IGCSE CHEMISTRY 0620 · CHAPTER 12 NOTES · ~15 MIN READ

Experimental techniques & chemical analysis

The practical skills behind every Paper 6 question: which apparatus to use, how to separate and purify, how to read a chromatogram and run a titration, and every test for ions and gases.

Apparatus | Separation methods | Chromatography | Purity | Tests for ions & gases

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No tag = **Core** (everyone) EXTENDED Extended only highlight = comes up again and again

Test yourself at the end

IN 30 SECONDS

- Choose apparatus for the **accuracy** you need: a burette or pipette is more accurate than a measuring cylinder.
- **Separation** depends on the mixture: filter (insoluble solid), crystallise (dissolved solid), distil (solvent), fractional distillation (liquids), chromatography (dyes).
- A **pure** substance has a **sharp** melting or boiling point. An impure one melts over a **range**.
- Tests: **flame test**, **NaOH and NH_3** for cations, **acidify then add a reagent** for anions, and a test for each **gas**.

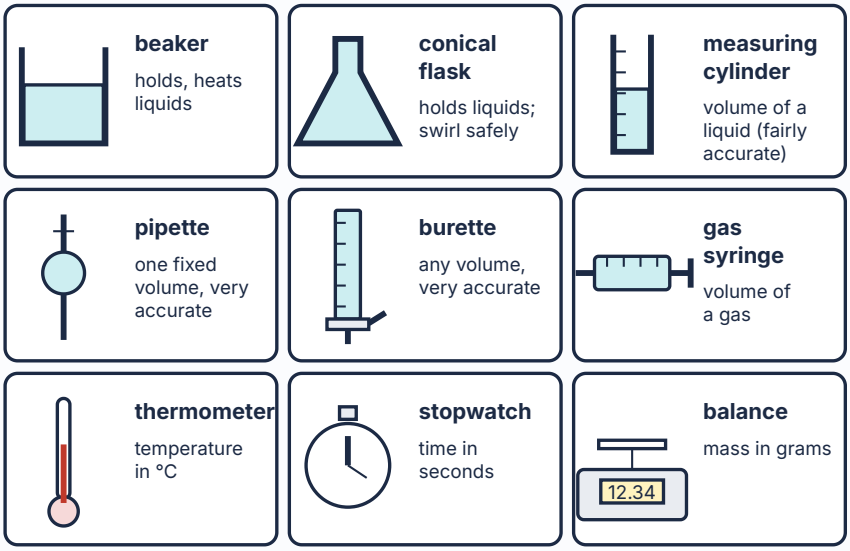
BY THE END YOU CAN

- Choose apparatus to measure time, temperature, mass and volume, and use the key words (solvent, solute, residue, filtrate).
- Select and describe separation methods, and use melting and boiling points to judge purity.
- Interpret chromatograms and calculate Rf values. EXT
- Describe an acid–alkali titration and find the end-point.
- Carry out and interpret tests for cations, anions and gases.

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1 Measuring and experimental design

Choosing apparatus



Common apparatus. Use the one that matches the accuracy you need.

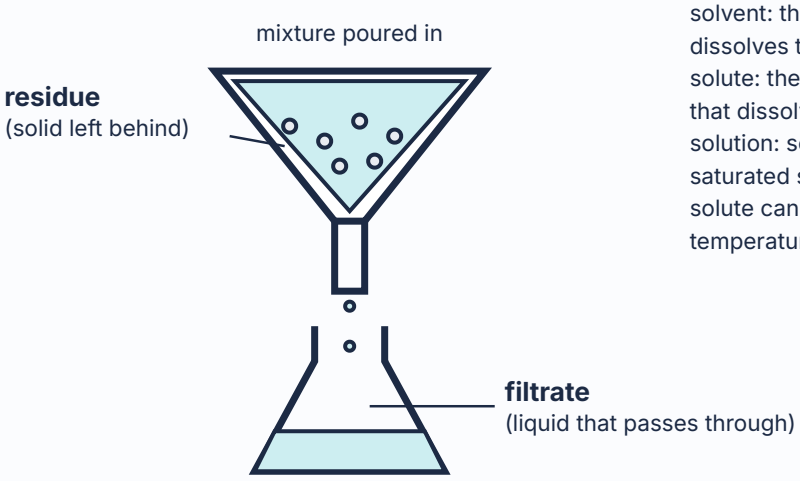
Measurement	Apparatus	Notes
time	stopwatch	read in seconds
temperature	thermometer	read in °C
mass	balance	read in grams
volume of liquid	measuring cylinder	fairly accurate — use for approximate volumes
exact, fixed volume	pipette	very accurate, one volume only (e.g. 25.0 cm^3)
any volume, accurately	burette	very accurate — read the bottom of the meniscus
volume of gas	gas syringe	accurate; or collect over water in an upturned measuring cylinder

- A **beaker** or **conical flask** is for holding and mixing — the markings are only approximate.
- A **gas syringe** is more accurate than collecting a gas over water, but can stick. Over water, a gas that dissolves (such as CO_2) cannot be collected accurately.

Fair tests

- Change **one** thing (the independent variable), measure **one** thing (the dependent variable), and keep everything else the **same** (the control variables).
- Repeat the experiment and calculate a **mean** to improve reliability. Ignore results that are obviously wrong (anomalies).

Key words



solvent: the liquid that dissolves the solute
solute: the substance that dissolves
solution: solute + solvent
saturated solution: no more solute can dissolve at that temperature

separates an insoluble solid from a liquid

Filtration. The insoluble solid that stays on the paper is the residue; the liquid that goes through is the filtrate. The panel on the right defines the main words.

Solvent — the liquid that dissolves a substance. **Solute**

Saturated solution — a solution in which no more solute can dissolve at that temperature.

Residue — the solid left on the filter paper. **Filtrate**

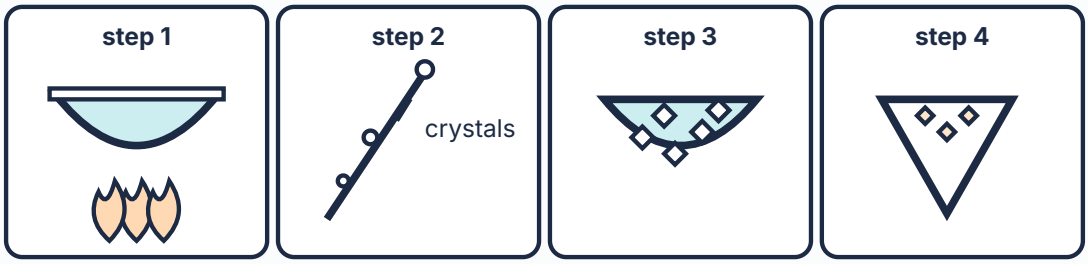
2 Separating and purifying

Choosing the method

Mixture	Method	Why it works
insoluble solid in a liquid (sand in water)	**filtration**	the solid is too big to pass through the paper
a dissolved solid, solid wanted (salt from sea water)	**crystallisation**	heat to evaporate water, then cool
a solvent from a solution (pure water from salt water)	**simple distillation**	the solvent boils off and condenses; the solute stays
liquids that mix, with different boiling points (ethanol and water)	**fractional distillation**	the liquid with the lower boiling point evaporates first
mixture of dyes or colours	**chromatography**	different solubilities → move at different speeds

Crystallisation and distillation

getting crystals from a solution

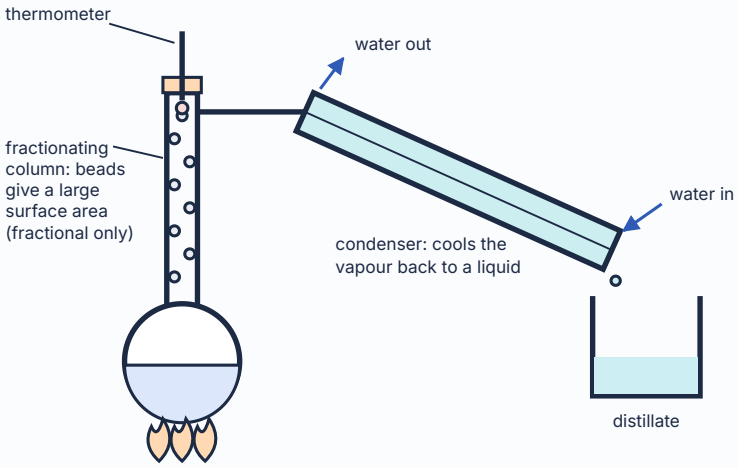


heat gently to evaporate some of the water	test: dip in a cold glass rod; crystals form = saturated	leave to cool slowly: crystals form (bigger)	filter, wash with a little cold water, dry on filter paper

stop heating when the solution is saturated: do not boil it dry
(hydrated crystals would be damaged and the crystals would be tiny)

Crystallisation. Heat to evaporate some of the water, test whether the solution is saturated, leave it to cool slowly, then filter, wash and dry.

- **Crystallisation:** heat gently to evaporate **some** water. Test by dipping in a cold glass rod: **crystals form** when the solution is **saturated**. Leave to **cool slowly** (bigger crystals), then **filter**, wash with a little **cold** water and dry between filter papers.
- Do **not** evaporate to dryness: the crystals could be damaged or lose their water of crystallisation.



simple distillation: no column, e.g. pure water from salt water
fractional distillation: separates liquids with different boiling points

Simple and fractional distillation. In fractional distillation the column of beads gives a large surface area, so the liquids separate better.

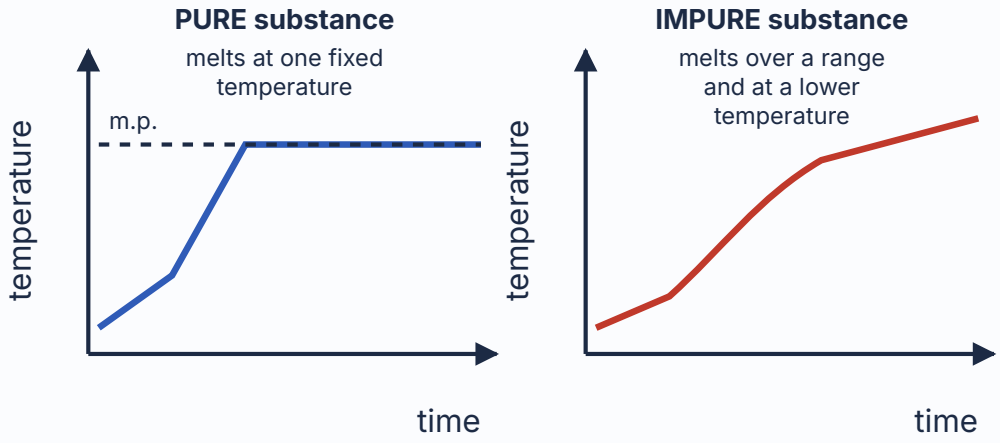
- **Distillation:** the solution is heated, the **solvent evaporates**, and the vapour is **condensed** in a water-cooled condenser. The pure liquid (the **distillate**) is collected. The solute stays in the flask.
- **Fractional distillation:** the mixture of liquids is heated. The one with the **lowest boiling point** evaporates and reaches the top of the **fractionating column** first. Used for ethanol from water, crude oil, and liquid air.
- The **thermometer bulb** is at the **side arm**, level with the exit, so it reads the temperature of the vapour.

WORKED EXAMPLE

A mixture contains sand, salt and iron filings. Describe how to separate all three.

1. use a **magnet** to remove the iron filings
2. add **water** and stir to dissolve the salt, then **filter**: sand is the residue
3. **evaporate the filtrate (crystallise) to get the salt**

Purity



same idea for boiling: a pure liquid boils at one sharp temperature (water: 100 °C)
impurities lower the melting point and raise the boiling point

Melting a pure and an impure substance. A pure substance melts at one fixed temperature. An impure one melts over a range and at a lower temperature.

- A **pure substance** has a **sharp, fixed** melting point and boiling point. **Impurities** make a substance melt over a **range of temperatures** and at a **lower** temperature than the pure substance. They **raise** the boiling point.
- Examples: pure water melts at 0 °C and boils at 100 °C. Salt water boils above 100 °C.
- Purity matters: foods and medicines must be pure so that they are safe.

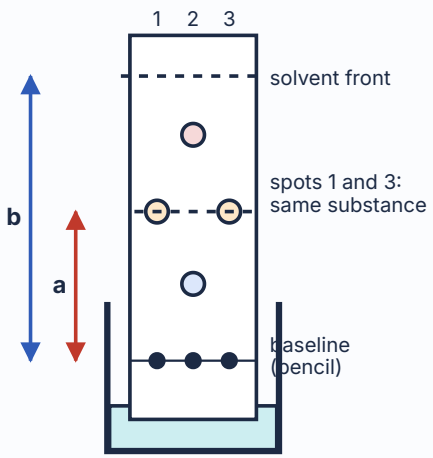
COMMON MISTAKE

Saying that "pure" means "natural" or "has no additives". In chemistry, **pure** means it is a **single substance** with nothing else mixed in.

3 Chromatography

Separating dyes

paper chromatography



1 draw the baseline in pencil
2 put a spot of each sample on it
3 stand it in solvent (below the line)
4 remove when the solvent is near the top; mark the solvent front

Rf = a ÷ b (never more than 1)

a = distance moved by the spot
b = distance moved by the solvent
e.g. 3.0 cm ÷ 6.0 cm = 0.50

colourless spots: spray with a locating agent first (Extended)

Paper chromatography. A pencil baseline carries spots of each sample. The solvent rises and the colours separate. Spots 1 and 3 have moved the same distance, so they are the same substance.

- **Method:** draw a **pencil** baseline (pencil does not dissolve). Put a **spot** of each sample on it. Stand the paper in a **solvent** with the **baseline above the solvent level**. When the solvent is near the top, remove the paper and mark the **solvent front**.
- **Reading it:** each spot is a **different substance**. A pure substance gives **one spot**. If two spots from different samples are at the **same height**, they are the **same substance**.
- Substances separate because they are **different in how well they dissolve in the solvent** and how strongly they stick to the paper.
- EXT **Colourless substances** (such as amino acids) are made visible by spraying with a **locating agent**.

EXT $R_f = \frac{\text{distance moved by the spot}}{\text{distance moved by the solvent}}$

no units; always between 0 and 1

WORKED EXAMPLE

EXT **A spot moves 3.6 cm and the solvent front moves 7.2 cm. Calculate its R_f.**

1. $R_f = 3.6 \div 7.2$
2. **= 0.50**

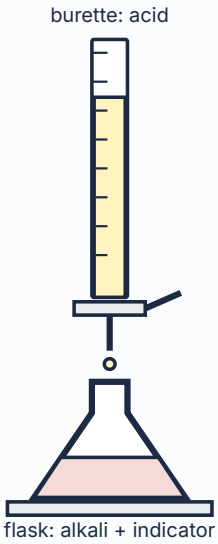
COMMON MISTAKE

Drawing the baseline in **ink**, or placing the solvent level above the baseline. Both let the sample wash into the solvent.

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4 Acid–alkali titration

The method



1 rinse the burette with acid, fill it, read it
2 pipette 25.0 cm^3 of alkali into the flask
3 add 2–3 drops of indicator
4 add acid slowly, swirling the flask
5 stop at the first permanent colour change (the end-point), read the burette
6 repeat until two titres agree (within 0.10 cm^3)

methyl orange: red in acid, yellow in alkali
phenolphthalein: colourless in acid, pink in alkali

calculate: moles = concentration × volume (dm^3)
use the equation to compare moles of acid and alkali
concentration = moles ÷ volume

The titration apparatus and method. Acid goes in the burette. A known volume of alkali and a few drops of indicator go in the flask on a white tile.

- **1.** Rinse the burette with the acid, fill it and read the starting volume.
- **2.** Use a **pipette** to measure 25.0 cm^3 of the alkali into a conical flask. Add 2–3 drops of **indicator**.
- **3.** Add the acid **slowly**, swirling the flask, until the **end-point** — the first **permanent** colour change.
- **4.** Read the final volume. The **titre** = final reading − starting reading.
- **5.** Repeat until you get **two or three titres within 0.10 cm^3** of each other (**concordant** results). Use the **mean** of those.
- **Indicators:** methyl orange (red in acid, yellow in alkali) or phenolphthalein (colourless in acid, pink in alkali). The white tile makes the colour change easier to see.

WORKED EXAMPLE

Titres: 24.30, 23.40, 23.50 and 23.45 cm^3. Find the mean titre. Then find the concentration of 25.0 cm^3 of sodium hydroxide neutralised by this volume of 0.100 mol/dm^3 HCl.

1. the first (24.30) is not concordant, so ignore it
2. mean = (23.40 + 23.50 + 23.45) ÷ 3 = 70.35 ÷ 3 = **23.45 cm^3**
3. n(HCl) = 0.100 × 23.45 ÷ 1000 = 0.002345 mol
4. 1 : 1 ratio, so n(NaOH) = 0.002345 mol
5. **c(NaOH) = 0.002345 ÷ 0.0250 = 0.0938 mol/dm^3 (more on calculations in stoichiometry)**

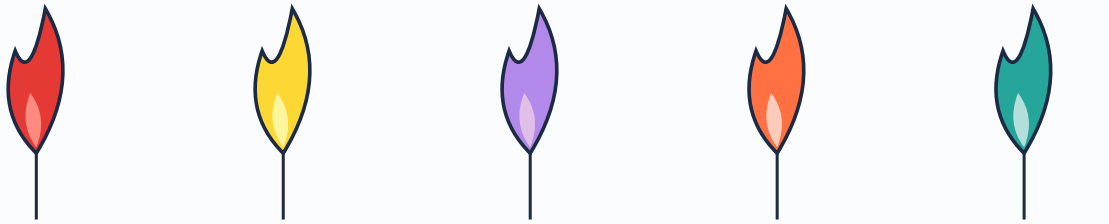
COMMON MISTAKE

Including the first rough titre in the mean. Use only **concordant** values.

5 Tests for ions and gases

Flame tests

flame tests: the colour tells you the metal ion



Li^+	Na^+	K^+	Ca^{2+}	Cu^{2+}
lithium	sodium	potassium	calcium	copper
crimson red	yellow	lilac	orange-red	blue-green

clean a wire loop in acid, dip it in the sample, hold it in a blue Bunsen flame

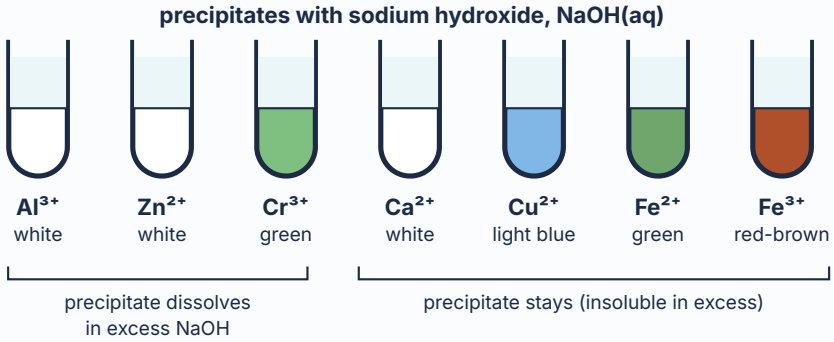
Flame test colours: lithium red, sodium yellow, potassium lilac, calcium orange-red and copper blue-green.

- Clean a wire loop in acid, dip it in the sample and hold it in a **blue Bunsen flame**.

Ion	Flame colour
lithium Li^+	crimson red
sodium Na^+	yellow
potassium K^+	lilac
calcium Ca^{2+}	orange-red
copper(II) Cu^{2+}	blue-green

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Cations: sodium hydroxide and ammonia



with aqueous ammonia, $NH_3(aq)$: the same precipitates form, but only Zn^{2+} and Cu^{2+} precipitates dissolve in excess (Cu^{2+} gives a deep blue solution)
Ca^{2+}: no precipitate or only a trace

ammonium ions: warm with NaOH(aq): ammonia gas is given off (turns damp red litmus paper blue)

Precipitates with sodium hydroxide. The colour identifies the ion; whether it dissolves in excess separates similar ones.

- Add **NaOH(aq)** drop by drop, then in **excess**, to the solution. Then repeat with **NH_3(aq)**.

Ion	NaOH(aq)	In excess NaOH	NH_3(aq)	In excess NH_3
aluminium Al^{3+}	white precipitate	dissolves (colourless)	white precipitate	stays
calcium Ca^{2+}	white precipitate	stays	no precipitate or trace	–
chromium(III) Cr^{3+}	green precipitate	stays	green precipitate	stays
copper(II) Cu^{2+}	light blue precipitate	stays	light blue precipitate	dissolves → deep blue solution
iron(II) Fe^{2+}	green precipitate	stays	green precipitate	stays
iron(III) Fe^{3+}	red-brown precipitate	stays	red-brown precipitate	stays
zinc Zn^{2+}	white precipitate	dissolves (colourless)	white precipitate	dissolves (colourless)

- **Ammonium ions (NH_4^+):** warm with NaOH(aq) → **ammonia gas** (pungent; turns damp red litmus **blue**).

MEMORY TRICK

Al and Zn are the two **white** precipitates that **dissolve** in excess NaOH. To split them, add **ammonia**: **Zn dissolves**, **Al does not**.

Anions

test for halide and sulfate ions: acidify, then add a solution



Cl^-
white precipitate
add $AgNO_3(aq)$

Br^-
cream precipitate
add $AgNO_3(aq)$

I^-
yellow precipitate
add $AgNO_3(aq)$

SO_4^{2-}
white precipitate
add $Ba(NO_3)_2(aq)$

halides: acidify with dilute nitric acid, then add silver nitrate
sulfate: acidify with dilute nitric (or hydrochloric) acid, then add barium nitrate (or chloride)
(the acid removes carbonate ions, which would also give a precipitate)

carbonate: add dilute acid → fizzing; the gas turns limewater milky (CO_2)
nitrate: warm with NaOH(aq) and aluminium foil → ammonia gas (turns damp red litmus blue)

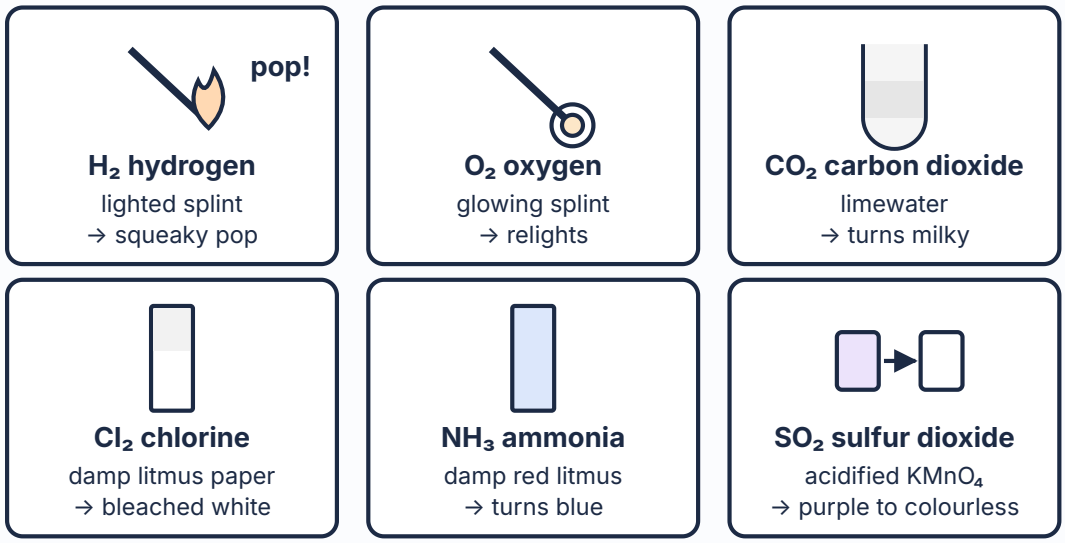
Tests for halide and sulfate ions. Acidify first, then add the reagent.

Ion	Test	Positive result
carbonate CO_3^{2-}	add dilute acid	fizzing; gas turns limewater milky
chloride Cl^-	acidify with dilute nitric acid, then add silver nitrate	white precipitate
bromide Br^-	acidify with dilute nitric acid, then add silver nitrate	cream precipitate
iodide I^-	acidify with dilute nitric acid, then add silver nitrate	yellow precipitate
sulfate SO_4^{2-}	acidify with dilute nitric (or hydrochloric) acid, then add barium nitrate (or chloride)	white precipitate
nitrate NO_3^-	add NaOH(aq) and aluminium foil, warm	ammonia gas (turns damp red litmus blue)
sulfite SO_3^{2-}	add dilute hydrochloric acid, warm	sulfur dioxide (turns acidified potassium manganate(VII) from purple to colourless)

- **Why acidify first?** The acid removes carbonate (and hydroxide) ions, which would also give a precipitate with silver or barium ions and give a false result.

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Gases



Tests for six gases: H_2, O_2, CO_2, Cl_2, NH_3 and SO_2.

Gas	Test	Result
hydrogen H_2	lighted splint	squeaky pop
oxygen O_2	glowing splint	relights
carbon dioxide CO_2	bubble through limewater	turns milky
chlorine Cl_2	damp litmus paper	bleached white
ammonia NH_3	damp red litmus paper	turns blue
sulfur dioxide SO_2	acidified potassium manganate(VII)	purple → colourless

WORKED EXAMPLE

A solution gives a green precipitate with sodium hydroxide, which turns red-brown on standing. A separate sample, acidified and treated with barium nitrate, gives a white precipitate. Name the compound.

1. green precipitate → **Fe^{2+}** (it slowly oxidises, so goes red-brown)
2. white precipitate with acidified barium nitrate → **sulfate**
3. **the compound is iron(II) sulfate, $FeSO_4$**

WORKED EXAMPLE

A white solid dissolves in water. NaOH gives a white precipitate that dissolves in excess. Ammonia gives a white precipitate that does not dissolve in excess. A flame test gives no colour. Which cation is present?

1. white precipitate that dissolves in excess NaOH → Al^{3+} or Zn^{2+}
2. **does not dissolve in excess NH_3 → Al^{3+} (zinc would dissolve)**

EXAM TIP

In an unknown-solution question, make a **table**: test → observation → conclusion. Name the **precipitate colour** and say **whether it dissolves** in excess. Never write "turns cloudy" — write "white precipitate".

COMMON MISTAKE

Doing the silver nitrate test **without** acidifying with nitric acid. Carbonates and hydroxides would also form precipitates.

6 Test yourself

Test yourself — cover the answers first

QUICK CHECK

1. What apparatus gives the most accurate fixed volume of 25.0 cm^3?
2. What is the filtrate?
3. Which method separates ethanol from water?
4. Why is the baseline in chromatography drawn in pencil?
5. What is the colour of the precipitate of iron(III) with sodium hydroxide?
6. What is the flame colour of potassium?
7. What is the test for chlorine?
8. **EXT** Calculate the R_f for a spot that has moved 2.4 cm when the solvent has moved 6.0 cm.

EXAM-STYLE

9. Describe how to separate a mixture of sand and salt and obtain a pure, dry sample of each. [4]
10. Describe the test for sulfate ions. State the observation for a positive result. [3]
11. Describe how to carry out a titration to find the volume of acid needed to neutralise 25.0 cm^3 of alkali. [5]
12. **EXT** The chromatogram of a food dye shows three spots. What does this tell you about the dye, and how would you identify each spot? [3]

Show answers

1. pipette
2. the liquid that passes through the filter paper
3. fractional distillation
4. pencil does not dissolve in the solvent (ink would run)
5. red-brown
6. lilac
7. damp blue litmus paper is bleached white
8. 2.4 ÷ 6.0 = 0.40
9. add water and stir to dissolve the salt
   filter to separate the sand (residue); wash and dry it
   evaporate the filtrate until saturated, then leave to cool so that salt crystallises
   filter, wash and dry the salt crystals
10. acidify the solution with dilute nitric acid
    add barium nitrate solution
    a white precipitate forms
11. pipette 25.0 cm^3 of alkali into a flask and add indicator
    fill the burette with acid and record the volume
    add acid slowly with swirling until the colour changes
    record the final volume and calculate the titre
    repeat until two titres agree within 0.10 cm^3
12. the dye is a mixture of at least three substances
    calculate the R_f of each spot
    compare with R_f values (or spots on the same chromatogram) of known substances

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