RP 01 Make up a volumetric solution and carry out a simple acid–base titration

APPARATUS & TECHNIQUES a d e f k

Aim. Prepare a standard solution of known concentration, then use it in an acid–base titration to find the concentration of another solution.

Making the standard solution

1. Weigh the solid accurately **by difference** on a balance.
2. Dissolve it fully in distilled water in a beaker, stirring with a glass rod.
3. Transfer to a volumetric flask through a funnel; rinse the beaker, rod, and funnel into the flask so none is lost.
4. Add distilled water to just below the graduation mark, then dropwise until the **bottom of the meniscus** sits on the line at eye level.
5. Stopper and invert several times to mix.

The titration

1. Rinse the burette with the solution it will hold, fill it, and clear the air bubble below the tap.
2. Pipette 25.0 cm^3 of the other solution (pipette rinsed with that solution) into a conical flask; rinse the flask with distilled water only.
3. Add a few drops of indicator and record the initial burette reading to 0.05 cm^3.
4. Add titrant with constant swirling; slow to dropwise near the end-point; stop at the first permanent colour change. Record the final reading.
5. Run a rough titration first, then repeat for **concordant** titres (within 0.10 cm^3). Mean the concordant titres only.

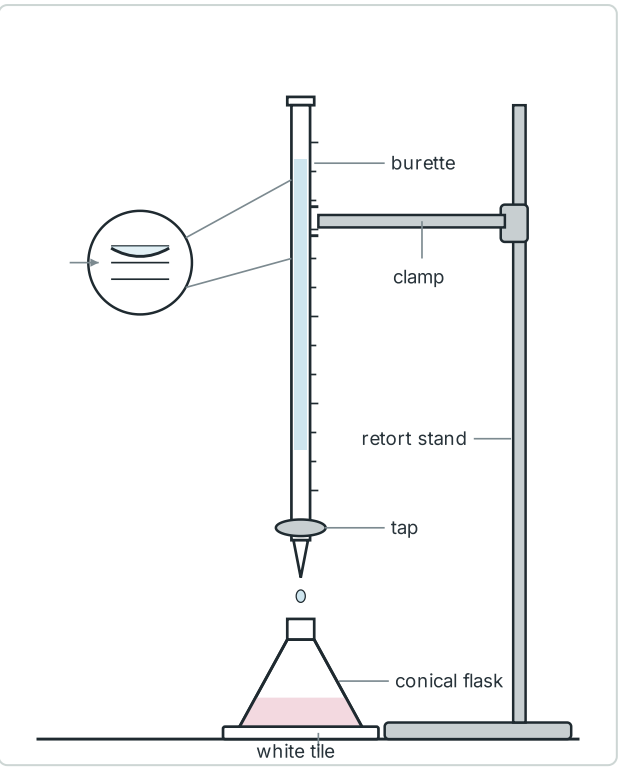
Choosing the indicator

Titration	Indicator
Strong acid – strong base	methyl orange or phenolphthalein
Strong acid – weak base	methyl orange
Weak acid – strong base	phenolphthalein
Weak acid – weak base	none — no sharp end-point

Common exam errors

Funnel left in the burette. Solution held in the funnel can drip into the burette after you take a reading, changing the volume and making the titre inaccurate. Remove the funnel before reading.

Air bubble below the tap. If the jet below the tap is not filled, that air fills with solution during the titration, so the burette reads a larger volume than was actually delivered — the titre reads too high. Run solution through to fill the tip first.



Standard titration set-up. **Inset:** read the bottom of the meniscus at eye level to avoid parallax error.

Worked example

25.0 cm^3 of NaOH needs 23.50 cm^3 of 0.100 mol dm^{-3} HCl.

$n(HCl) = 0.100 \times 23.50 \div 1000 = 2.35 \times 10^{-3}$ mol

$HCl + NaOH \rightarrow NaCl + H_2O$ (1 : 1)

$n(NaOH) = 2.35 \times 10^{-3}$ mol

$c(NaOH) = 2.35 \times 10^{-3} \div (25.0 \div 1000)$

$= 0.0940$ mol dm^{-3}

Maximise accuracy

- Read all volumes from the bottom of the meniscus at eye level.
- Rinse burette and pipette with their own solutions; rinse the flask with distilled water only.
- Use concordant titres within 0.10 cm^3; discard the rough titre.
- Burette uncertainty ±0.05 cm^3 per reading (±0.10 cm^3 per titre) — larger titres give a smaller percentage error.

Safety. Eye protection throughout. Acids and alkalis are irritant or corrosive; rinse any splashes with plenty of water.

RP 02 Measurement of an enthalpy change

APPARATUS & TECHNIQUES a d k

Aim. Use calorimetry to measure the temperature change of a reaction (for example neutralisation or a displacement reaction) and calculate the enthalpy change per mole.

Method

1. Measure a known volume of solution into an insulated polystyrene cup with a lid; record its temperature every 30 s for 2–3 minutes to set a baseline.
2. At a recorded time, add the second reactant (for example excess metal powder, or the second solution); stir continuously.
3. Keep recording the temperature every 30 s for several more minutes.
4. Plot temperature against time. **Extrapolate** the cooling line back to the moment of mixing to find the corrected maximum temperature, and hence ΔT.

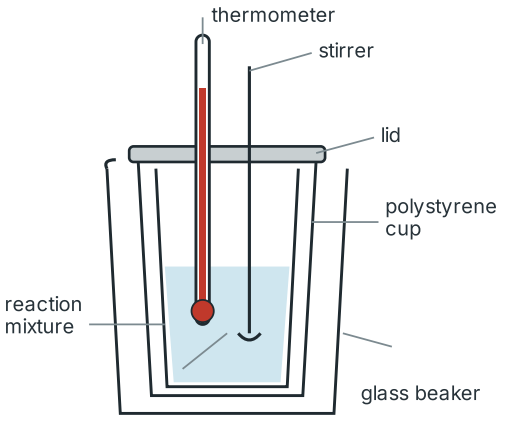
Maximise accuracy

- Lid and polystyrene cup reduce heat loss; the beaker adds insulation and support.
- Stir so the temperature is even throughout.
- Use excess of one reactant so the other is fully used.
- Extrapolation corrects for heat lost while the reaction proceeds.

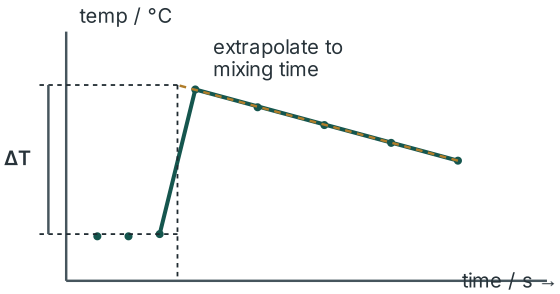
Common exam errors

Heat loss to the surroundings. The measured ΔT is too small, so ΔH is underestimated. Use a lid, insulation, and extrapolate the cooling curve back to the time of mixing.

Assuming a complete, instant reaction. Reactions take time, so the true peak is missed. Reading the highest recorded value (not the extrapolated value) gives an inaccurate ΔH.



Polystyrene-cup calorimeter, insulated in a glass beaker with a lid.



Extrapolate the cooling line back to the mixing time for the true ΔT.

Worked example

$q = mc\Delta T$

50.0 g solution, $\Delta T = 5.0$ °C, $c = 4.18$ J g^{-1} K^{-1}

$q = 50.0 \times 4.18 \times 5.0 = 1045$ J

moles of limiting reactant n = 0.0050 mol

$\Delta H = -q \div n = -1045 \div 0.0050$

$= -209\,000$ J mol^{-1} $= -209$ kJ mol^{-1}

Safety. Eye protection. Many reagents are irritant or corrosive; some reactions get hot. Add solids carefully to avoid splashing.

RP 03 Investigate how the rate of a reaction changes with temperature

APPARATUS & TECHNIQUES a b k

Aim. Use the sodium thiosulfate–hydrochloric acid "disappearing cross" reaction to investigate the effect of temperature on the rate of reaction.

Method

1. Measure a fixed volume of sodium thiosulfate solution into a conical flask standing on paper marked with a cross.
2. Warm the flask in a water bath to a chosen temperature and record it.
3. Add a fixed volume of dilute hydrochloric acid and start the timer.
4. Looking down through the solution, stop the timer when the cross can no longer be seen (sulfur clouds the mixture).
5. Repeat across a range of temperatures, keeping all volumes and concentrations the same.

$Na_2S_2O_3 + 2HCl \rightarrow 2NaCl + S + SO_2 + H_2O$

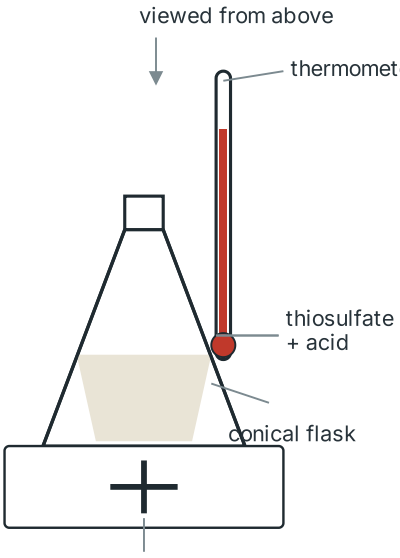
Analysing the results

Rate is proportional to 1 ÷ time. Plot 1/time (y-axis) against temperature (x-axis): the rate rises steeply with temperature, roughly doubling for every 10 °C rise, because more molecules exceed the activation energy.

Common exam errors

Judging the end-point. Deciding when the cross "disappears" is subjective. The same person should judge every run, using the same cross, flask, and depth of solution.

Temperature drifting. The mixture cools (or warms) during timing, so record the temperature at the start and end and use the mean — and keep everything except temperature constant.



The cross is viewed from above and disappears as sulfur is precipitated.

Worked example

At 40 °C the cross disappears after 50 s.

rate ∝ 1 ÷ t = 1 ÷ 50 = 0.020 s^{-1}

Plot 1/t against temperature; a steeper rise at higher temperatures shows a faster rate.

Why temperature speeds it up

- Particles move faster and collide more often.
- A greater proportion of collisions have energy ≥ the activation energy — the larger effect.

Safety. Eye protection. Sulfur dioxide is toxic and irritant — work in a ventilated area and take care if asthmatic. Hydrochloric acid is irritant.

RP 04 Test-tube reactions to identify cations and anions

APPARATUS & TECHNIQUES d k IONS: GROUP 2, NH_4^+ · HALIDES, OH^-, CO_3^{2-}, SO_4^{2-}

Aim. Use simple test-tube reactions to identify common cations and anions from their characteristic reactions and precipitates.

Cation tests

Ion	Test	Observation
Group 2 (M^{2+})	add NaOH(aq)	☐ white precipitate (hydroxide); less soluble up to Mg^{2+}
NH_4^+	add NaOH(aq) and warm	pungent gas turns damp red litmus blue (ammonia)

Anion tests (in this order)

Ion	Test	Observation
CO_3^{2-}	add dilute acid	effervescence; gas turns limewater milky (CO_2)
SO_4^{2-}	acidify with HCl, add $BaCl_2$	☐ white precipitate ($BaSO_4$)
Cl^-	acidify with dilute HNO_3, add $AgNO_3$	☐ white precipitate (soluble in dilute NH_3)
Br^-		☐ cream precipitate (soluble in conc. NH_3)
I^-		☐ yellow precipitate (insoluble in conc. NH_3)
OH^-	test with red litmus	turns blue (alkaline)



Halide ions give distinctive precipitate colours with acidified silver nitrate.

Common exam errors

Wrong order of tests. Carbonate also gives precipitates with Ba^{2+} and Ag^+, so test for and remove it first by acidifying — otherwise you get false positives for sulfate and halide.

Using HCl before silver nitrate. Hydrochloric acid adds chloride ions, giving a false white precipitate. Acidify with dilute **nitric** acid before adding silver nitrate.

Safety. Eye protection. Silver nitrate stains skin and is an irritant; barium compounds are toxic; acids and alkalis are irritant or corrosive.

RP 05 Distillation of a product from a reaction

APPARATUS & TECHNIQUES b d k

Aim. Separate a volatile liquid product from a reaction mixture by simple distillation — for example distilling an aldehyde out of an oxidising mixture as it forms.

Method

1. Place the reaction mixture and a few anti-bumping granules in a round-bottomed (or pear-shaped) flask.
2. Fit a still head with a thermometer, its bulb level with the side-arm, and a Liebig condenser sloping downwards.
3. Connect the condenser water so it flows counter-current — in at the bottom, out at the top.
4. Heat gently with a water bath or electric heater (flammable liquids — no naked flame).
5. Collect the fraction that distils over within the boiling range of the product in the receiving flask.

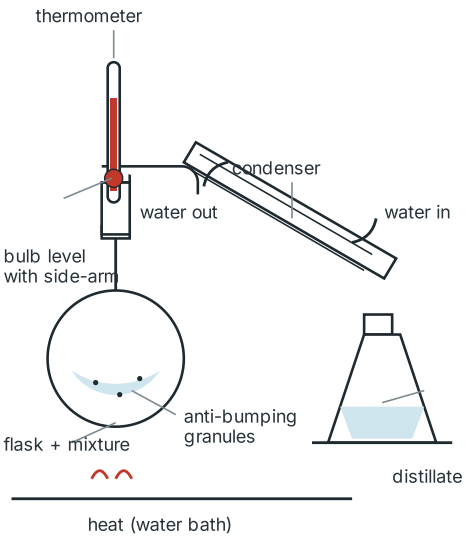
Common exam errors

Thermometer in the wrong place. The bulb must sit level with the side-arm so it reads the temperature of the vapour entering the condenser; too low or too high gives the wrong boiling point.

Water flowing the wrong way. Water must enter at the bottom and leave at the top so the condenser stays full and cools efficiently (counter-current). Never seal the apparatus — trapped vapour builds up pressure.

Key idea

For oxidation of a primary alcohol, distilling the aldehyde off as it forms removes it before it can be oxidised further to a carboxylic acid.



Simple distillation. Thermometer bulb at the side-arm; condenser water counter-current.

Safety. Eye protection. Organic liquids are flammable and often harmful — heat with a water bath or electric heater, never a naked flame, and work in a fume cupboard where required.

RP 06 Tests for alcohol, aldehyde, alkene and carboxylic acid

APPARATUS & TECHNIQUES b d k

Aim. Use chemical tests to distinguish the alkene, alcohol, aldehyde, and carboxylic acid functional groups.

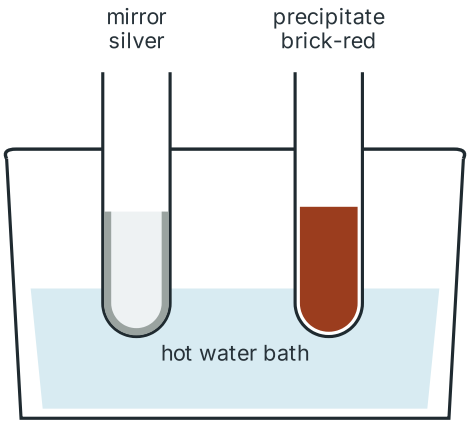
Tests and observations

Group	Reagent	Positive result
Alkene (C=C)	bromine water	orange decolourises to colourless
Carboxylic acid	sodium carbonate	effervescence; gas turns limewater milky (CO_2)
1°/2° alcohol	warm with acidified $K_2Cr_2O_7$	orange to green (tertiary stays orange)
Aldehyde	warm with Tollens' reagent	silver mirror forms (ketone: no change)
Aldehyde	warm with Fehling's solution	blue to brick-red precipitate

Common exam errors

Heating Tollens' or Fehling's over a flame. They must be warmed gently in a water bath — the organics are flammable, and gentle heat gives the clean silver mirror or brick-red precipitate.

Confusing aldehyde with carboxylic acid. Both contain C=O chemistry, but only the aldehyde gives a silver mirror, and only the acid releases CO_2 with carbonate.



Tollens' and Fehling's tests are warmed in a water bath, not heated directly.

Distinguishing the alcohols

- Primary → oxidised to an aldehyde, then a carboxylic acid.
- Secondary → oxidised to a ketone.
- Tertiary → not oxidised; dichromate stays orange.

Safety. Eye protection. Many reagents are irritant, corrosive, or toxic; the organics are flammable. Tollens' reagent must be used fresh and not stored.

RP 07 Measuring rate by an initial-rate method and by continuous monitoring

APPARATUS & TECHNIQUES a k l

Aim. Find a reaction rate two ways: an initial-rate (clock) method that times a fixed early change, and a continuous method that follows a property throughout the reaction.

Initial-rate (clock) method

1. Mix the reagents (for example H_2O_2, KI, thiosulfate, and starch) and start the timer.
2. Time how long until a sudden, fixed change appears — here the blue-black colour when the thiosulfate is used up.
3. The initial rate is proportional to 1 ÷ time.
4. Repeat, changing one reactant's concentration at a time, to find the order with respect to each.

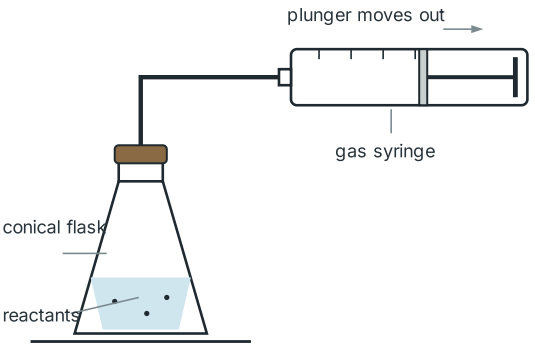
Continuous monitoring

1. Follow a property as the reaction proceeds: gas volume in a gas syringe, mass loss on a balance, or colour by colorimetry.
2. Record the measurement at regular time intervals.
3. Plot the property against time; the gradient of the tangent gives the rate, which falls as reactants are used up.

Common exam errors

Gas lost before the bung is in. Any gas escaping before the bung is fitted is not measured, so the volume — and the rate — is underestimated. Add the last reactant and stopper quickly.

No plug for the mass-loss method. Use a loose cotton-wool plug so gas escapes but spray does not, otherwise lost liquid exaggerates the mass change.



Continuous monitoring by gas collection. Mass loss and colorimetry work the same way.

Worked example — finding an order

Doubling [A] makes the initial rate 4× faster.

rate ∝ $[A]^n$, so $2^n = 4 \rightarrow n = 2$

The reaction is second order with respect to A.

Safety. Eye protection. Hydrogen peroxide and many reagents are irritant or oxidising; some reactions produce gas — work in a ventilated space and do not seal the apparatus.

RP 08 Measuring the EMF of an electrochemical cell

APPARATUS & TECHNIQUES j k

Aim. Build electrochemical cells from two half-cells, measure the EMF (cell potential), and compare it with values from standard electrode potentials.

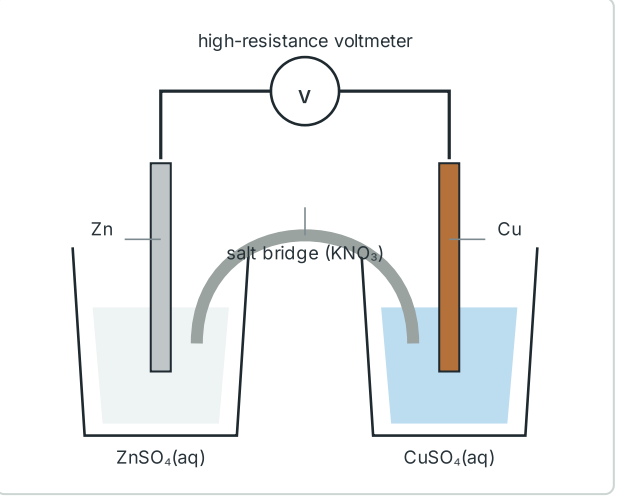
Method

1. Set up two half-cells — for example a zinc rod in zinc sulfate solution and a copper rod in copper sulfate solution. Clean both electrodes with emery paper.
2. Connect the solutions with a salt bridge (filter paper soaked in potassium nitrate).
3. Connect the electrodes to a high-resistance voltmeter and record the EMF.
4. For standard EMF, use 1.00 mol dm^{-3} solutions at 298 K. Investigate the effect of changing concentration.

Common exam errors

Using an ordinary voltmeter. A high-resistance voltmeter draws almost no current, so the cell is not discharging and you measure the maximum potential difference (the EMF). If current flows, the reading falls and changes over time.

The wrong salt-bridge electrolyte. The salt bridge completes the circuit and balances charge, so it must contain ions that do not react with either solution — potassium nitrate, not (for example) chloride with silver ions.



A zinc–copper cell. The salt bridge completes the circuit; the voltmeter reads the EMF.

Worked example

$E°_{cell} = E°(\text{positive}) - E°(\text{negative})$

$E°(Cu^{2+}/Cu) = +0.34$ V, $E°(Zn^{2+}/Zn) = -0.76$ V

$E°_{cell} = +0.34 - (-0.76) = +1.10$ V

Copper is the positive electrode; zinc is oxidised.

Safety. Eye protection. Copper and zinc salts are harmful to aquatic life and irritant; wash hands after handling.

RP 09 How pH changes during weak-acid and weak-base titrations

APPARATUS & TECHNIQUES a c d k

Aim. Follow the pH as base is added to acid to produce titration curves for a weak acid with a strong base, and a strong acid with a weak base, and read key features from them.

Method

1. Calibrate the pH meter with buffer solutions of known pH.
2. Place a known volume of acid in a beaker with the pH probe; stir.
3. Add base from a burette in small portions, recording the pH after each addition.
4. Add smaller portions near the equivalence point where the pH changes fastest.
5. Continue past the equivalence point, then plot pH against volume of base added. Repeat for each acid–base combination.

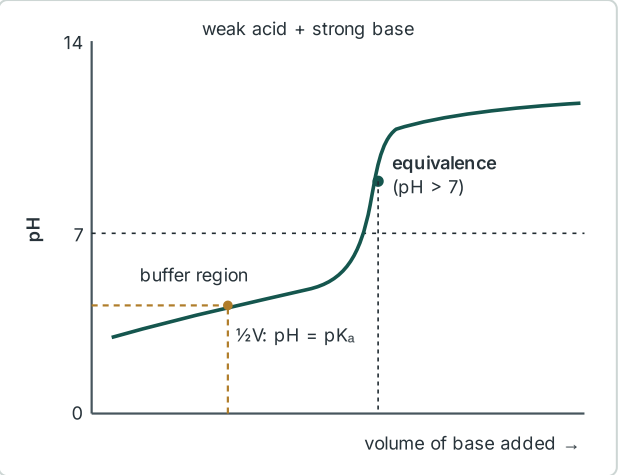
Reading the curve

Weak acid with strong base: equivalence above pH 7, with a buffer region before it; at half-neutralisation pH = pK_a; use phenolphthalein. Strong acid with weak base: equivalence below pH 7; use methyl orange. Choose an indicator whose colour-change range lies on the steep section.

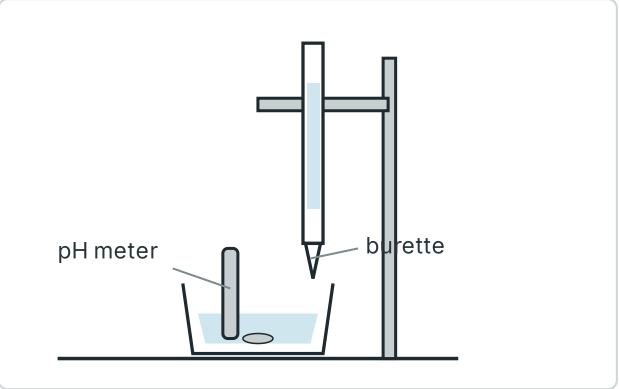
Common exam errors

Forgetting to calibrate. An uncalibrated pH meter gives readings that are systematically wrong; calibrate with buffers first.

Reading the equivalence point wrongly. It lies at the midpoint of the near-vertical section, not where the indicator first changes; for a weak acid–strong base it is above pH 7, not at 7.



Weak acid–strong base curve, showing the buffer region, half-neutralisation, and equivalence point.



Add base from the burette; track pH with a calibrated meter.

Safety. Eye protection. Acids and bases are irritant or corrosive; rinse splashes with plenty of water.

RP 10 Prepare a pure organic solid and a pure organic liquid

APPARATUS & TECHNIQUES a b d g h k

Aim. Prepare an organic product, purify it, and check its purity — a solid by recrystallisation and melting point, a liquid by washing, drying, and redistillation.

Purifying a solid (recrystallisation)

1. Dissolve the crude solid in the **minimum volume of hot solvent**.
2. Filter the hot solution to remove insoluble impurities.
3. Cool to crystallise the product; soluble impurities stay in solution.
4. Filter the crystals under reduced pressure (Büchner), wash with a little cold solvent, and dry.
5. Check purity by melting point: pure gives a sharp value at the literature figure; impurities lower and broaden the range.

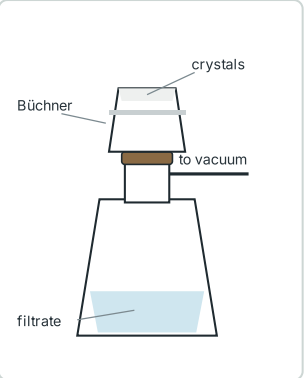
Purifying a liquid

Shake the product with a washing solution in a separating funnel, let the layers settle, and run off the lower layer. Dry with an anhydrous drying agent, then redistil, collecting the correct boiling range.

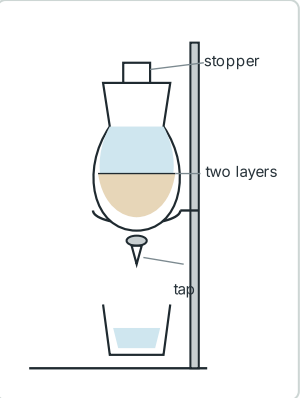
Common exam errors

Too much hot solvent. Excess solvent keeps product dissolved on cooling, so the yield drops. Use the minimum volume that just dissolves the solid when hot.

Washing with warm solvent. Wash crystals with a little **cold** solvent so the product does not redissolve while impurities are rinsed away.



Filtration under reduced pressure.



Separating immiscible layers.

Melting point as a purity test

- Pure solid: sharp melting point at the literature value.
- Impure solid: melts lower and over a wider range.

Safety. Eye protection. Organic solvents are flammable and harmful; heat with a water bath, not a flame, and work in a fume cupboard where required.

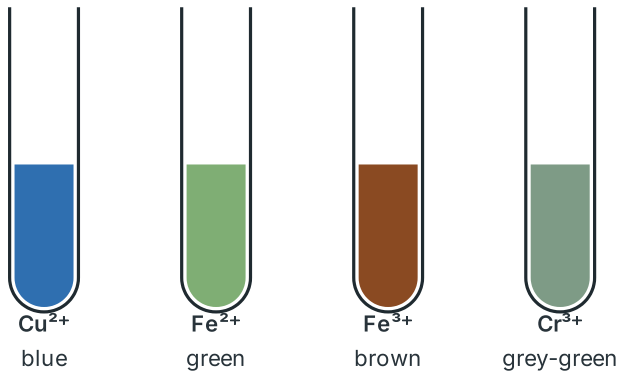
RP 11 Test-tube reactions to identify transition metal ions

APPARATUS & TECHNIQUES b d k

Aim. Identify transition metal ions in solution from the colour of the hydroxide precipitate formed with sodium hydroxide and with ammonia, and whether it dissolves in excess.

Reactions with sodium hydroxide and ammonia

Ion (colour)	+ NaOH (dropwise)	+ excess NaOH	+ NH_3 (dropwise)	+ excess NH_3
Cu^{2+} (blue)	blue precipitate	insoluble	blue precipitate	dissolves, deep blue solution
Fe^{2+} (pale green)	green precipitate	insoluble	green precipitate	insoluble (browns in air)
Fe^{3+} (yellow)	brown precipitate	insoluble	brown precipitate	insoluble
Mn^{2+} (pale)	light brown precipitate	insoluble	light brown precipitate	insoluble (darkens in air)
Cr^{3+} (green)	grey-green precipitate	dissolves, dark green	grey-green precipitate	dissolves, violet solution



Characteristic hydroxide precipitate colours.

Common exam errors

Adding alkali all at once. Add NaOH or ammonia dropwise first, then in excess — the point is to see whether the precipitate redissolves (Cr^{3+} does; Cu^{2+} dissolves only in excess ammonia).

Observing Fe^{2+} too late. Iron(II) hydroxide is oxidised to brown iron(III) hydroxide on standing in air, so record the green colour promptly.

Safety. Eye protection. Sodium hydroxide is corrosive and ammonia is irritant with a pungent vapour; transition metal salts are harmful.

RP 12 Separation of species by thin-layer chromatography

APPARATUS & TECHNIQUES i k

Aim. Separate the components of a mixture by thin-layer chromatography (TLC) and identify them by calculating R_f values.

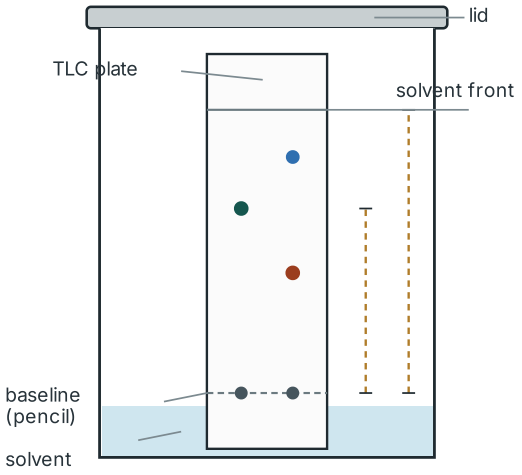
Method

1. Draw a **pencil** baseline near the bottom of the TLC plate and spot the samples on it as small, concentrated spots; let them dry.
2. Add solvent to the tank to a depth **below the baseline**, then stand the plate inside and cover the tank.
3. Let the solvent rise by capillary action; components separate by their relative affinity for the stationary phase and solubility in the mobile phase.
4. Remove the plate before the solvent reaches the top, mark the solvent front in pencil, and let it dry.
5. Reveal colourless spots under UV light or with a locating agent, then measure each spot.

Common exam errors

Drawing the baseline in ink. Ink dissolves and travels up the plate with the solvent, ruining the result. Always use pencil.

Solvent above the baseline. If the solvent starts above the spots, the samples dissolve straight into it instead of separating. Keep the solvent level below the baseline, and cover the tank so the atmosphere is saturated.



Developing tank. The dashed lines show the distances used for R_f: spot (left) and solvent front (right).

Worked example — R_f value

R_f = distance moved by spot ÷ distance moved by solvent

spot moves 2.4 cm; solvent front moves 6.0 cm

$R_f = 2.4 \div 6.0 = 0.40$

R_f is always less than 1 and is constant for the same conditions.

Safety. Eye protection. Chromatography solvents are flammable and harmful; keep the tank covered and work in a ventilated area or fume cupboard.