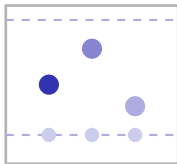


Experimental Techniques & Analysis

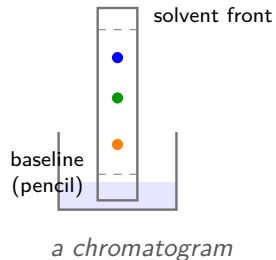
IGCSE Chemistry ■ Topic 12

IGCSE Chemistry



What we will cover

- 1 Apparatus & key words
- 2 Titration
- 3 Chromatography
- 4 Separation & purification
- 5 Tests for ions
- 6 Tests for gases & flames





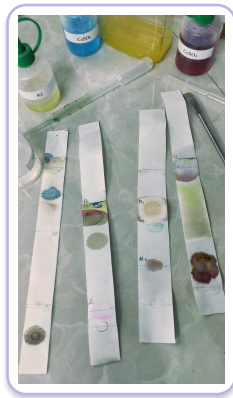
Apparatus and key words

apparatus

burette / **pipette** for exact volume; measuring cylinder for rough

key words

solute dissolves in **solvent** ; **residue** stays, **filtrate** passes through



Chromatography separates mixtures of dyes

The apparatus, and the words that go with it

Measuring

Volume: a measuring cylinder, pipette or burette. Time: a **stop-watch** . Temperature: a **thermometer** . Mass: a balance.

Simple distillation

Simple distillation separates a solvent – the liquid – from a solution; fractional distillation separates two liquids.

Saturated solution

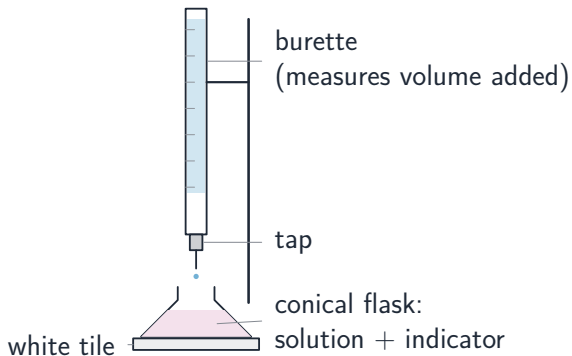
A **saturated solution** holds the most solute that will dissolve at that temperature.

Purity

Check purity with the **melting point** and boiling point: a pure substance melts and boils at sharp fixed temperatures, and impurities lower the melting point and raise the boiling point.

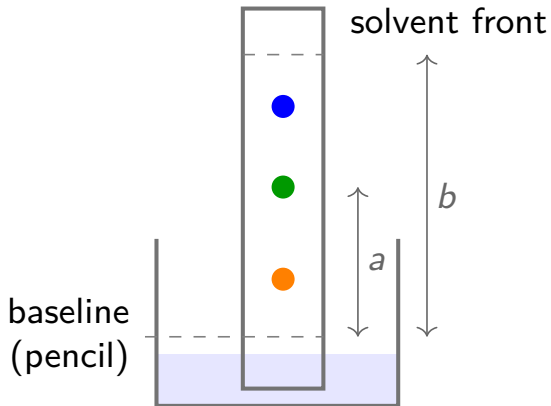
“Pure” in chemistry means one substance – not the everyday sense of clean or natural.

Titration



- 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.

Chromatography



Paper chromatography separates a soluble mixture as the solvent rises.

$$R_f = \frac{\text{spot distance}}{\text{solvent distance}}$$

A pure substance gives **one** spot.

Reading a chromatogram

the baseline

a **pencil** line – ink would dissolve and run

locating agent

for **colourless** substances, sprayed on to make the spots show up

the R_f value

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

a worked value

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

measuring

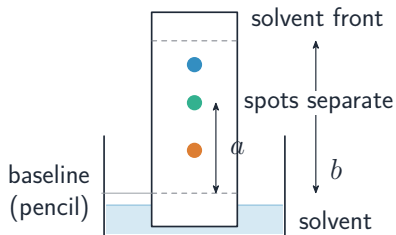
both distances from the **baseline**, and the spot's distance to the **centre** of the spot

the range

an R_f is always between 0 and 1, and it has **no units**

An R_f above 1 means the measurement was made from the wrong line. It is the quickest check on the whole calculation.

As the solvent rises

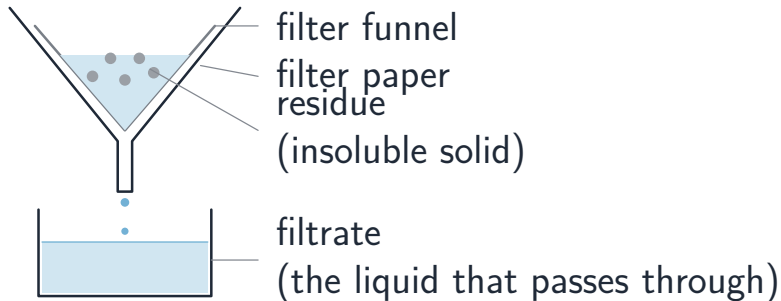


$$R_f = \frac{a}{b} = \frac{\text{spot}}{\text{solvent}}$$

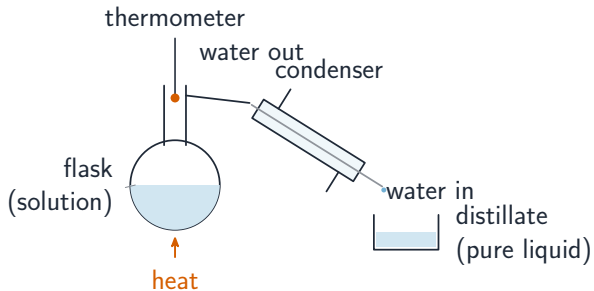
As the solvent rises, the substances travel different distances and separate; R_f is the spot distance (a) divided by the solvent distance (b)

Separation and purification

- **filtration** – insoluble solid from liquid
- **crystallisation** – soluble solid from solution
- **distillation** – solvent from a solution
- **fractional distillation** – liquids by boiling point



Distillation



The solvent boils off, the **condenser** cools it back, and the pure **distillate** is collected.

Check purity by melting/boiling point: a pure substance has sharp, fixed values.



Tests for anions

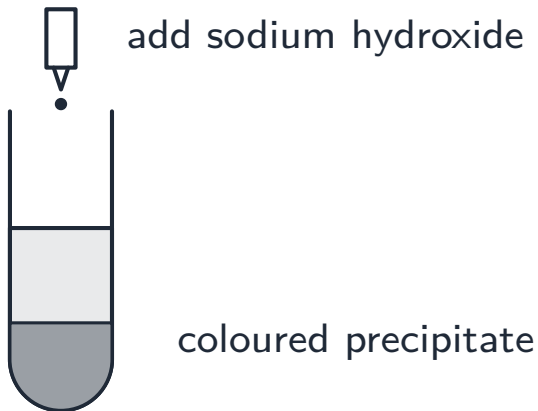
carbonate: add acid → fizzes; gas turns **limewater** milky

chloride/bromide/iodide: HNO_3 then **silver nitrate** →
white/cream/yellow

sulfate: HNO_3 then barium nitrate → white precipitate

nitrate: aluminium + NaOH, warm → ammonia gas

Tests for cations



Add **sodium hydroxide** & watch the **precipitate** :

- copper(II): light blue
iron(II): green
- iron(III): red-brown
- Al, Zn: white, dissolve in
excess

Two cations that need care

Zinc

Zinc (Zn^{2+}): a white precipitate with sodium hydroxide *and* with ammonia – and it dissolves in excess of either.

Aluminium

Al^{3+} also gives a white precipitate that dissolves in excess sodium hydroxide, but *not* in excess ammonia. That is the test that separates them.

Ammonium

Ammonium (NH_4^+): warm with sodium hydroxide and ammonia gas is released – it turns damp red litmus blue.

With the white precipitates, the answer is always in the *excess*: add more and watch which one redissolves.

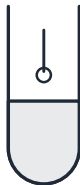
Tests for gases

- **hydrogen** : a lighted splint **pops**
- **oxygen** : relights a glowing splint
- CO_2 : turns limewater milky
- **chlorine** : bleaches damp litmus
- ammonia: turns red litmus blue



hydrogen

lit splint:
squeaky pop



oxygen

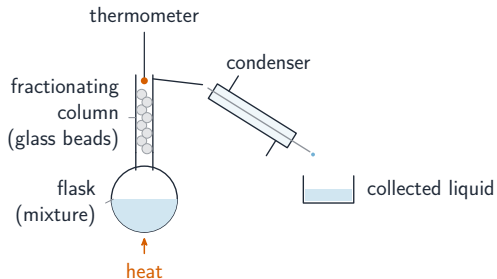
glowing splint:
relights

Flame tests

Li red	Na yellow	K lilac
Ca orange-red	Ba green	Cu blue-green

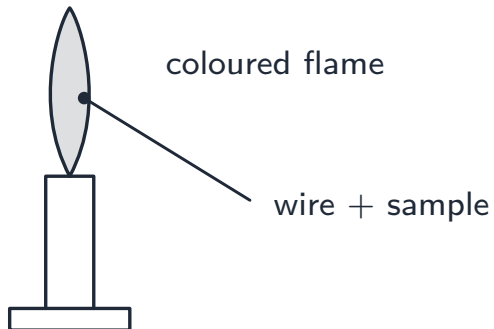
A **flame test** identifies a metal cation by the colour it gives the flame.

Separation and purification, continued



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

Flame tests, continued



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

The flame test colours, and the two purple fades

lithium

red

sodium

yellow

potassium

lilac (purple)

calcium

orange-red

copper(II)

blue-green

acidified potassium manganate(VII)its purple colour fades with both **sulfite** ions and sulfur dioxide gas – the same reagent, two different tests

Use a clean nichrome wire dipped in acid between tests. Sodium's yellow is so strong that a trace of it masks every other colour.

Acid–base titrations



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

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.



Worked example – a titration calculation

25.0 cm³ of NaOH needs 20.0 cm³ of 0.100 mol dm⁻³ HCl. Find the NaOH concentration.

- Moles HCl = $\frac{20.0}{1000} \times 0.100 = 2.00 \times 10^{-3}$
- $\text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O}$: the ratio is 1 : 1.
- So moles NaOH = 2.00×10^{-3} .
- Concentration
$$= \frac{2.00 \times 10^{-3}}{25.0/1000} = 0.0800 \text{ mol dm}^{-3}$$

Three steps every time:
moles of the known →
ratio from the equation
→ concentration. Divide
volumes by 1000 to get
dm³.

Topic 12 – key takeaways

1

Titration

exact volume; stop at the end-point

2

Chromatography

$R_f = \text{spot} / \text{solvent}$; pure = one spot

3

Anions

silver nitrate & barium nitrate precipitates

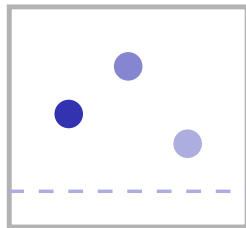
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Gases

H₂ pops, O₂ relights, CO₂ milky

Check yourself

- 1 What is the end-point of a titration?
- 2 How many spots does a pure substance give?
- 3 What reagent tests for a chloride ion?
- 4 What flame colour does sodium give?



Answers 1. where the indicator just changes colour 2. one 3. silver nitrate (after nitric acid) 4. yellow