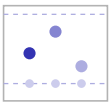
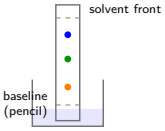
Experimental Techniques & Analysis

IGCSE Chemistry ▪ Topic 12

IGCSE Chemistry



What we will cover



a chromatogram

1 Techniques

Apparatus and key words

apparatus

burette 滴定管/**pipette** 移液管 for exact volume; measuring cylinder for rough

key words

solute 溶质 dissolves
vent 溶剂; **residue** 残
filtrate 滤液 passes t

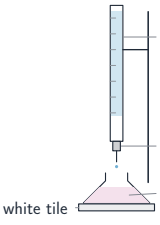
Chromatography separates mixtures of dyes

The apparatus, and the words that go with it

Measuring	Volume: a measuring cylinder, pipette or burette. Time: a **stopwatch** 秒表. Temperature: a **thermometer** 温度计. Mass: a balance.
Simple distillation	**Simple distillation** 简单蒸馏 separates a solvent – the liquid – from a solution; fractional distillation separates two liquids.
Saturated solution	A **saturated solution** 饱和溶液 holds the most solute that will dissolve at that temperature.
Purity	Check purity with the **melting point** 熔点 and boiling point: a pure substance melts and boils at sharp fixed temperatures, and impurities lower the melting point and raise the boiling point.

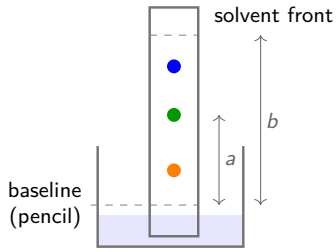
> "Pure" in chemistry means one substance – not the everyday sense of clean or natural.

Titration



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

Chromatography



Paper chromatography 纸色谱法 separates a soluble mixture as the solvent rises.

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

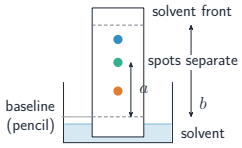
A pure substance gives one spot.

Reading a chromatogram

the baseline	a pencil line – ink would dissolve and run
locating agent 显色剂	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

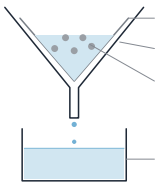


$$R_f = \frac{a}{b} = \frac{\text{spot}}{\text{solvent}}$$

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

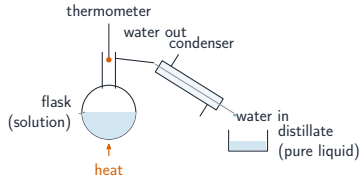
- **filtration** 过滤 – insoluble solid from liquid
- **crystallisation** 结晶 – soluble solid from solution
- **distillation** 蒸馏 – solvent from a solution
- **fractional distillation** 分馏 – liquids by boiling point



filter funnel
filter paper
residue
(insoluble solid)

filtrate
(the liquid that passes through)

Distillation



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

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

2 Analysis

Tests for anions

- **carbonate**: add acid → fizzes; gas turns **limewater** 石灰水 milky
- **chloride/bromide/iodide**: HNO_3 then **silver nitrate** 硝酸银 → white/cream/yellow
- **sulfate**: HNO_3 then barium nitrate → white precipitate
- **nitrate**: aluminium + NaOH, warm → ammonia gas

Tests for cations

add sodium hydroxide

coloured precipitate

Add **sodium hydroxide** 氢氧化钠 & watch the **precipitate** 沉淀:

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

Two cations that need care

Zinc	**Zinc** 锌 (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** 铵 (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** 氢气: a lighted splint pops
- **oxygen** 氧气: relights a glowing splint
- CO_2: turns limewater milky
- **chlorine** 氯气: bleaches damp litmus
- ammonia: turns red litmus blue

lit splint: squeaky pop

hydrogen

glowing splint: relights

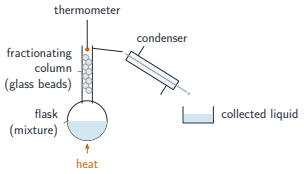
oxygen

Flame tests

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

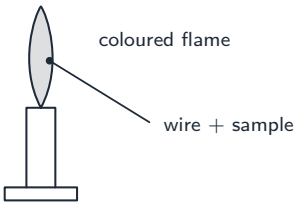
A **flame test** 焰色试验 identifies a metal cation by the colour it gives the flame.

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 钾	lilac (purple)
calcium	orange-red
copper(II)	blue-green
acidified potassium manganate(VII) 高锰酸钾	its purple colour fades with both **sulfite** 亚硫酸盐 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.
- $R_f = \dfrac{\text{distance moved by the substance}}{\text{distance moved by the solvent}}$, 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.

3 Recap

Worked example – a titration calculation

$25.0\,\text{cm}^3$ of NaOH needs $20.0\,\text{cm}^3$ of $0.100\,\text{mol dm}^{-3}$ HCl. Find the NaOH concentration.

- Moles HCl $= \dfrac{20.0}{1000} \times 0.100 = 2.00 \times 10^{-3}$
- NaOH + HCl → NaCl + H_2O: the ratio is $1 : 1$.
- So moles NaOH $= 2.00 \times 10^{-3}$.
- Concentration $= \dfrac{2.00 \times 10^{-3}}{25.0/1000} = 0.0800\,\text{mol dm}^{-3}$

Three steps every time: moles of the known → ratio from the equation → concentration. Divide volumes by 1000 to get dm^3.

Topic 12 – key takeaways

1 Titration

exact volume; stop at the end-point

2 Chromatography

R_f = spot / solvent; pure = one spot

3 Anions

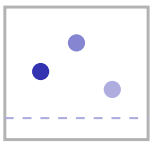
silver nitrate & barium nitrate precipitates

4 Gases

H_2 pops, O_2 relights, CO_2 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