

Experimental techniques and chemical analysis

IGCSE Chemistry 0620 · Topic 12 revision notes

Everything from the 6 lessons of this topic on a few pages: the key idea of each lesson, the diagram to remember, the points to learn and the traps examiners set. Read it, cover it, test yourself.

1. Experimental design and apparatus

2. Acid-base titrations

3. Chromatography

4. Separation and purification

5. Tests for anions and cations

6. Tests for gases

1 Experimental design and apparatus

Syllabus 12.1

Choose apparatus to match the quantity and the precision needed. **Time** is measured with a stopwatch or stop-clock, **temperature** with a thermometer, **mass** with a balance, and **volume** with a **burette** (variable volumes, to 0.05 cm^3), a **volumetric pipette** (one fixed volume, very accurately), a **measuring cylinder** (quick but less precise) or a **gas syringe** (volumes of gas). A **solvent** dissolves a **solute** to make a **solution**; a **saturated solution** holds the maximum amount of solute at that temperature. After filtration, the solid left in the filter paper is the **residue** and the liquid that passes through is the **filtrate**.

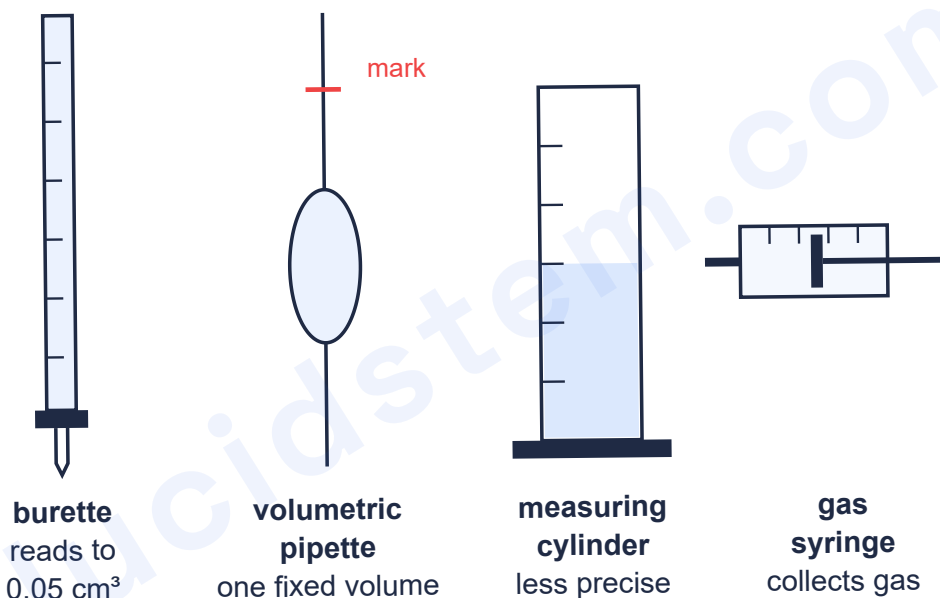


Fig. 1: four ways to measure a volume, from most to least precise for liquids

Quantity, apparatus, precision.

- ✓ **Time:** stopwatch. **Temperature:** thermometer. **Mass:** balance.
- ✓ **Volume:** burette (variable), volumetric pipette (fixed), measuring cylinder (rough), gas syringe (gas).
- ✓ **Solvent** dissolves **solute** to give a **solution**.
- ✓ **Residue** stays in the filter paper; **filtrate** passes through.

Watch out!

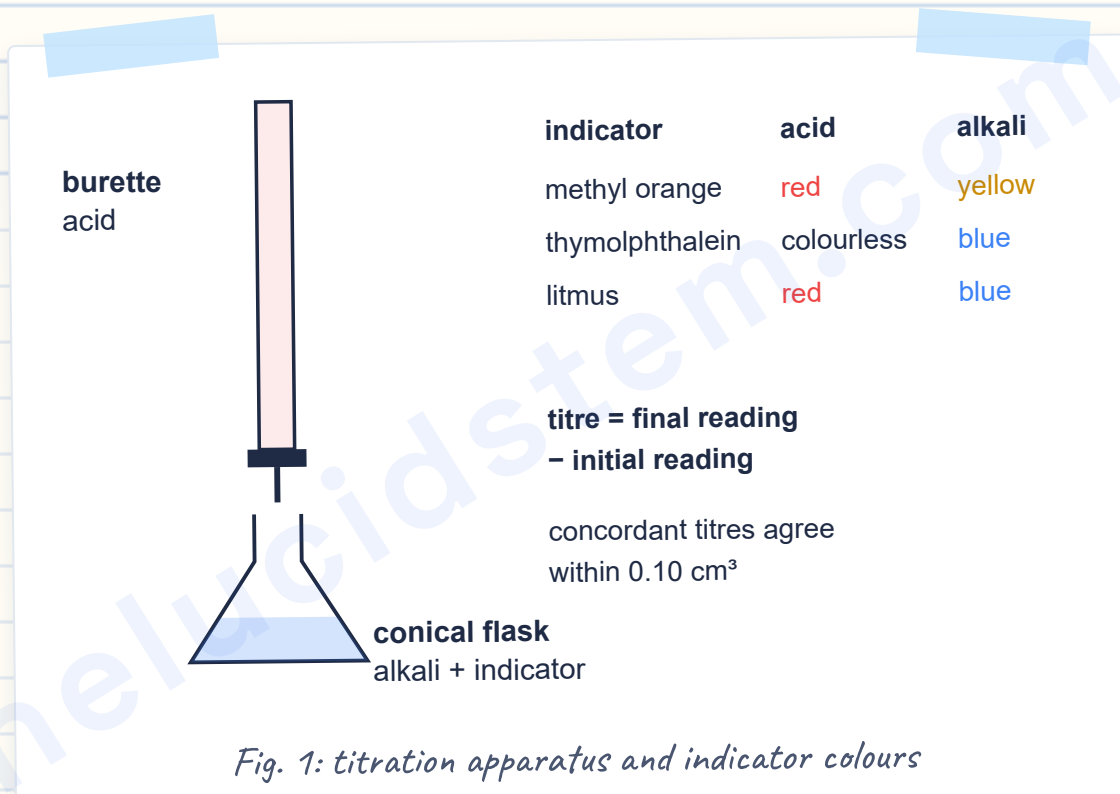
The **filtrate** is what passes through the paper, the **residue** is what stays behind: do not swap them. A measuring cylinder is **not** suitable for a titration, and a gas collected over water must be only slightly soluble (carbon dioxide and ammonia are better measured with a gas syringe). Always give a volume read from a burette to **2 decimal places**, ending in 0 or 5.

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2 Acid-base titrations

Syllabus 12.2

In an **acid-base titration** a **volumetric pipette** transfers a fixed volume of alkali (for example 25.0 cm^3) into a conical flask, a few drops of **indicator** are added, and acid is run in from a **burette** until the indicator just changes colour: the **end-point**. The volume of acid added is the **titre** (final reading minus initial reading). A rough titre is followed by accurate titrations, adding acid drop by drop near the end-point, until **concordant results** (titres within 0.10 cm^3 of each other) are obtained; their mean is used. **Methyl orange** is red in acid and yellow in alkali; **thymolphthalein** is colourless in acid and blue in alkali. Litmus is not used for titrations because its colour change is not sharp.



Pipette, indicator, burette, repeat.

- ✓ **Pipette** a fixed volume of alkali;
burette adds the acid.
- ✓ **Methyl orange**: red in acid, yellow in alkali. **Thymolphthalein**: colourless in acid, blue in alkali.
- ✓ **Titre** = final – initial reading;
concordant = within 0.10 cm^3 .
- ✓ **Extended**: moles = concentration $\times$ volume in dm^3 , then use the mole ratio.

Watch out!

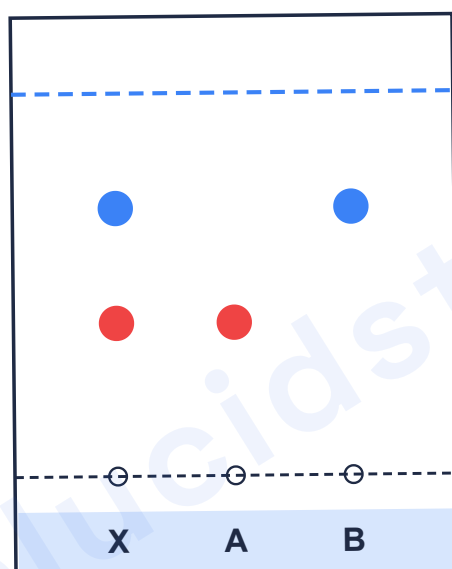
The **rough titre** overshoots the end-point, so leave it out of the mean. **Titre** = final reading **minus** initial reading, not the final reading alone. Use the mole ratio from the equation: with H_2SO_4 , moles of NaOH are **twice** the moles of acid. And convert cm^3 to dm^3 by dividing by 1000.

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3 Chromatography

Syllabus 12.3

In **paper chromatography** spots of the samples are placed on a **pencil baseline**, and the paper stands in a **solvent** whose level is below the baseline. The solvent moves up the paper and carries each substance a different distance, depending on its solubility in the solvent and its attraction to the paper. A **pure** substance gives **one spot**; a mixture gives several. An unknown is identified when its spots match those of known substances at the same heights. **Extended:** colourless substances are made visible with a **locating agent**, and each substance has an **Rf value** = distance moved by the substance $\div$ distance moved by the solvent front.



solvent front (6.5 cm)

X: two spots, so a mixture of A and B

$$R_f(A) = 2.6 \div 6.5 = 0.40$$

$$R_f(B) = 4.55 \div 6.5 = 0.70$$

baseline in pencil

solvent below the baseline

Fig. 1: a chromatogram showing that X is a mixture of A and B

Pencil line, low solvent, match the spots.

- ✓ **Baseline in pencil**; solvent level **below** it.
- ✓ **One spot** = pure; several spots = mixture.
- ✓ **Same height** as a known substance = same substance.
- ✓ **Extended:** $R_f = \text{spot distance} \div \text{solvent front distance}$; locating agent shows colourless spots.

Watch out!

Measure both distances from the **baseline**, not from the bottom of the paper, and measure to the **centre** of the spot. R_f is $\text{spot} \div \text{solvent front}$, so it can never be bigger than 1; if you get 2.5, you divided the wrong way round. A substance that gives one spot in one solvent may still be a mixture; a second solvent can split it.

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4 Separation and purification

Syllabus 12.4

Choose a separation method from the difference between the substances. **Filtration** separates an insoluble solid from a liquid. **Crystallisation** obtains a soluble solid from its solution: heat to evaporate some solvent until the solution is saturated, then cool so crystals form. **Simple distillation** separates a solvent from a solution (for example water from salt water). **Fractional distillation** separates miscible liquids with different boiling points (for example ethanol and water). Dissolving in a **suitable solvent** separates a soluble solid from an insoluble one. A **pure** substance melts and boils at **fixed, sharp** temperatures; impurities **lower the melting point** and make it melt over a **range**, and raise the boiling point. Purity matters for food and drugs.

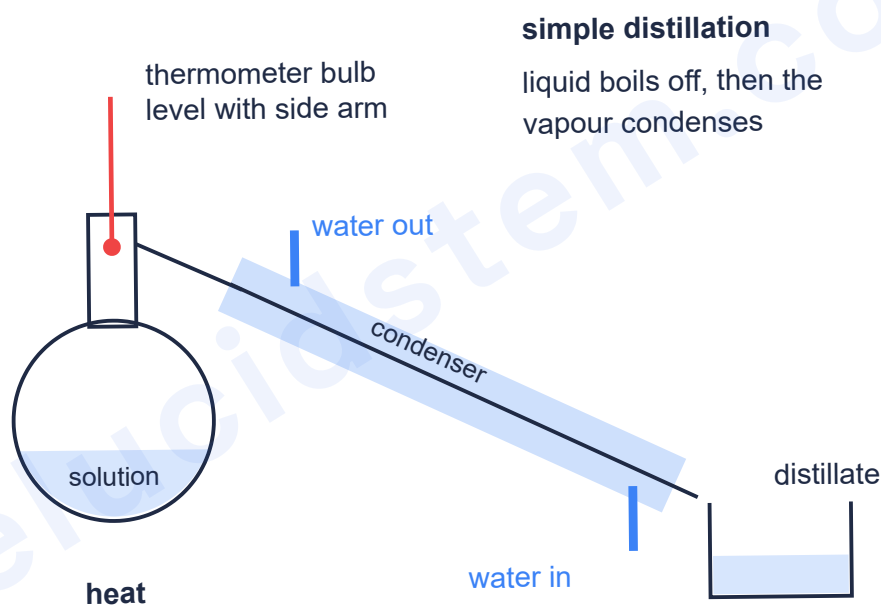


Fig. 1: simple distillation of a solution

Find the difference, pick the method.

- ✓ **Insoluble solid:** filtration. **Soluble solid:** crystallisation.
- ✓ **Solvent from solution:** simple distillation.
- ✓ **Miscible liquids:** fractional distillation.
- ✓ **Pure:** sharp, fixed m.p. and b.p.; impure: lower m.p. over a range.

Watch out!

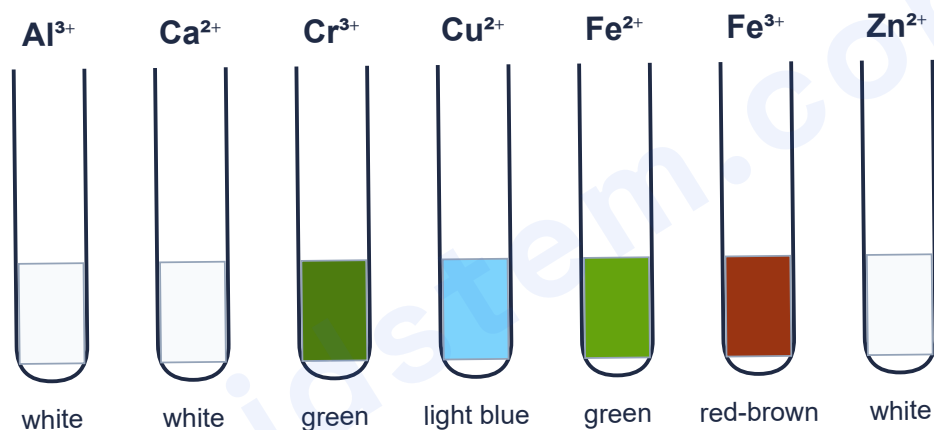
Do not heat a solution **to dryness** when crystallising a hydrated salt: you lose the water of crystallisation and may decompose it. Condenser water goes **in at the bottom** so the jacket stays full. Impurities **lower** the melting point (and widen the range) but **raise** the boiling point.

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5 Tests for anions and cations

Syllabus 12.5

Anions: carbonate fizzes with dilute acid, giving CO_2 that turns limewater milky; **halides** are acidified with dilute nitric acid and tested with aqueous **silver nitrate** (chloride white, bromide cream, iodide yellow precipitate); **sulfate** is acidified and gives a white precipitate with aqueous **barium nitrate**; **sulfite** turns acidified aqueous potassium manganate(VII) from purple to colourless; **nitrate** warmed with aqueous sodium hydroxide and aluminium foil gives ammonia. **Cations** are tested with aqueous **sodium hydroxide** and aqueous **ammonia**: the colour of the precipitate, and whether it dissolves in excess, identifies the ion. **Flame tests** identify Li^+ , Na^+ , K^+ , Ca^{2+} , Ba^{2+} and Cu^{2+} by colour.



precipitates with a few drops of aqueous sodium hydroxide
in excess NaOH: the Al^{3+} , Cr^{3+} and Zn^{2+} precipitates dissolve

Fig. 1: the colours of the hydroxide precipitates formed with aqueous sodium hydroxide

Reagent, observation, ion.

- ✓ **Halides:** HNO_3 then AgNO_3 : Cl^- white, Br^- cream, I^- yellow.
- ✓ **Sulfate:** acidify, $\text{Ba}(\text{NO}_3)_2$: white ppt.
Sulfite: acidified KMnO_4 purple to colourless.
- ✓ **NaOH:** Cu^{2+} light blue, Fe^{2+} green, Fe^{3+} red-brown; Al^{3+} , Zn^{2+} , Cr^{3+} dissolve in excess.
- ✓ **Flames:** Li red, Na yellow, K lilac, Ca orange-red, Ba light green, Cu blue-green.

Watch out!

Halides are acidified with **nitric acid**, never hydrochloric (it adds chloride ions) or sulfuric acid. For sulfate, use **barium nitrate** after acidifying. Fe^{2+} gives a **green** precipitate and Fe^{3+} a **red-brown** one; do not swap them. Copper(II) hydroxide is **light blue**; the **dark blue solution** appears only with excess ammonia. Sodium's strong yellow flame can hide other colours, so the wire must be clean.

6 Tests for gases

Syllabus 12.5

Ammonia turns damp red litmus paper blue. Carbon dioxide turns limewater milky. Chlorine bleaches damp litmus paper. Hydrogen gives a squeaky pop with a lighted splint. Oxygen relights a glowing splint. Sulfur dioxide turns acidified aqueous potassium manganate(VII) from purple to colourless. The litmus must be damp, because a gas has to dissolve in water before it can act as an alkali, acid or bleach.

H₂ lighted splint squeaky pop	O₂ glowing splint relights	CO₂ bubble through limewater turns milky
NH₃ damp red litmus turns blue	Cl₂ damp litmus paper bleached white	SO₂ acidified aqueous KMnO ₄ purple to colourless

Fig. 1: the six gas tests and their positive results

Splints, litmus, limewater, manganate.

- ✓ **H₂**: lighted splint, squeaky pop. **O₂**: glowing splint, relights.
- ✓ **CO₂**: limewater turns milky.
- ✓ **NH₃**: damp red litmus turns blue. **Cl₂**: bleaches damp litmus.
- ✓ **SO₂**: acidified KMnO₄(aq) purple to colourless.

Watch out!

A **lighted** splint is for hydrogen (squeaky pop); a **glowing** splint is for oxygen (relights). It is the **limewater** that turns milky, not the gas. Ammonia turns **red** litmus blue, so blue litmus would show no change. Chlorine **bleaches** litmus; "turns red" alone does not score.