

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 秒表
temperature	thermometer 温度计
mass	balance 天平
volume (accurate)	burette 滴定管 or volumetric pipette 移液管
volume (rough)	measuring cylinder 量筒
volume of a gas	gas syringe 注射器

Key words

These words are used throughout practical chemistry:

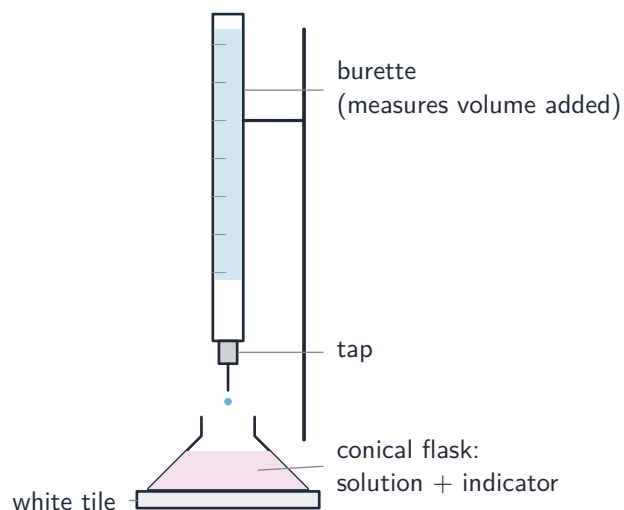
- A **solvent** 溶剂 is a substance that dissolves a solute.
- A **solute** 溶质 is the substance that dissolves in the solvent.
- A **solution** 溶液 is a mixture of one or more solutes dissolved in a solvent.
- A **saturated solution** 饱和溶液 holds the maximum amount of solute that will dissolve at a given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

Acid–base titrations

A **titration** 滴定 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** 指示剂.
- Add the other solution from a **burette**, slowly, until the colour just changes.

The **end-point** 终点 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)

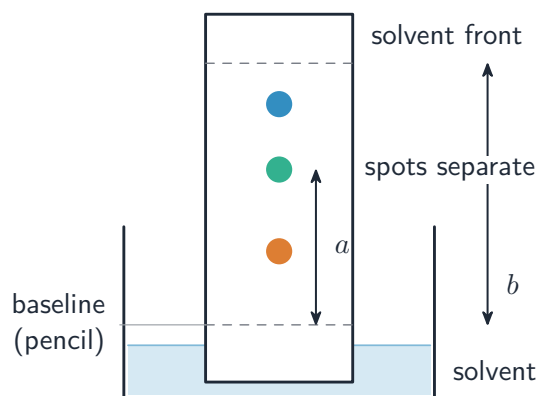


A real titration: solution from the burette is added to the flask, where the indicator has turned orange

Image: Alina.Popova.26, CC BY 4.0 (commons.wikimedia.org)

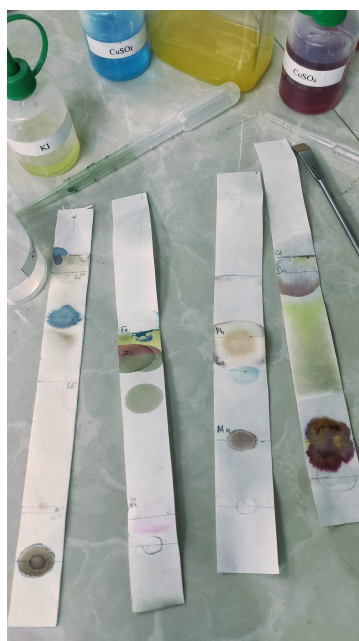
Chromatography

Paper chromatography 纸色谱法 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

Image: Yuliiiaoleksii, CC BY 4.0 (commons.wikimedia.org)

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

The finished paper is a **chromatogram** 色谱图. 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.

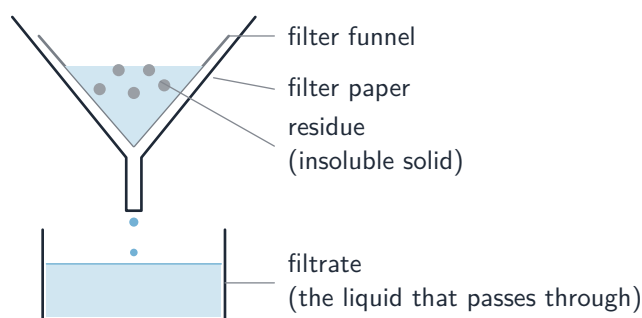
$$R_f = \frac{6.0}{8.0} = 0.75$$

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.

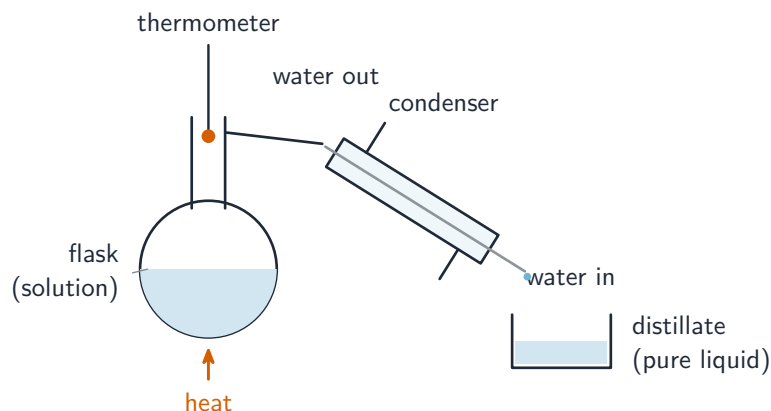
Separation and purification

You choose a method based on the mixture:

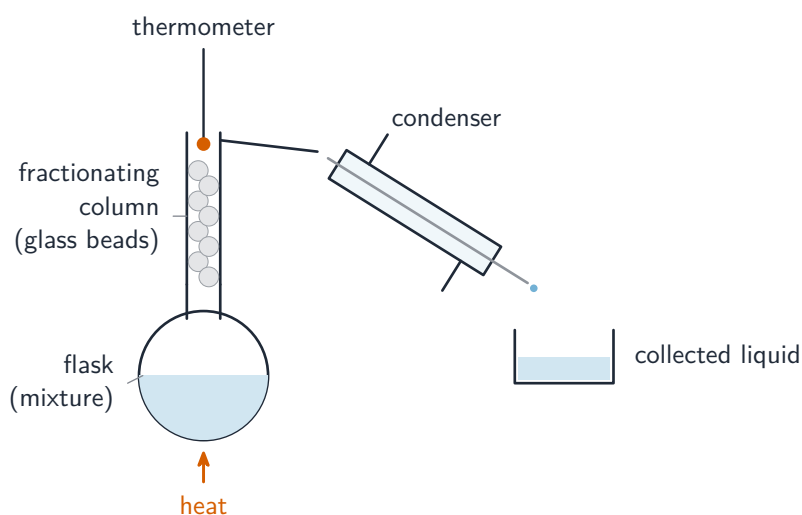
Method	Used to separate
dissolving in a suitable solvent, then filtration 过滤	an insoluble solid from a liquid
crystallisation 结晶	a soluble solid from its solution
simple distillation 蒸馏	a solvent (the liquid) from a solution
fractional distillation 分馏	two or more liquids with different boiling points



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** 熔点 and **boiling point** 沸点: 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

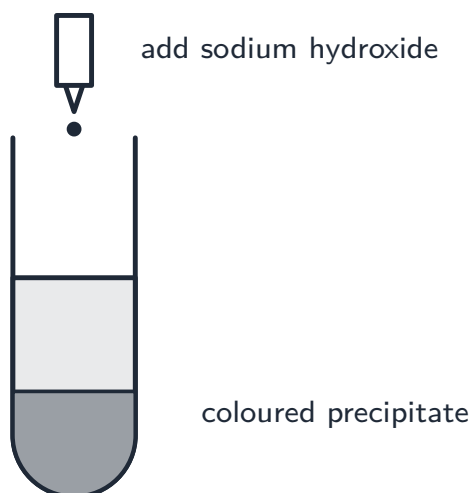
Tests for anions

An **anion** 阴离子 is a negative ion.

Anion	Test	Result
carbonate 碳酸盐 (CO_3^{2-})	add dilute acid	fizzes; the gas turns limewater milky (carbon dioxide 二氧化碳)
chloride 氯化物 (Cl^-)	add dilute nitric acid 硝酸, then silver nitrate 硝酸银	white precipitate
bromide 溴化物 (Br^-)	add dilute nitric acid, then silver nitrate	cream precipitate
iodide 碘化物 (I^-)	add dilute nitric acid, then silver nitrate	yellow precipitate
nitrate 硝酸盐 (NO_3^-)	add aluminium 铝 foil and sodium hydroxide 氢氧化钠, warm	ammonia 氨气 gas given off
sulfate 硫酸盐 (SO_4^{2-})	add dilute nitric acid, then barium nitrate 硝酸钡	white precipitate
sulfite 亚硫酸盐 (SO_3^{2-})	add acidified potassium manganate(VII) 高锰酸钾	purple colour fades

Tests for cations

A **cation** 阳离子 is a positive ion. Add aqueous sodium hydroxide, or aqueous ammonia, and look at the **precipitate** 沉淀 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 铵 (NH_4^+)	ammonia gas when warmed	—
calcium 钙 (Ca^{2+})	white, stays	no precipitate
chromium (III) 铬 (Cr^{3+})	green, dissolves in excess	green, stays
copper (II) 铜 (Cu^{2+})	light blue, stays	light blue, dissolves to deep blue
iron (II) 铁 (Fe^{2+})	green, stays	green, stays
iron(III) (Fe^{3+})	red-brown, stays	red-brown, stays
zinc 锌 (Zn^{2+})	white, dissolves in excess	white, dissolves in excess

Tests for gases

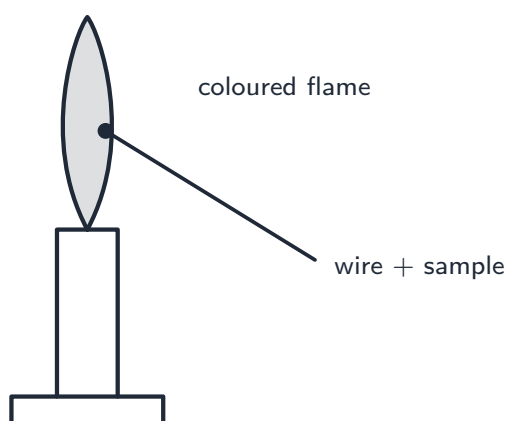
Gas	Test	Result
ammonia (NH_3)	damp red litmus 石蕊 paper	turns blue
carbon dioxide (CO_2)	bubble through limewater 石灰水	turns milky
chlorine 氯气 (Cl_2)	damp litmus paper	bleached white
hydrogen 氢气 (H_2)	a lighted splint	burns with a squeaky pop
oxygen 氧气 (O_2)	a glowing splint	relights
sulfur dioxide (SO_2)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



Testing gases: hydrogen pops, oxygen relights a glowing splint

Flame tests

A **flame test** 焰色试验 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 锂 (Li^+)	red
sodium 钠 (Na^+)	yellow
potassium 钾 (K^+)	lilac (purple)
calcium (Ca^{2+})	orange-red
barium 钡 (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.