

Experimental techniques and chemical analysis

IGCSE Chemistry

Experimental design

■ Apparatus

You should know which piece of apparatus to use for each measurement:

Measurement	Apparatus
time	stop-watch
temperature	thermometer
mass	balance
volume (accurate)	burette or volumetric pipette
volume (rough)	measuring cylinder
volume of a gas	gas syringe

■ Key words

These words are used throughout practical chemistry:

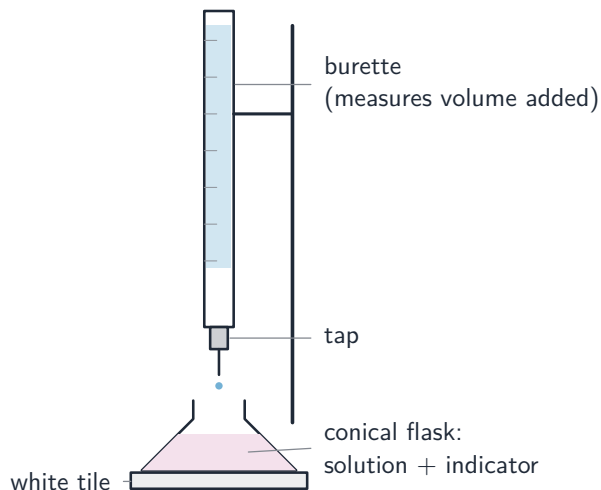
- A **solvent** is a substance that dissolves a solute.
- A **solute** is the substance that dissolves in the solvent.
- A **solution** is a mixture of one or more solutes dissolved in a solvent.
- A **saturated solution** holds the maximum amount of solute that will dissolve at a given temperature.
- A **residue** is the substance left behind after evaporation, distillation or filtration.
- A **filtrate** is the liquid that has passed through a filter.

Acid–base titrations

A **titration** finds the exact volume of one solution that reacts with another.

- Use a **volumetric pipette** to measure a fixed volume of one solution into a flask.
- Add a few drops of a suitable **indicator** .
- Add the other solution from a **burette**, slowly, until the colour just changes.

The **end-point** is the moment the indicator changes colour, which shows the reaction is exactly complete. You read the burette to find the volume added.



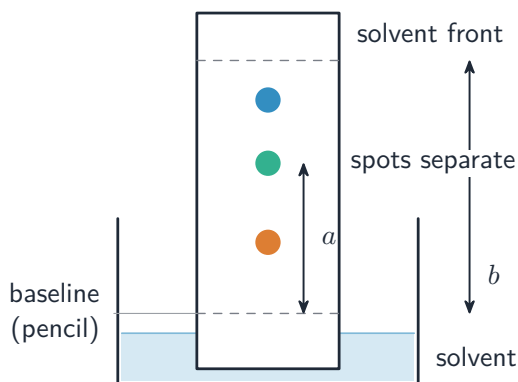
Solution is run from the burette into the flask until the indicator just changes colour (the end-point)



A real titration: solution from the burette is added to the flask, where the indicator has turned orange

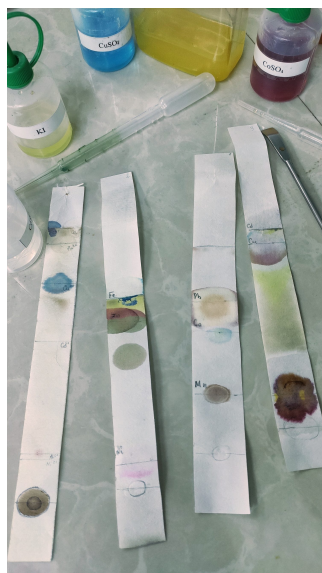
Chromatography

Paper chromatography separates a mixture of soluble substances. You put a spot of the mixture near the bottom of the paper, then stand the paper in a solvent. As the solvent rises up the paper, the substances move different distances, so they separate.



$$R_f = \frac{a}{b} = \frac{\text{spot}}{\text{solvent}}$$

As the solvent rises, the substances travel different distances and separate; R_f is the spot distance (a) divided by the solvent distance (b)



On real chromatograms, each substance in the mixture rises a different distance, leaving a separate coloured spot

- For **coloured** substances you can see the spots directly.
- For **colourless** substances you must spray a **locating agent** to make the spots show up.

The finished paper is a **chromatogram** . You can use it to:

- identify an unknown substance by comparing it with known substances;
- tell if a substance is pure (a pure substance gives only one spot).

You can also calculate the R_f value of a spot:

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

Worked example. On a chromatogram the solvent front has risen 8.0 cm from the baseline, and a spot has moved 6.0 cm. Find its R_f value.

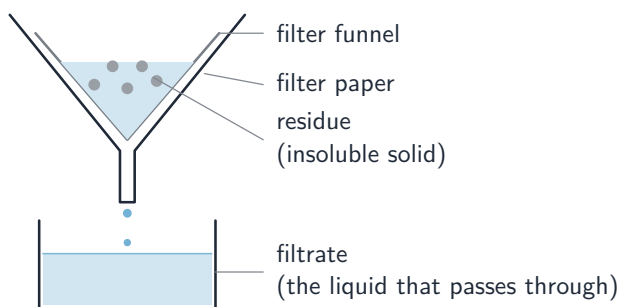
$$R_f = \frac{6.0}{8.0} = 0.75$$

Both distances are measured from the **baseline** (the pencil line), and the spot's distance is taken to the **centre** of the spot. An R_f has no unit, and it is always **less than 1**, because a spot can never travel further than the solvent carrying it - an answer above 1 means the two distances have been divided the wrong way round.

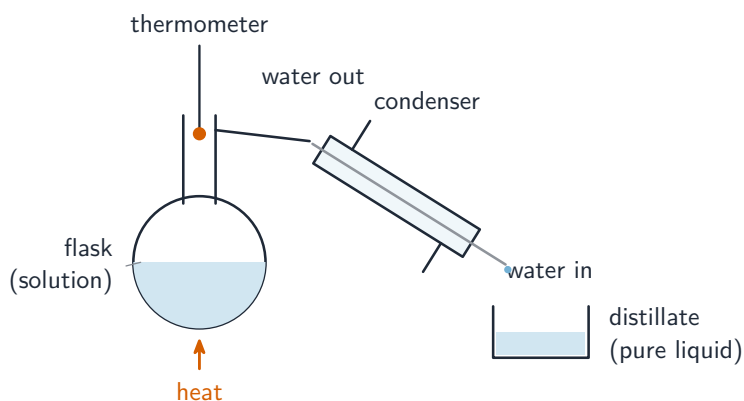
Separation and purification

You choose a method based on the mixture:

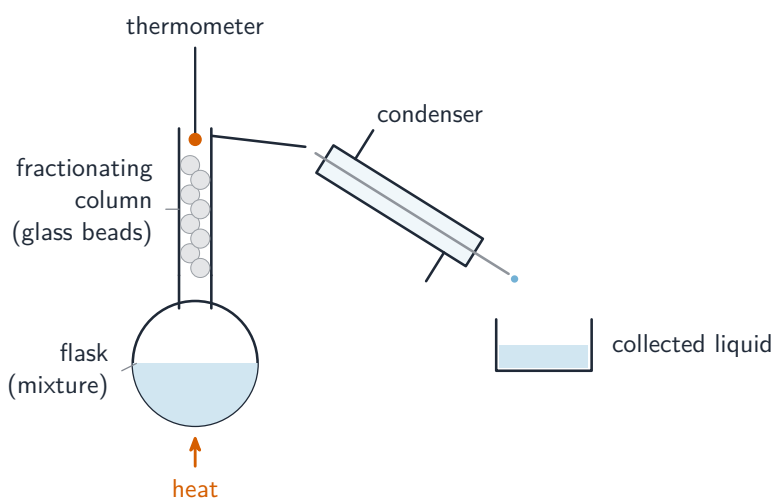
Method	Used to separate
dissolving in a suitable solvent, then filtration	an insoluble solid from a liquid
crystallisation	a soluble solid from its solution
simple distillation	a solvent (the liquid) from a solution
fractional distillation	two or more liquids with different boiling points



Filtration: the insoluble solid stays on the filter paper (residue) and the liquid passes through (filtrate)



Simple distillation: the solvent boils off, the condenser cools it back to a liquid, and the pure distillate is collected



Fractional distillation adds a fractionating column, so liquids with different boiling points separate cleanly

You can check the purity of a substance using its **melting point** and **boiling point** : a pure substance melts and boils at sharp, fixed temperatures, while impurities lower the melting point and raise the boiling point.

Identifying ions and gases

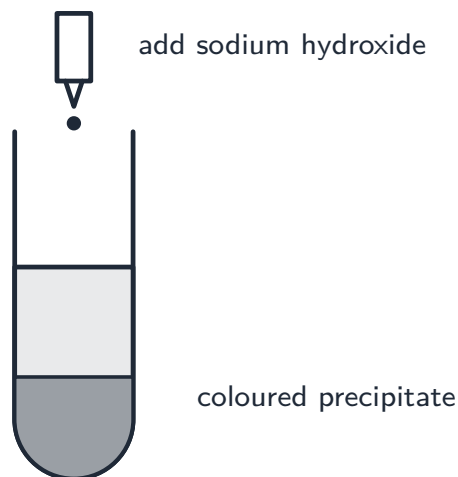
■ Tests for anions

An **anion** is a negative ion.

Anion	Test	Result
carbonate (CO_3^{2-})	add dilute acid	fizzes; the gas turns limewater milky (carbon dioxide)
chloride (Cl^-)	add dilute nitric acid , then silver nitrate	white precipitate
bromide (Br^-)	add dilute nitric acid, then silver nitrate	cream precipitate
iodide (I^-)	add dilute nitric acid, then silver nitrate	yellow precipitate
nitrate (NO_3^-)	add aluminium foil and sodium hydroxide , warm	ammonia gas given off
sulfate (SO_4^{2-})	add dilute nitric acid, then barium nitrate	white precipitate
sulfite (SO_3^{2-})	add acidified potassium manganate(VII)	purple colour fades

■ Tests for cations

A **cation** is a positive ion. Add aqueous sodium hydroxide, or aqueous ammonia, and look at the **precipitate** formed.



Adding sodium hydroxide forms a coloured precipitate for some metal ions

Cation	With sodium hydroxide	With aqueous ammonia
aluminium (Al^{3+})	white, dissolves in excess	white, stays
ammonium (NH_4^+)	ammonia gas when warmed	–
calcium (Ca^{2+})	white, stays	no precipitate
chromium(III) (Cr^{3+})	green, dissolves in excess	green, stays
copper(II) (Cu^{2+})	light blue, stays	light blue, dissolves to deep blue
iron(II) (Fe^{2+})	green, stays	green, stays
iron(III) (Fe^{3+})	red-brown, stays	red-brown, stays
zinc (Zn^{2+})	white, dissolves in excess	white, dissolves in excess

■ Tests for gases

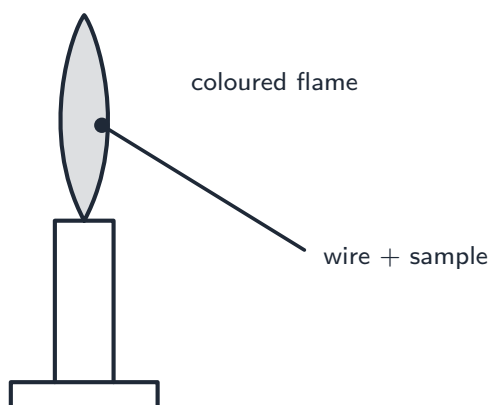
Gas	Test	Result
ammonia (NH_3)	damp red litmus paper	turns blue
carbon dioxide (CO_2)	bubble through limewater	turns milky
chlorine (Cl_2)	damp litmus paper	bleached white
hydrogen (H_2)	a lighted splint	burns with a squeaky pop
oxygen (O_2)	a glowing splint	relights
sulfur dioxide (SO_2)	acidified potassium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



Testing gases: hydrogen pops, oxygen relights a glowing splint

■ Flame tests

A **flame test** identifies some metal cations by the colour they give to a flame:



A flame test: the metal ion gives the flame a colour

Cation		Flame colour
lithium	(Li ⁺)	red
sodium	(Na ⁺)	yellow
potassium	(K ⁺)	lilac (purple)
calcium	(Ca ²⁺)	orange-red
barium	(Ba ²⁺)	light green
copper(II)	(Cu ²⁺)	blue-green

Exam tips

- Pick apparatus by the measurement: a **burette** or **pipette** for accurate volumes, a measuring cylinder for a rough volume, a gas syringe for a gas volume, a balance for mass.
- $R_f = \frac{\text{distance moved by the substance}}{\text{distance moved by the solvent}}$, so it is always less than 1. A pure substance gives a single spot.
- Learn the anion tests: a **carbonate** fizzes and the gas turns limewater milky; halides with silver nitrate give a chloride *white*, bromide *cream*, iodide *yellow* precipitate; a **sulfate** gives a white precipitate with barium nitrate.
- Learn the gas tests: **hydrogen** gives a squeaky pop, **oxygen** relights a glowing splint, **carbon dioxide** turns limewater milky, **chlorine** bleaches damp litmus, **ammonia** turns damp red litmus blue.
- For cations, add sodium hydroxide and read the precipitate colour: copper(II) light blue, iron(II) green, iron(III) red-brown.