Cambridge IGCSE · Chemistry 0620 · Notes

Experimental design and titrations

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Read first: **Acids, bases and oxides**

What you need to know

By the end of this topic you should be able to:

1. **Name the right apparatus to measure time, temperature, mass and volume**: a stop-watch (time), a thermometer (temperature), a balance (mass), and a burette, volumetric pipette, measuring cylinder or gas syringe (volume).
2. **Suggest advantages and disadvantages** of a piece of apparatus or of an experimental method, for example why a burette is better than a measuring cylinder, or why some gas escapes in a method.
3. **Use six words correctly**: solvent, solute, solution, saturated solution, residue and filtrate.
4. **Describe an acid–base titration**, using a burette, a volumetric pipette and a suitable indicator.
5. **Describe how the indicator shows the end-point** of a titration.

Everything here is Core. Working out a concentration from titration results is Extended and is taught in Relative masses and the mole; the colours of indicators in acids and alkalis are in Acids, bases and oxides.

Understand it

Four things you measure

Almost every experiment measures some of these four quantities, and each has its own instrument:

Quantity	Apparatus	Unit	Usually read to
time	stop-watch (or stop-clock)	s	1 s
temperature	thermometer	°C	0.5 °C or 1 °C
mass	balance	g	0.01 g
volume of a liquid	burette, volumetric pipette or measuring cylinder	cm^3	see below

Quantity	Apparatus	Unit	Usually read to
volume of a gas	gas syringe, or a measuring cylinder full of water turned upside down	cm^3	1 cm^3

Choose apparatus only for what the experiment actually measures. If a method measures a mass, a volume and a time but no temperature, a thermometer is not needed.

Three ways to measure a liquid

The three differ in how **exact** they are and how **flexible** they are.

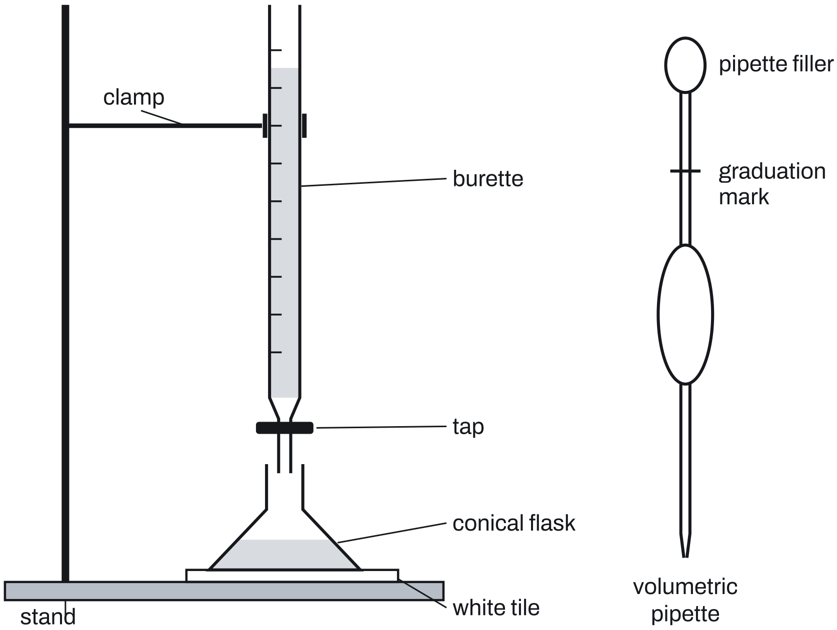
- A **burette** is a long tube with a tap, usually holding 50 cm^3, marked every 0.1 cm^3. It can deliver **any** volume up to 50 cm^3, and each reading is recorded to 0.1 cm^3 or to the nearest 0.05 cm^3 (for example 13.7 cm^3 or 21.50 cm^3). Its scale reads 0.00 at the top, and the numbers get bigger going down.
- A **volumetric pipette** has a bulb and one line on its stem. Filled to the line (with a **pipette filler**, never by mouth), it delivers **one fixed volume** very exactly, such as 25.0 cm^3 or 20.0 cm^3, and nothing else. It is not the same as a dropping pipette, which only adds drops and measures nothing.
- A **measuring cylinder** is quick and holds many volumes, but it is read only to about the nearest 0.5 cm^3 or 1 cm^3. It is fine for "50 cm^3 of acid" but not for "25.0 cm^3" or "25.00 cm^3".

Beakers and conical flasks have rough marks on the side. They hold and mix liquids; they never measure them.

The number of decimal places in the question is the clue. A volume such as 32.6 cm^3 or 5.00 cm^3 needs a burette (or a volumetric pipette, if one of exactly that size exists). "Exactly 25.0 cm^3" into a flask is the job a 25.0 cm^3 volumetric pipette is made for; a burette can do it too.

The titration set-up

A **titration** finds exactly what volume of one solution reacts with a measured volume of another. For an acid and an alkali, it finds the volume of acid that just neutralises the alkali (or the other way round).



The usual method:

1. **Prepare the burette.** Rinse it with distilled water, then with a little of the solution that will go in it (otherwise leftover water dilutes that solution). Fill it using a small funnel, remove the funnel, and run a little out so the tip below the tap is full. Read the starting volume.
2. **Measure the other solution** into a conical flask with a volumetric pipette, for example 25.0 cm^3 of alkali. The flask may be wet with distilled water: that doesn't change the amount of alkali in it.
3. **Add a few drops of a suitable indicator**, such as methyl orange or thymolphthalein.
4. **Stand the flask on a white tile**, so the colour change is easier to see.
5. **Add solution from the burette while swirling the flask.** Near the end-point, add it one drop at a time.
6. **Stop at the end-point**, when one drop makes the whole solution change colour for good. Read the burette again.
7. **Repeat.** The first run is a rough one to find roughly where the end-point is. Repeat carefully until you have **concordant** results: at least two titres within 0.20 cm^3 of each other. Take the mean of the concordant titres.

The end-point

The **end-point** is the point at which the acid and the alkali have exactly reacted. You can't see neutralisation happen, so an **indicator** shows it: it has one colour while the flask still contains alkali and changes colour as soon as the last of the alkali has reacted.

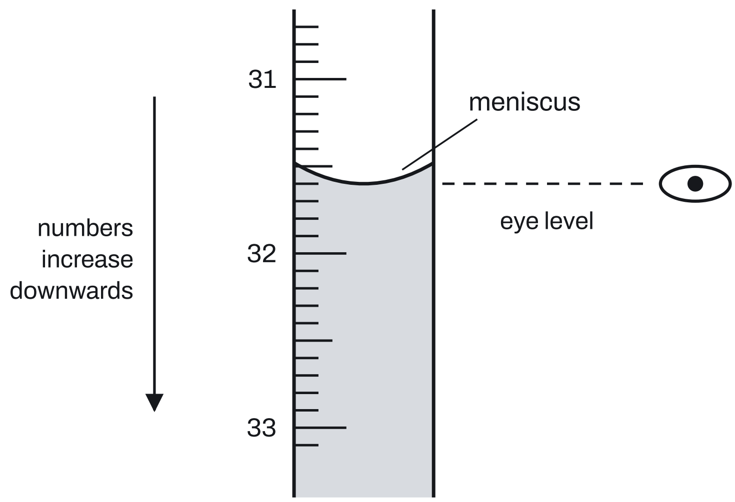
A titration needs an indicator that changes colour **sharply** at one point. **Universal indicator is not suitable**: it runs through many colours gradually, so there is no single sharp colour change to stop at.

Indicator	Alkali in the flask, acid from the burette	Acid in the flask, alkali from the burette
methyl orange	yellow → orange	red → orange

Indicator	Alkali in the flask, acid from the burette	Acid in the flask, alkali from the burette
thymolphthalein	blue → colourless	colourless → blue

Methyl orange is red in acid and yellow in alkali, and **orange** is the in-between colour you see at the end-point. If it goes all the way to red, you have added too much acid and **overshot** the end-point. Always write a colour change **from** the colour at the start **to** the colour at the end-point.

Reading a burette

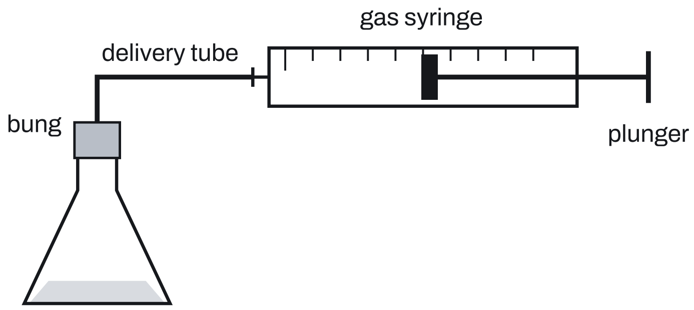


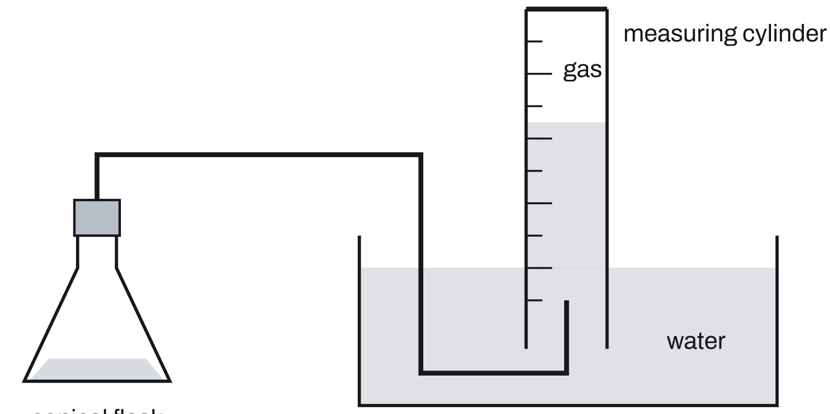
reading = 31.60 cm^3

- Read at the **bottom of the meniscus**, with your eye **level** with it.
- Count down the scale: the reading above is 31.60 cm^3, not 32.40 cm^3.
- Record **every** reading to 0.1 cm^3 (one decimal place) or to the nearest 0.05 cm^3 (two decimal places, ending in 0 or 5), and write all of them the same way: 8.0 cm^3 or 8.00 cm^3, never 8 cm^3; 0.0 or 0.00 cm^3, never 0.
- **Titre = final reading – initial reading.** The final reading is always the bigger number.

Measuring a gas

To follow a reaction that gives off a gas, the gas must be collected without any escaping.





- A **gas syringe** attached to the flask by a delivery tube: the gas pushes the plunger out and you read the volume on the scale.
- An upside-down **measuring cylinder** filled with water, over a trough of water: the gas pushes water out of the cylinder. This doesn't work for a gas that dissolves well in water, such as ammonia, because the gas dissolves instead of collecting.

Every joint must be gas-tight. Beakers, conical flasks and volumetric pipettes cannot measure a gas.

Solutions, residues and filtrates

- **Solvent**: the substance that does the dissolving (usually water).
- **Solute**: the substance that is dissolved.
- **Solution**: what you get when a solute (or several) has dissolved in a solvent; it is a mixture.
- **Saturated solution**: a solution in which the **most solute that can possibly dissolve** has dissolved, so no more will go in, **at a stated temperature**. The temperature matters because most solids dissolve more in hot water than in cold. That is why crystals appear when a hot saturated solution cools: less can stay dissolved.
- **Residue**: the substance that stays **left behind** when you filter, evaporate, distil or separate in a similar way, such as the solid in the filter paper, or the salt left after distilling sea water.
- **Filtrate**: whatever liquid or solution comes out the other side of a filter, having **passed through** it.

When sugar dissolves in water, sugar is the solute, water is the solvent and sugar water is the solution. Undissolved crystals at the bottom of a beaker are just undissolved solid: they are not a filtrate (nothing has been filtered) and not a solute (they haven't dissolved).

Advantages and disadvantages

Judge a piece of apparatus or a method by two questions: **is the measurement exact enough?** and **does anything get lost or added by mistake?**

Apparatus	Advantage	Disadvantage
burette	measures any volume up to 50 cm^3, to 0.1 cm^3 or the nearest 0.05 cm^3	slower to fill and use
volumetric pipette	measures its one volume very exactly	can measure only that one volume; drains slowly
measuring cylinder	quick; many volumes	not exact enough for titrations
gas syringe	reads a gas volume directly; works for gases that dissolve in water	holds only about 100 cm^3
upside-down measuring cylinder over water	cheap and simple	a gas that dissolves in water is not all collected

A disadvantage has to matter **in that experiment**. A pipette measuring only 25.0 cm^3 is no problem when 25.0 cm^3 is exactly what you need.

Common ways to improve a method:

- use a burette or pipette instead of a measuring cylinder, for more exact volumes;
- keep a gas in: add the solid and close the bung quickly, or keep the solid apart in a small tube until the bung is in;
- insulate a reaction vessel (a polystyrene cup with a lid) so less heat is lost when you measure a temperature change;
- **repeat and compare**: repeating on its own does not make results more accurate. Compare the repeats, leave out any anomalous one, and take the mean of the rest.

Key facts and formulas

Nothing in this topic is printed in the exam paper, so everything is "Learn it".

Formula	Status
Titre $=$ final burette reading $-$ initial burette reading	Learn it

Formula	Status
Concordant titres: within 0.20 cm³ of each other; mean titre $=$ mean of the concordant titres only	Learn it
$1\ \mathrm{dm}^3 = 1000\ \mathrm{cm}^3$	Learn it
Time: stop-watch; temperature: thermometer; mass: balance	Learn it
Any exact volume of liquid: burette; one fixed exact volume: volumetric pipette; rough volume: measuring cylinder	Learn it
Gas volume: gas syringe (or upside-down measuring cylinder over water)	Learn it
End-point with alkali in the flask: methyl orange yellow → orange; thymolphthalein blue → colourless	Learn it
Saturated solution: no more solute can dissolve (the most possible has dissolved), at a stated temperature	Learn it

How to do it

In the questions we have tagged for this topic (45 questions, 2021–2025; many multiple-choice questions appear on several papers, so there are 31 different ones), the types came up this often. 2 of the 45 questions mix two types, so the counts add up to 47.

Question type	Questions
Choosing apparatus for a measurement	24
Titration apparatus, indicator and end-point	14
Solution words: solute, saturated, residue, filtrate	4
Measuring the volume of a gas	3
Advantages and disadvantages of apparatus	1
Reading a burette	1

1. Choosing apparatus for a measurement (24 questions)

1. **List what is measured**: a mass, a volume of liquid, a volume of gas, a time, a temperature. Ignore anything the method doesn't measure.
2. **Mass → balance. Time → stop-watch or stop-clock. Temperature → thermometer.**
3. **For a liquid volume, look at the number:**

   - a volume like 13.7 cm^3, 21.50 cm^3 or 5.00 cm^3 → **burette** (it is the only one that measures any volume exactly);
   - "exactly 25.0 cm^3" (or 25.00 cm^3) → **volumetric pipette**, or a burette if no volumetric pipette is offered;
   - a rough volume such as "50 cm^3" → a measuring cylinder is fine.
4. Rule out a beaker, conical flask, test-tube or dropping pipette for any measurement.

Variations you will meet:

- **"Which apparatus is essential?"** with a numbered list: tick only what the stated measurements need. Measuring "the time taken for 1.5 g of zinc to react with 40 cm^3 of acid" needs a balance, a measuring cylinder and a stop-watch, but no thermometer.
- **"Which apparatus can measure only one fixed volume?"**: a volumetric pipette.
- **A size that is too small**: a 25 cm^3 volumetric pipette cannot give 5.00 cm^3 (it gives only 25.0 cm^3); a 50 cm^3 burette can.
- **Pictures instead of names**: recognise them from the diagrams in "Understand it": a burette has a tap at the bottom; a volumetric pipette has a bulb and one mark; a measuring cylinder is a straight tube with a spout and a flat base.

2. Titration apparatus, indicator and end-point (14 questions)

1. **The solution in the flask** is measured with a **volumetric pipette**.
2. **The solution added** comes from a **burette**.
3. **An indicator** is added to the flask; it shows the end-point.
4. **The end-point** is the first permanent colour change. Give the colour change from → to, for the indicator named (table in "Understand it").
5. **Enough results**: at least two concordant titres, within 0.20 cm^3 of each other.

Variations you will meet:

- **"Name the process"** that finds the volume of acid needed to neutralise an alkali: **titration**. The reaction is **neutralisation**.
- **"What else must be added to the flask?"**: an indicator.
- **"Why is universal indicator not used?"**: it changes colour gradually through many colours, so there is no sharp end-point.
- **"Name a suitable indicator"**: methyl orange or thymolphthalein (spell it so it can be recognised).
- **"Which use of apparatus is correct?"** in a described titration: the alkali measured with a volumetric pipette and the acid added from a burette. The marks on a conical flask, a measuring cylinder, or a pipette for the added solution are all wrong.
- **A titration used to make a salt**: after finding the volume needed, repeat with the same volumes but **no indicator**, so the salt is not coloured by it. (The crystallising steps belong to preparing salts.)

3. Solution words: solute, saturated, residue, filtrate (4 questions)

1. Pick out what dissolves (solute), what it dissolves in (solvent) and the mixture (solution).
2. For **"saturated solution"**, give both parts: the **maximum** amount (concentration) of solute dissolved, **at a stated temperature**. "It can dissolve no more solute at that temperature" says the same thing.
3. **Residue** is what stays behind (in the filter paper, or in the flask after distillation or evaporation); **filtrate** is what passes through a filter.

Variations you will meet:

- **Multiple choice mixing the terms up**: for example "the water is the solute" (wrong: water is the solvent) or "a saturated solution contains the maximum amount of solvent" (wrong: maximum solute). The other options may be from another topic, such as saturated hydrocarbons, which are a different meaning of "saturated".
- **Why crystals form as a hot saturated solution cools**: the solubility decreases as the temperature decreases.

4. Measuring the volume of a gas (3 questions)

1. **Name the apparatus**: a gas syringe, or an upside-down measuring cylinder over water.
2. **To draw it**: a conical flask (or boiling tube) with a bung, a delivery tube from the bung, and a gas syringe on the end. Label the flask and the gas syringe. Draw the joints closed so no gas can escape; don't heat it unless the question says so.
3. **To read it**: take the reading from the scale on the syringe barrel, or the level of water in the cylinder.

Variations you will meet:

- **Complete a diagram** where the flask is drawn: add a gas-tight connection and a collecting device.
- **Name another piece of apparatus** that could measure the gas: if the diagram uses a measuring cylinder over water, the other one is a gas syringe (and the other way round). A burette is not accepted, because its scale does not run all the way to the tap.
- **Read a volume from a graph** of gas volume against time, then say why the line levels off: one reactant is used up (this is rate of reaction).

5. Advantages and disadvantages of apparatus (1 question)

1. **Advantage**: say what it does better, usually "measures the volume more exactly (more accurately)" for a burette or volumetric pipette compared with a measuring cylinder.
2. **Disadvantage**: say what it can't do, such as "a volumetric pipette measures only one volume", "a gas syringe holds only about 100 cm^3".
3. Check that both halves are **true** and **relevant**. A made-up limit, such as a container that "can only be used for its full volume", is a trap; the real limit of a volumetric pipette is that it measures only its one volume.

6. Reading a burette (1 question)

1. Find the whole numbers on the scale either side of the meniscus. They **increase downwards**.
2. Count the small marks (0.1 cm^3 each) **down** from the upper number to the bottom of the meniscus.
3. Write the reading to at least one decimal place: a meniscus two marks below 12 reads 12.2 cm^3.

Worked examples

The bold codes show where marks are earned, as in Cambridge mark schemes. The number is how many marks.

- **M1** method mark: for a correct method, even if the numbers slip.
- **A1** accuracy mark: for a correct answer; it needs the M mark before it.
- **B1** mark for a correct result on its own, with no method needed.

Example 1

A student carries out three experiments.

- **P**: find the temperature rise when 1.20 g of calcium oxide is added to exactly 40.0 cm^3 of water.
- **Q**: add exactly 32.6 cm^3 of aqueous potassium hydroxide to a beaker.
- **R**: find how long a sugar cube takes to dissolve in 100 cm^3 of stirred water.

(a) Name the apparatus used to measure the mass of calcium oxide and the temperature rise in **P**. **[2]**

(b) Name the apparatus used to measure the volume in **Q**. Explain why a measuring cylinder is not suitable. **[2]**

(c) Name the apparatus used to measure the time in **R**. **[1]**

(d) The student makes a saturated solution of potassium nitrate. State what is meant by *saturated solution*. **[2]**

(a) Mass: a **balance B1**. Temperature: a **thermometer B1**.

(b) A **burette B1**. A measuring cylinder is read only to about the nearest 1 cm^3, so it cannot measure 32.6 cm^3 exactly **B1**. (A volumetric pipette won't do: it gives only its own fixed volume.)

(c) A **stop-watch B1**.

(d) Water in which as much potassium nitrate has dissolved as possible, so no more will dissolve **B1**, at that particular temperature **B1**.

Example 2

A student finds the volume of dilute nitric acid needed to neutralise 20.0 cm^3 of aqueous lithium hydroxide.

(a) Name the apparatus used to put exactly 20.0 cm^3 of aqueous lithium hydroxide into the conical flask. **[1]**

(b) The student adds thymolphthalein to the flask. State the colour change at the end-point. **[2]**

(c) Suggest why the conical flask stands on a white tile. **[1]**

(d) The student's burette readings are shown.

	rough	1	2	3
final reading / cm^3	22.40	21.15	21.65	22.60
initial reading / cm^3	0.50	0.00	0.40	1.05

Work out the four titres. Use the concordant results to find the mean titre. **[3]**

(e) Another student rinses the conical flask with aqueous lithium hydroxide before adding the 20.0 cm^3. Suggest the effect on the titre. **[2]**

(a) A **volumetric pipette B1**.

(b) From **blue B1** to **colourless B1**. (The flask starts alkaline, so thymolphthalein starts blue.)

(c) So that the colour change is easier to see **B1**. ("To see the colour" is not enough: the tile makes the change clearer.)

(d) Titre = final – initial:

	rough	1	2	3
titre / cm^3	21.90	21.15	21.25	21.55

B1 for all four titres.

Titres 1 and 2 differ by $21.25 - 21.15 = 0.10$ cm^3, so they are concordant. Titre 3 is 0.30 cm^3 from titre 2, and the rough titre is not used. **M1** for choosing 1 and 2 only.

$$\text{mean titre} = \frac{21.15 + 21.25}{2} = 21.20\ \text{cm}^3$$

A1

(e) The titre would be **larger B1**: the lithium hydroxide left in the flask from rinsing is added to the 20.0 cm^3, so more acid is needed to neutralise it all **B1**.

Example 3

A student measures the volume of oxygen made when 1.0 g of manganese(IV) oxide is added to 40 cm^3 of hydrogen peroxide solution in a conical flask.

(a) Draw a labelled diagram of the apparatus to collect and measure the oxygen. **[3]**

(b) The student tips the powder into the flask and then puts the bung in. Suggest why the volume of oxygen measured is lower than it should be, and how to change the method to stop this. **[2]**

(c) The student repeats the experiment three times. The volumes of oxygen after 60 s are 46 cm^3, 58 cm^3 and 47 cm^3. Use these results to give the best value for the volume after 60 s. **[2]**

(d) The student wants to measure the 40 cm^3 of hydrogen peroxide solution more exactly than with a measuring cylinder. Suggest a better piece of apparatus. **[1]**

(a) A conical flask with a bung **B1**, a delivery tube from the bung to a **gas syringe B1**, and every joint drawn closed so no gas can escape **B1**. (The top half of the third diagram in "Understand it" would score all three.)

(b) Some oxygen escapes before the bung is in place **B1**. Keep the manganese(IV) oxide separate in a small tube inside the flask, put the bung in, then tip the flask to mix them **B1**.

(c) 58 cm^3 is anomalous, so leave it out **M1**. Mean $= \frac{46 + 47}{2} = 46.5$ cm^3 **A1**.

(d) A **burette B1**. It can measure any volume up to 50 cm^3 to 0.1 cm^3; volumetric pipettes come only in fixed sizes such as 25.0 cm^3.

Common mistakes

- **Picking a measuring cylinder for an exact volume.** A volume given as 25.0 cm^3 or 21.50 cm^3 needs a burette or a volumetric pipette. Examiners report "measuring cylinder" as a common wrong answer.
- **Mixing up the two pipettes.** A volumetric pipette measures one exact volume; a dropping pipette just adds drops. Advantages or disadvantages that only make sense for a dropping pipette score nothing.
- **Swapping the burette and the pipette.** The pipette measures the solution into the flask; the burette adds the other solution.
- **Getting the end-point colour wrong.** With alkali in the flask, methyl orange starts **yellow**, not orange, and finishes **orange**. Many answers start from orange or write the change the wrong way round.
- **Writing "clear" for "colourless".** Clear means see-through, and a coloured solution can be clear. Thymolphthalein goes blue → **colourless**.
- **Choosing universal indicator** for a titration. It has no sharp end-point.
- **Vague words for concordant results.** "Similar", "close" or "take the average" are not enough: say "at least two titres within 0.20 cm^3 of each other".
- **Saying repeating makes results accurate or reliable.** Repeats only help when you **compare** them, leave out anomalous results, and average the rest.

- **Burette readings as whole numbers**, such as 8 instead of 8.0, or readings with different numbers of decimal places. Some candidates also add the two readings instead of subtracting, or use the final reading as the titre.
- **Forgetting the second rinse.** A burette is rinsed with water to clean it, then with the solution it will hold, to remove the water that would dilute that solution.
- **Leaving the temperature out of "saturated solution"**, or swapping solute and solvent.
- **Leaky gas diagrams.** Gaps between tubes, a bung missing, a measuring cylinder joined straight onto the tube the right way up, or no labels. Draw a closed system and label the flask and the gas syringe.

How to get the marks

- **"Name"** means the exact name: "volumetric pipette" or "pipette", "burette", "gas syringe" (not "cylinder", "tube" or "graduated cylinder").
- **Apparatus in a plan** earns credit only when you say what it measures: "use a burette to add the acid", not a list of apparatus at the start.
- **Colour changes** earn one mark for each colour, and only in the right order: "from yellow to orange".
- **"Suggest why"** a step is done needs its purpose: the white tile makes the colour change easier to see; rinsing the flask with water removes what's left from the last run; the drop-by-drop finish stops you overshooting.
- **Readings and results:** record all readings from one instrument to the same number of decimal places; give units (cm^3, g, s, °C); never round a burette reading to a whole number.
- **Definitions** such as saturated solution have two marking points; learn the full wording in your own words, including "at a specified temperature".
- **Advantages and disadvantages** must be about this experiment. "More accurate" wins the advantage mark for a burette or pipette over a measuring cylinder; "it's fragile" does not.
- **Multiple choice**: look for the number of decimal places, and for whether the question wants a measuring instrument or just a container.

Quick check

1. Which apparatus would you use to measure exactly 18.4 cm^3 of dilute sulfuric acid: a beaker, a burette, a 25 cm^3 volumetric pipette or a measuring cylinder?
2. Aqueous ammonia and methyl orange are in a conical flask, and dilute hydrochloric acid is added from a burette. State the colour change at the end-point.
3. Four titres are 24.60 cm^3 (rough), 23.85 cm^3, 24.25 cm^3 and 23.95 cm^3. Which are concordant, and what is the mean titre?
4. A student fills a burette with acid straight after rinsing it only with distilled water. Suggest the effect on the titre, and explain it.
5. A student makes coffee by pouring hot water through ground coffee in a paper filter. Name the solvent, the residue and the filtrate.

Answers

1. A burette. 18.4 cm^3 is not the pipette's fixed volume, and a beaker or measuring cylinder is not exact enough.
2. From yellow to orange.
3. 23.85 cm^3 and 23.95 cm^3 are concordant (they differ by 0.10 cm^3; 24.25 cm^3 is 0.30 cm^3 from 23.95 cm^3, and the rough titre is not used). Mean $= \dfrac{23.85 + 23.95}{2} = 23.90$ cm^3.
4. The titre would be larger. Water left in the burette dilutes the acid, so more of it is needed to neutralise the same amount of alkali.
5. Solvent: water. Residue: the ground coffee left in the filter paper. Filtrate: the coffee solution that passes through.

Past questions to try

- **0620/41/M/J/22 Q3**: part (c), the pipette, methyl orange's colour change and when you have enough titres.
- **0620/43/O/N/24 Q4**: part (a), thymolphthalein's end-point colour and why universal indicator is not used.
- **0620/32/F/M/25 Q3**: part (b)(i), drawing the apparatus to measure a gas.
- **0620/12/F/M/24 Q39**: solute, filtrate and residue in two experiments.