ethanol is flammable — keep it from naked flames.

PAG 07

Qualitative analysis of organic functional groups

WHAT THIS PAG COVERS

7.1 tests for alkenes and haloalkanes. **7.2** tests for alcohols, aldehydes and carboxylic acids. **7.3** confirming the functional groups in naturally occurring compounds.

KEY TECHNIQUES

Functional-group test-tube reactions; gentle heating in a water bath or electric heater; recording clear observations.

TESTS TO KNOW

Group	Test	Positive result
Alkene	shake with bromine water	orange → colourless
Carbonyl	2,4-DNP	orange precipitate
Aldehyde	warm with Tollens'	silver mirror
1°/2° alcohol, aldehyde	warm, acidified $K_2Cr_2O_7$	orange → green
Carboxylic acid	add Na_2CO_3	fizzing (CO_2)
$CH_3CO–$ / $CH_3CH(OH)–$	warm with I_2 + NaOH	pale yellow ppt (CHI_3)
Haloalkane	warm $AgNO_3$ in ethanol	slow white/cream/yellow ppt

CARBONYL FOLLOW-UP

A 2,4-DNP precipitate confirms an aldehyde or ketone; the melting point of the purified orange derivative, compared with data values, identifies **which** carbonyl compound it is.

BROMINE WATER: ALKENE OR PHENOL

An alkene decolourises bromine water by addition across the C=C. A **phenol** also decolourises it but forms a **white precipitate** of 2,4,6-tribromophenol, which tells the two apart.

FEHLING'S AND BENEDICT'S

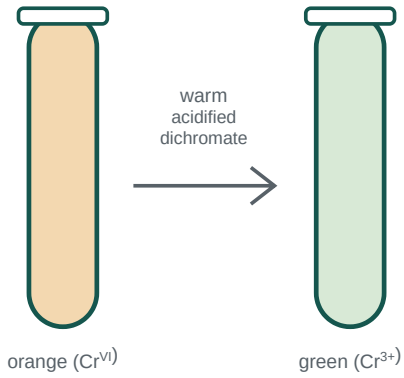
An alternative test for an aldehyde: warming with Fehling's or Benedict's solution gives a **brick-red** precipitate of copper(I) oxide, while a ketone leaves the blue solution unchanged.

OXIDATION PRODUCTS

Acidified dichromate oxidises a **primary** alcohol to an aldehyde, then to a carboxylic acid — distil to stop at the aldehyde, or reflux for the acid. A **secondary** alcohol gives a ketone; a **tertiary** alcohol is not oxidised.

THE IODOFORM TEST

Warming with iodine and sodium hydroxide gives a pale-yellow precipitate of triiodomethane (CHI_3) with an antiseptic smell. It is positive for a $CH_3CO–$ group or a $CH_3CH(OH)–$ group, so ethanal and ethanol give it but propanal does not.



Warming a primary or secondary alcohol, or an aldehyde, with acidified dichromate turns it from orange to green.

USEFUL DISTINCTIONS

- **Tertiary** alcohols are not oxidised by dichromate, so they give no colour change.
- 2,4-DNP confirms a carbonyl; **Tollens'** then separates aldehyde (silver mirror) from ketone (no change).
- Haloalkane rate follows the C–X bond strength: C–I fastest, C–Cl slowest.
- **Phenols** react with sodium and NaOH but not with Na_2CO_3, unlike carboxylic acids.

COMMON EXAM ERRORS

! Using a naked flame to warm volatile, flammable reagents — use a water bath.

! Forgetting that tertiary alcohols give no change with dichromate.

! Not acidifying before the silver nitrate step in the haloalkane test.

PUTTING TESTS TOGETHER

Combine the results to place each group. A compound that decolourises bromine water and gives an orange 2,4-DNP precipitate but no silver mirror has a C=C and a **ketone**; if it also fizzes with Na_2CO_3, it has a carboxylic acid as well.

TESTING IN ORDER

Work from the least to the most specific. Bromine water and acidified dichromate place a C=C or an oxidisable alcohol first; 2,4-DNP then Tollens' pin down a carbonyl; the iodoform test finds a CH_3CO or $CH_3CH(OH)$ group. Record each observation as you go.

CONFIRMING NATURAL COMPOUNDS (7.3)

Natural compounds often carry several functional groups at once, so no single test is enough. Test in sequence and read the results together: a molecule that decolourises bromine water and gives a silver mirror with Tollens' holds both a C=C and an aldehyde.

Safety: Tollens' reagent must be made fresh and not stored — it can become explosive on standing. Dichromate is toxic and a category 2 carcinogen; wear gloves.

PAG 08

Electrochemical cells

WHAT THIS PAG COVERS

8.1 a simple cell from two half-cells (a metal electrode; an Fe^{3+}/Fe^{2+} half-cell on an inert electrode). **8.2** measuring cell potentials and ranking oxidising agents. **8.3** a microscale cell built on one piece of filter paper.

KEY TECHNIQUES

Setting up electrochemical half-cells and measuring the cell potential.

METHOD ESSENTIALS

1. Set up each half-cell: a metal in a solution of its ions, or an inert graphite or platinum electrode where both species are in solution.
2. Clean and degrease metal electrodes with emery paper and propanone.
3. Join the half-cells with a salt bridge — filter paper soaked in saturated potassium nitrate.
4. Connect a **high-resistance** voltmeter and read the cell potential.
5. Compare under standard conditions: 1.0 mol dm^{-3} solutions, 298 K, 100 kPa.

KEY EQUATION

$E°_{cell}$ = E°(positive electrode) − E°(negative electrode)

WORKED EXAMPLE (CELL POTENTIAL)

$E°(Cu^{2+}/Cu)$ = +0.34 V and $E°(Zn^{2+}/Zn)$ = −0.76 V.

$E°_{cell}$ = +0.34 − (−0.76)

= **+1.10 V**

Cu^{2+}/Cu is the positive electrode (reduction); Zn is oxidised.

AT EACH ELECTRODE

At the negative electrode the metal is oxidised, $Zn \rightarrow Zn^{2+} + 2e^-$; at the positive electrode the ion is reduced, $Cu^{2+} + 2e^- \rightarrow Cu$. Electrons released at the negative electrode pass through the wire to the positive one.

STANDARD CONDITIONS

Quote potentials against the standard hydrogen electrode (0.00 V) under standard conditions — 1.0 mol dm^{-3}, 298 K and 100 kPa. The more positive electrode is the one reduced, the more negative the one oxidised.

RANKING OXIDISING AGENTS (8.2)

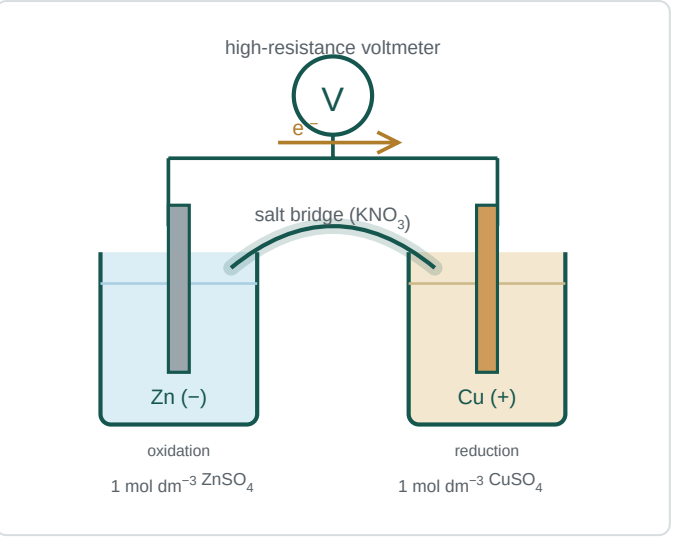
The more positive the electrode potential, the stronger the oxidising agent, since that species is the most readily reduced. Measuring several half-cells against one reference puts the oxidising agents in order of strength.

PREDICTING FEASIBILITY

A positive $E°_{cell}$ means the reaction is feasible under standard conditions. It does not give the rate, so a feasible reaction may still be too slow to see.

MICROSCALE CELL (8.3)

The same cell works on a single piece of filter paper soaked in potassium nitrate, with a small drop of each solution and the two electrodes — quick, and sparing with chemicals.



Electrons flow through the wire from the more negative electrode (oxidation) to the more positive (reduction); the salt bridge completes the circuit.

WHY THESE CHOICES

- A **high-resistance** voltmeter lets almost no current flow, so it reads the maximum (true) cell potential.
- **Potassium nitrate** in the salt bridge does not react; chloride would form complexes with copper or a precipitate with silver.
- A wire is not used for the bridge — it would set up its own electrode system.
- An inert **platinum** electrode is used where both species are in solution, as in Fe^{3+}/Fe^{2+}.

COMMON EXAM ERRORS

! A low-resistance voltmeter, or letting current flow, drops the reading **below** the true cell potential.

! A salt-bridge ion that reacts with the contents of a half-cell.

! Not using standard conditions, or unequal Fe^{2+} and Fe^{3+} concentrations.

THE CONVENTIONAL CELL DIAGRAM

By convention the cell is written left to right, Zn(s) | Zn^{2+}(aq) ‖ Cu^{2+}(aq) | Cu(s), with the most oxidised species either side of the salt-bridge ‖. The right-hand electrode is the positive one, where reduction occurs.

CHANGING THE CONDITIONS

Concentration shifts each half-cell equilibrium and the measured potential. Raising the concentration of the ion being reduced makes that electrode more positive and raises the cell potential, as Le Chatelier predicts.

CELLS AND THE DATA BOOKLET

Standard potentials are listed as reduction half-equations. The more positive one runs forwards as reduction, the more negative one reverses as oxidation. Subtract them for $E°_{cell}$, then add the two half-equations and cancel the electrons for the overall reaction.

Safety: Solutions of transition-metal and heavy-metal salts are harmful; avoid skin contact and wash your hands afterwards.

PAG 09

Rates of reaction — continuous monitoring

WHAT THIS PAG COVERS

9.1 catalytic decomposition of hydrogen peroxide (MnO_2), following the oxygen given off. **9.2** calcium carbonate with hydrochloric acid, by mass loss and by gas volume. **9.3** magnesium with hydrochloric acid, following the hydrogen given off.

KEY TECHNIQUES

Continuous monitoring of a single reaction over time; recording time; processing the data, often with software.

METHOD ESSENTIALS

1. Mix the reactants and **start timing immediately**.
2. Record the gas volume (gas syringe or over water) at regular intervals, or the falling mass on a balance.
3. Continue until the reaction stops; the rate falls to zero as a reactant is used up.
4. Plot volume (or mass lost) against time; the gradient of the tangent gives the rate.

READING THE GRAPH

The reaction is **fastest at the start**, where the gradient is steepest, and the line levels off when a reactant runs out. A constant half-life on a concentration–time graph shows a first-order reaction.

KEY EQUATIONS

$2H_2O_2 \rightarrow 2H_2O + O_2$ (MnO_2 catalyst, not used up)

$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2$

$Mg + 2HCl \rightarrow MgCl_2 + H_2$

MASS-LOSS METHOD

When a gas escapes, the falling mass tracks the reaction. A loose cotton-wool plug stops acid spray leaving, but gas still escapes; the method suits a heavy gas such as CO_2 better than light H_2.

CALCULATING A RATE

Draw a tangent to the curve and find its gradient: rate = change in volume (or mass) ÷ change in time. The initial rate is the gradient at t = 0.

FOLLOWING THE REACTION TWO WAYS

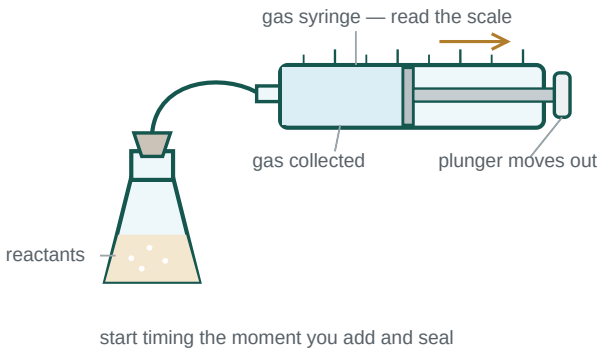
9.2 can be followed by **mass loss** and by **gas volume**, and the two should agree. Mass loss needs only a balance and suits the heavy CO_2; gas volume needs a syringe and works for any gas. Running both is a check on the result.

CONTINUOUS OR INITIAL RATES

Continuous monitoring follows one reaction throughout, giving the whole curve and the order from its shape or half-life. The initial-rates (clock) method in PAG 10 instead compares the starting rate across several runs. Both lead to the rate equation.

SHOWING THE CATALYST IS UNCHANGED

In 9.1 the manganese(IV) oxide speeds the decomposition but is left chemically unchanged. Filter, dry, and reweigh it at the end: a recovered mass close to the start confirms it is a catalyst, not a reactant.



A gas syringe records the volume of gas as it is produced. Seal the flask so none escapes, and scale the quantities so the total fits within the syringe.

WAYS TO FOLLOW A RATE

- Gas volume, with a gas syringe or over water, when the number of moles of gas changes.
- Mass loss on a balance, when a gas escapes — better for a heavy gas such as CO_2.
- Colorimetry, when a reactant or product is coloured.

COMMON EXAM ERRORS

! Gas lost in the moment before the bung is fitted makes the early readings too low.

! Producing more gas than the syringe holds pushes the plunger off-scale and loses data.

! In the mass-loss method, light gases such as H_2 give too small a change to measure well.

ORDER FROM HALF-LIFE

From a concentration–time graph, a **constant** half-life means a first-order reaction; a half-life that lengthens as the reaction proceeds points to second order.

WORKED EXAMPLE (RATE FROM A TANGENT)

A tangent to the volume–time curve at t = 0 rises from 0 to 36 cm^3 over 30 s.

initial rate = 36 ÷ 30 = **1.2 cm^3 s^{-1}**

Divide by 24 000 to convert to mol s^{-1} if needed.

FOLLOWING A RATE BY COLOUR

Colorimetry tracks a coloured reactant or product: set the filter to the colour absorbed, calibrate with known concentrations, then record the absorbance over time. It suits reactions such as the fading of a halogen or a transition-metal ion.

KEEPING IT FAIR

Hold the temperature steady in a water bath, since rate is very sensitive to it, and keep the apparatus, volumes, and concentrations the same between runs. Repeat each run and average, discarding any clearly anomalous result.

Safety: Hydrogen peroxide is an irritant and an oxidiser; wear eye protection and mop up spills promptly.

PAG 10

Rates of reaction — initial rates

WHAT THIS PAG COVERS

10.1 an iodine clock — finding the order with respect to I^- and the rate constant. **10.2** thiosulfate with acid, timed by the sulfur precipitate hiding a cross. **10.3** activation energy from the manganate(VII)–ethanedioic acid reaction, using the Arrhenius equation.

KEY TECHNIQUES

The initial-rates (clock) method; identifying and controlling variables; processing data.

METHOD ESSENTIALS

1. Change one variable — a concentration, or the temperature — and keep the others controlled.
2. Time how long it takes to reach a fixed, visible end point (a colour appears, or a cross disappears).
3. Take the initial rate as proportional to 1 ÷ time.
4. For order, plot log(rate) against log[X] (gradient = order); for E_a, plot ln k against 1/T.

KEY RELATIONSHIPS

rate = $k[A]^m[B]^n$ and initial rate ∝ 1 ÷ t

ln k = constant − E_a/RT

gradient of ln k vs 1/T = − E_a ÷ R

THE IODINE CLOCK (10.1)

Hydrogen peroxide oxidises iodide to iodine; a small, fixed amount of thiosulfate removes the iodine until it runs out, when starch turns the mixture blue-black. Vary $[I^-]$ and time to the colour to find the order.

FINDING THE ORDER

Halve a reactant's concentration and watch the initial rate: if it halves, the reaction is **first order** in that reactant; no change means **zero order**; a fourfold rate means **second order**. The clock time, proportional to 1 ÷ rate, makes this quick to test.

WHY A CLOCK WORKS

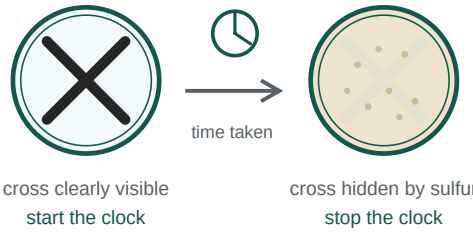
A clock reaction reaches a sharp, visible end point only after a fixed, small amount of product has formed, so the time to that point measures the **initial** rate while the concentrations have barely changed.

THE MANGANATE REACTION (10.3)

Manganate(VII) and ethanedioic acid react slowly at first, then speed up as the product Mn^{2+} autocatalyses the reaction. For an activation-energy run, measure the initial rate at several temperatures and plot ln k against 1/T.

ORDERS AND UNITS OF K

The overall order is the sum of the individual orders, and the units of the rate constant follow from it: s^{-1} for first order, and mol^{-1} dm^3 s^{-1} for second order.



Looking down into the flask, time how long the cross takes to vanish as sulfur clouds the solution. A shorter time means a faster initial rate.

WORKED EXAMPLE (10.3)

A plot of ln(rate) against 1/T has a gradient of −5680 K.

E_a = − gradient × R = 5680 × 8.31

= 47 200 J mol^{-1} = **47.2 kJ mol^{-1}**

COMMON EXAM ERRORS

- ! Not controlling the temperature between runs — rate is very temperature-sensitive.
- ! Judging the end point inconsistently (different observers or lighting) scatters the times.
- ! Letting the total volume vary between runs, which changes the concentrations you meant to fix.
- ! The quickest runs, at the highest concentrations, carry the largest percentage error.

WORKED EXAMPLE (RATE CONSTANT)

For a step first order in iodide, rate = $k[I^-]$.

rate = 2.0×10^{-4} mol dm^{-3} s^{-1} when $[I^-]$ = 0.10 mol dm^{-3}

k = 2.0×10^{-4} ÷ 0.10 = **2.0×10^{-3} s^{-1}**

TEMPERATURE AND RATE

A rise of about 10 K roughly doubles the rate, which is why temperature is held constant when finding orders and varied carefully when finding the activation energy.

INITIAL RATE FROM A CLOCK

Because the initial rate is proportional to 1 ÷ t, you can plot 1 ÷ t against concentration directly. A straight line through the origin shows the reaction is first order in that reactant; a curve means it is not. This avoids working out the true rate for every run.

Safety: Ethanedioic (oxalic) acid is toxic; manganate(VII) stains and is an irritant. Wear eye protection.

PAG 11

pH measurement

WHAT THIS PAG COVERS

11.1 identifying unknown acids and alkalis by planning your own tests. **11.2** plotting pH titration curves for strong and weak acid–base combinations. **11.3** preparing acids and buffers and following the pH as alkali is added.

KEY TECHNIQUES

Measuring pH with a meter, a probe on a data logger, or narrow-range pH paper.

PH AND $[H^+]$

pH = −log$[H^+]$, so each whole pH unit is a tenfold change in $[H^+]$. A strong monoprotic acid is fully dissociated, so its $[H^+]$ equals its concentration; a weak acid is only slightly dissociated and reads a higher pH at the same concentration.

METHOD ESSENTIALS

1. Calibrate the pH meter against a buffer (often pH 4) before use — meters drift on storage.
2. Add alkali from a burette in small portions, swirl, and record the pH after each addition.
3. Use **smaller** increments near the rapid-change region to capture the curve, and hold the temperature steady.
4. Plot pH against the volume of alkali added.

CURVE SHAPES

Combination	Equivalence pH	Steep part
strong acid – strong base	7	~3 to 10
weak acid – strong base	> 7	~7 to 10
strong acid – weak base	< 7	~4 to 7
weak acid – weak base	—	no steep part

IDENTIFYING UNKNOWNS (11.1)

Measure the pH of each solution: a strong acid reads about 1, a weak acid 3 to 4, a weak alkali 9 to 11, a strong alkali about 13. Confirm with a reaction — a carbonate fizzes with acid, and an ammonium salt warmed with NaOH releases ammonia.

BUFFERS (11.3)

A buffer of a weak acid and its salt resists pH change: adding a little acid or alkali shifts the HA / A^- ratio only slightly. Its pH is set by pK_a and that ratio, and barely changes on dilution.

WORKED EXAMPLE — PH OF A WEAK ACID

A weak acid is only slightly dissociated, so $[H^+] = \sqrt{(K_a \times c)}$.

0.10 mol dm^{-3} ethanoic acid, $K_a = 1.8 \times 10^{-5}$:

$[H^+] = \sqrt{(1.8 \times 10^{-5} \times 0.10)} = 1.34 \times 10^{-3}$

pH = −log(1.34×10^{-3}) = **2.87**

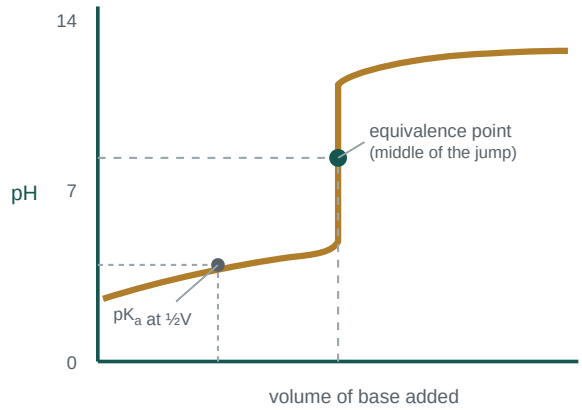
WORKED EXAMPLE — PH OF A BUFFER

0.20 mol dm^{-3} sodium ethanoate with 0.10 mol dm^{-3} ethanoic acid, $K_a = 1.8 \times 10^{-5}$.

$[H^+] = K_a \times [\text{acid}] \div [\text{salt}] = 1.8 \times 10^{-5} \times 0.10 \div 0.20$

$= 9.0 \times 10^{-6}$

pH = −log(9.0×10^{-6}) = **5.05**



A titration curve. The equivalence point is the centre of the steep, near-vertical section; the indicator's colour change must fall within it.

END POINT VS EQUIVALENCE POINT

The **end point** is where the indicator changes colour; the **equivalence point** is where stoichiometric amounts have been mixed, at the centre of the vertical section. Choose an indicator whose range lies in that section.

READING PK_A

For a weak acid, at half the neutralisation volume (½V) [HA] = $[A^-]$, so $K_a = [H^+]$ and **pH = pK_a**. Read pK_a straight off the curve at ½V.

COMMON EXAM ERRORS

- ! Confusing the end point with the equivalence point.
- ! Too few readings through the steep region, so the vertical section is poorly defined.
- ! Not calibrating or rinsing the probe, so the pH values drift.

CHOOSING THE INDICATOR

The indicator's colour-change range must lie within the steep section. **Phenolphthalein** (about 8.3 to 10) suits a weak acid–strong base; **methyl orange** (about 3.1 to 4.4) suits a strong acid–weak base; either works for strong acid–strong base; a weak acid–weak base has no sharp end point, so a meter is used.

WORKED EXAMPLE — PH OF A STRONG BASE

A strong base is fully dissociated; use $K_w = [H^+][OH^-] = 1.0 \times 10^{-14}$.

0.10 mol dm^{-3} NaOH: $[OH^-] = 0.10$

$[H^+] = 10^{-14} \div 0.10 = 1.0 \times 10^{-13}$

pH = −log(1.0×10^{-13}) = **13.0**

WHY EQUIVALENCE IS NOT ALWAYS PH 7

Only a strong acid with a strong base meets at pH 7. A weak acid with a strong base forms a salt that is slightly basic, so equivalence sits above 7; a strong acid with a weak base forms a slightly acidic salt, so it sits below 7.

Safety: Acids and alkalis are irritants, and corrosive when concentrated; wear eye protection throughout.

PAG 12

Research skills

WHAT THIS PAG COVERS

12.1 finding the mass of iron in an iron tablet, by titration with manganate(VII). **12.2** finding the copper content of brass, by titration. **12.3** investigating the effect of acid on the manganate(VII)–ethanedioic acid reaction.

KEY TECHNIQUES

Applying titration to a real sample; the self-indicating manganate(VII) titration; researching and referencing a method.

METHOD ESSENTIALS

1. Research a suitable, **referenced** method and plan the apparatus and quantities.
2. Identify the variables and the main hazards in your own risk assessment.
3. Carry out the titration or investigation and process the data.
4. Reference every source fully and consistently.

CHOOSING THE ACID (MANGANATE)

Use only dilute **sulfuric acid** to supply the H^+. Hydrochloric acid is oxidised to chlorine; nitric acid oxidises Fe^{2+}; a weak acid cannot supply enough H^+; and too little acid gives brown MnO_2 that masks the end point.

KEY EQUATIONS

$$MnO_4^- + 8H^+ + 5Fe^{2+} \rightarrow Mn^{2+} + 4H_2O + 5Fe^{3+} \quad (12.1)$$

$$[Cu(H_2O)_6]^{2+} + EDTA^{4-} \rightarrow [Cu(EDTA)]^{2-} + 6H_2O \quad (1:1)$$

PLANNING THE TITRATION

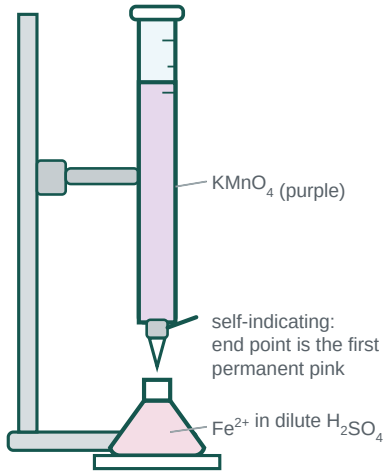
Dissolve the tablet in excess dilute sulfuric acid, make up to a known volume in a volumetric flask, and titrate measured portions. Work out a rough expected titre first, so the burette range and the concentrations are sensible.

FINDING COPPER IN BRASS (12.2)

Dissolve the brass in concentrated nitric acid in a fume cupboard, since brown nitrogen dioxide is given off, then neutralise the excess acid. Titrate the copper(II) with EDTA, which forms a 1 : 1 complex, using an indicator that changes colour as the last free copper is bound.

THE ACID INVESTIGATION (12.3)

Vary the amount of sulfuric acid and time how long the purple takes to fade in the manganate(VII)–ethanedioic acid reaction. It is slow at first, then speeds up as the Mn^{2+} product autocatalyses it, so judge the end point the same way each time.



A manganate(VII) titration is self-indicating: the end point is the first permanent pink as the purple MnO_4^- is no longer decolourised.

WORKED EXAMPLE (12.1)

10.0 cm^3 of dissolved iron needs 9.80 cm^3 of 0.0200 mol dm^{-3} $KMnO_4$.

$n(MnO_4^-) = 0.0200 \times 9.80 \div 1000 = 1.96 \times 10^{-4}$ mol

ratio 1 : 5, so $n(Fe^{2+}) = 9.80 \times 10^{-4}$ mol in 10.0 cm^3

in 100 cm^3: 9.80×10^{-3} mol; mass = $9.80 \times 10^{-3} \times 55.8$ = **0.547 g**

REFERENCING WELL

A complete reference gives the author, year, title, the source, and — for a website — the date you accessed it. Any standard style is fine; **consistency** matters more than the particular one.

COMMON EXAM ERRORS

- ! Using the wrong acid for manganate: HCl gives chlorine, HNO_3 oxidises Fe^{2+}, too little acid gives brown MnO_2.
- ! A method that could not be repeated because concentrations or volumes are missing.
- ! A bare web address given as a reference, with no author, title or date.

READING THE END POINT

Read the burette from the **top** of the meniscus for the deeply coloured manganate(VII), since the purple hides the bottom. The end point is one drop sharp — the first permanent pink — so add dropwise and swirl as you approach it.

REPORTING THE RESULT

Turn the mass of iron into a percentage of the tablet's stated mass and compare it with the label. A small shortfall can come from iron(II) being air-oxidised to iron(III) before titrating, which the manganate(VII) does not count.

Safety: Manganate(VII) stains and is an irritant; ethanedioic acid is toxic. Treat the acids as in the earlier PAGs and wear eye protection.