

The AS Chemistry Practical Paper

The practical paper is for 40 marks and the duration is 2 hours. This means that you must not spend more than three minutes for every mark available on a question.

The paper will usually allocate around 25 marks to quantitative analysis (titration/ gravimetric analysis/ rate experiments/ enthalpy experiments) and around 15 marks to qualitative analysis (salt analysis/ organic analysis). This may be distributed between 2 or 3 questions of differing lengths.

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Precision in Practical Work

- All burette readings should be given to the nearest **0.05 cm³**.
This means that every burette reading must have **2 decimal places**, with the 2nd decimal place being either 0 or 5.
- All temperature readings should be given to the nearest **0.5 °C**.
Hence every thermometer reading must have **1 decimal place**, with the 1st decimal place being either 0 or 5.
- All mass readings should be given to the nearest **0.01 g**.
Hence every mass reading must have **2 decimal places**, where the 2nd decimal place can be any digit.
- All time readings should be given to the **nearest second**.
Hence every stopwatch reading should be rounded off to the nearest **whole number**.

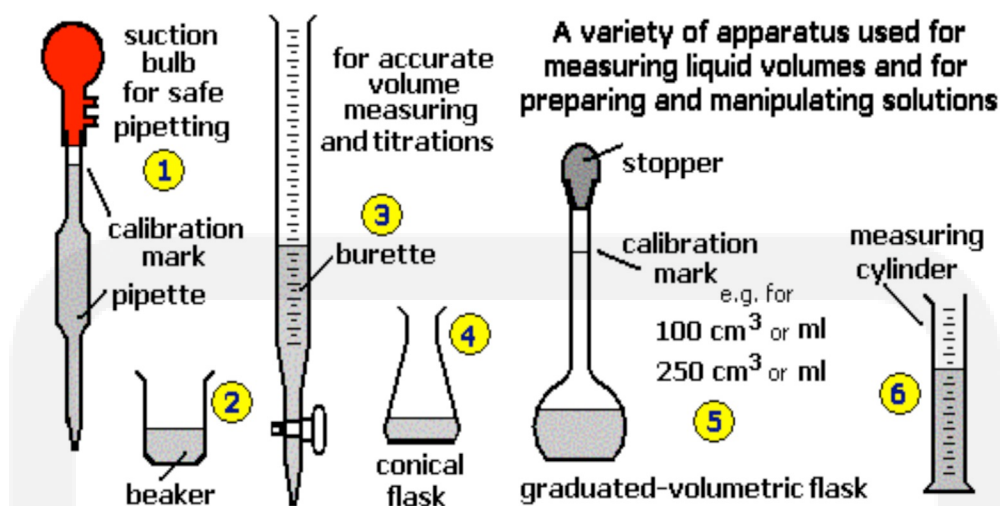
Uncertainties

- The uncertainty in any measurement is **± half of the smallest division** on the apparatus.
For example, a burette is graduated (marked) at every 0.1 cm³. The uncertainty in a single burette reading is therefore **± 0.05 cm³**.
- If a value is based on more than one reading, the individual uncertainties will add up.
For example, the volume of solution run from a burette is found from two burette readings (an initial reading and a final reading). Each of these two readings had an uncertainty of **± 0.05 cm³** so the uncertainty in the volume used is **± 0.10 cm³**.

Titration

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Apparatus



source: www.docbrown.info

Make sure you do not get the apparatus mixed up! For example, a volumetric flask (which has a long neck with a line marking a fixed volume, usually 250.0 cm³) is used to make a solution of a particular concentration, whereas a conical flask (shaped like a cone) is where the second reactant is added to the first reactant (e.g. the acid is added to the base).

Cleaning the apparatus

- Any apparatus used for **measuring** solution volumes (e.g. burette, pipette, measuring cylinder) should be **rinsed with the solution** it is will be used to measure.
- Any apparatus used for **receiving** a solution volume that has already been measured out (e.g. conical flask, volumetric flask) should first be **washed with tap water** several times and then **rinsed with distilled water**.

For example, if an alkali has to be pipetted out into a conical flask and then acid has to be added to the flask from a burette:

the conical flask should be rinsed with

the pipette should be rinsed with

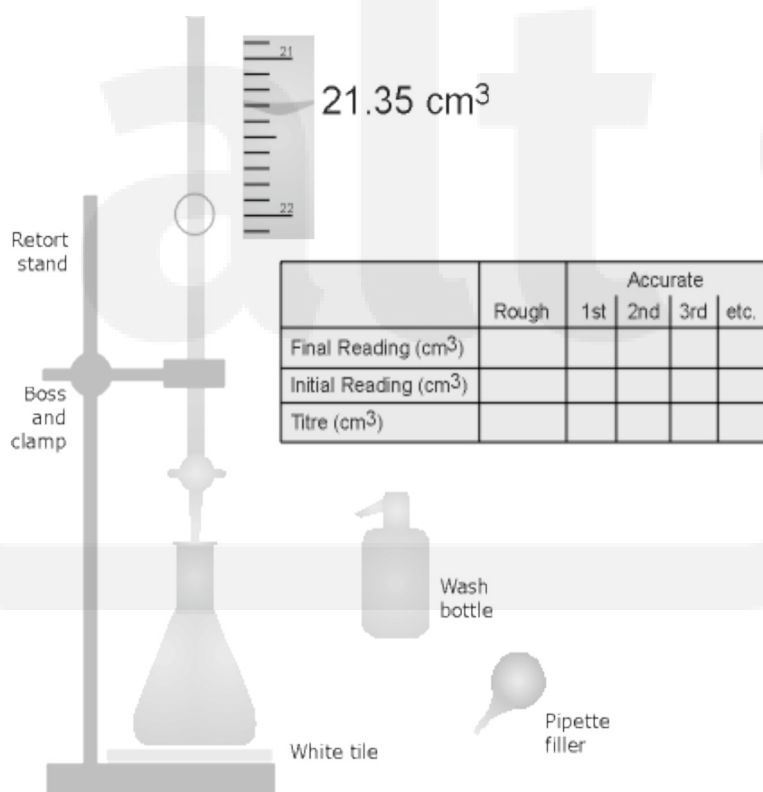
the burette should be rinsed with

Filling up a burette

- Using a funnel, rinse out the burette with the solution it is going to contain and empty it out.
- Next fill up the burette to close to the top, keeping an eye on the level of the solution to prevent spillage.
- **Do not forget to remove the funnel before proceeding.** If you leave the funnel in, it will continue to drip into the burette, making your volume readings inaccurate.
- Open the stopcock (tap) and allow solution to run through and hence fill up the jet of the burette (the part underneath the tap). There should be no air bubbles.
- Close the tap. The burette is now ready for use.
(It does not matter if the initial burette reading is not 0.00 cm³.)

Using a burette

- The burettes you will be using are graduated (marked) at every 0.1 cm³.
- If the lower meniscus is just touching one of these lines, the 2nd decimal place in your reading will be 0. If the lower meniscus is between the lines, the 2nd decimal place will be 5. For example, in the diagram below, the lower meniscus is between the 21.3 and 21.4 cm³ lines. The reading is then taken to be 21.35 cm³ (to the nearest 0.05 cm³).



source: www.avogadro.co.uk

- Make sure to take the readings at eye level to avoid parallax error. You can move the burette up and down in the stand if needed. Please do not tiptoe on the edge of a lab stool – if you lose your balance, you will land face first in glassware and chemicals.
- Be careful not to get the initial and final readings mixed up. The 0 cm³ mark is towards the top of the burette and the 50 cm³ mark is towards the bottom.

Repeating the titration

- Make sure there is enough solution in the burette before starting a second titration.

For example, if in your first titration the initial burette reading was 0.15 cm³ and the final burette reading was 23.50 cm³, the first titre was It is possible to perform a second titration without re-filling the burette, as the expected final reading for the second titration will be, which is within the 50 cm³ maximum limit.

However if in your first titration the initial burette reading was 2.10 cm³ and the final burette reading was 28.50 cm³, the first titre was It is **not** possible now to perform a second titration without re-filling the burette.

- Keep performing titrations until you have results which are concordant (i.e. in agreement). This means that there should be **a difference of no more than 0.10 cm³** between two of your readings. Obtaining concordant results is the only way you can be confident about having the correct reading. Only the concordant results will be averaged for use in the calculations.

Common end-points

- Methyl orange: yellow to orange/ peach. (If you get pink, you have overshoot it.)
- Potassium manganate (VII): colourless to pink. (Purple = too much.)
- Iodine with starch: blue-black to colourless

Other indicators may also be used. The details of the end-points of any unusual indicators will be given in the question.

QUANTITATIVE ANALYSIS

TITRATIONS

Many experiments that you do during your A-level course give only approximate results, but titrations should be accurate.

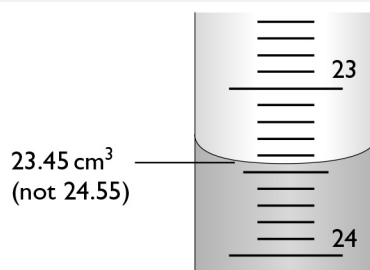
It is essential that you are able to use volumetric flasks, pipettes and burettes confidently and reliably.

The procedure to be followed when carrying out a titration is always the same and the correct techniques must be learned.

MANIPULATION AND MEASUREMENT

You should read a burette to the nearest 0.05 cm^3 , in other words halfway between two of the smallest divisions.

You cannot estimate more precisely than this because the smallest drop a burette can deliver is approximately 0.05 cm^3 .



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QUANTITATIVE ANALYSIS

MANIPULATION AND MEASUREMENT

A common mistake students make is in calculating the average burette reading used in a titration.

This cannot have a greater degree of accuracy than the burette can produce.

Therefore, the average burette reading has to end in $.00 \text{ cm}^3$ or $.05 \text{ cm}^3$. Anything else is wrong and will be penalised.

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QUANTITATIVE ANALYSIS

TITRATIONS

In a titration you need to carry out sufficient repeat titres to establish the 'correct' end point. For a titration with a sharp end point this should be within 0.10 cm^3 .

Once you have established the approximate end point, say 22.60 cm^3 , for the accurate titrations, you can run in 20 cm^3 , swirling the mixture, before adding a few drops at a time. This will save time.

Do not add more than two or three drops of indicator unless instructed otherwise. Using more indicator does not give a more precise end point. In fact, it increases error in the titre value.

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QUANTITATIVE ANALYSIS

TITRE VOLUME RECORDINGS

	ROUGH	1	2	3
final burette reading / cm^3				
initial burette reading / cm^3				
volume of titre / cm^3				

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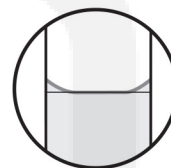
QUANTITATIVE ANALYSIS

USING A PIPETTE

Used correctly, a pipette delivers a precise volume. Since its use requires sucking liquid into the pipette, a safety bulb must always be used.

A 25.0 cm³ pipette is usually capable of measuring a volume of liquid with a maximum error of $\pm 0.06\text{cm}^3$

When the pipette is filled, the meniscus of the liquid should sit on the volume mark on the next of the pipette. To do this reliably requires practice.



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QUANTITATIVE ANALYSIS

USING A PIPETTE

The solution in the pipette should be allowed to run out freely.

A small amount of solution will remain in the bottom of the pipette. Touch the tip of the pipette on the surface of the liquid that has been run out and then ignore any further solution that remains in the pipette.

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QUANTITATIVE ANALYSIS

USING A BURETTE

After filling the burette, run some solution out so that the space below the tap becomes filled.

When using a burette, the volume used is measured as the difference between the readings made at the start and at the finish.

It is usual to read from the bottom of the meniscus but, because you are subtracting two readings, you could use the top of the solution.

This may be helpful if the solution in the burette is coloured — for example, when using potassium manganate(vii) for a redox titration.

USING A BURETTE

Burettes usually have gradations at every 0.1 cm^3 and any reading has a maximum error of $\pm 0.05 \text{ cm}^3$.

Hence, this is the maximum precision that should be recorded. Remember that this precision also applies to the zero, which should be recorded as 0.00 cm^3 .

Values such as 26.25 cm^3 , 26.00 cm^3 and 26.05 cm^3 are all valid readings.

Values such as 24.0 cm^3 (only one decimal place), 24.13 cm^3 (a greater level of precision than is justified) are not.

VOLUMETRIC (GRADUATED) FLASK

As with all volumetric equipment, the volumetric flask is very accurate. The flask holds the specified volume when it is filled so that the meniscus of the solution sits on the mark on the neck.

If you are dissolving a solid in water to make a solution some care is needed. It is usual to dissolve the solid first in a container such as a beaker containing some distilled water.

Once you have dissolved the solid and, if necessary, allowed the solution to cool back to room temperature you can transfer the solution to the volumetric flask, pouring it in through a funnel.

VOLUMETRIC (GRADUATED) FLASK

Wash any remaining solution from the beaker into the flask and be careful not to lose any solution while doing this. You should now add distilled water to the flask until the meniscus is sitting on the mark.

Finally, invert the flask a few times (with the stopper securely in place) to ensure that the solution is mixed completely.

A volumetric flask when filled carefully so that the meniscus is on the line usually gives a reading with a maximum accuracy of $\pm 0.2 \text{ cm}^3$ (or sometimes $\pm 0.3 \text{ cm}^3$). The volume should be quoted to one decimal place, for example 250.0 cm^3 .

MASS BALANCES

These are simple to use but most are very sensitive. Make sure that the pan of the balance is clean before using it and do not attempt to weigh anything that is much warmer than room temperature.

Currents of air may make the reading unsteady and unreliable.

Be careful to read any instructions carefully — for example, you may usually use the tare on the balance when weighing a solid but, in a task, you should check whether this is expected or not.

MASS BALANCE RECORDINGS

	experiment 1	experiment 2
mass of tube + FA 1 /g		
mass of tube + residual FA 1 /g		
mass of FA 1 used /g		

MEASURING CYLINDERS

You should appreciate that measuring cylinders have limited accuracy and so are not appropriate for the most accurate work.

The maximum accuracy of measuring cylinders should be considered to correspond to half the distance between the markings.

For example, a 50cm³ cylinder might be marked in divisions of 1cm³. In which case, the maximum accuracy can be taken to be to the nearest 0.5cm³. Therefore, it would be inappropriate to include any decimal points when recording the volume measured.

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QUANTITATIVE ANALYSIS

VARIABLE VOLUME RECORDINGS

Exp	Volume of FA 5/cm ³	Volume of water /cm ³	time /s	1/time / s ⁻¹
1	50.0	0.0		
2	40.0	10.0		
3	30.0	20.0		
4	20.0	30.0		
5	10.0	40.0		

If a burette is used for measuring out volume then you record volume to 2 decimal place
e.g. 50.00 cm³

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QUANTITATIVE ANALYSIS

NON-DIGITAL THERMOMETERS

If you are reading the temperature of a solution, make sure the bulb is fully covered by the liquid.

As with all measuring equipment, be careful to take readings with your eyes at the same level as the top of the liquid.

Do not view it at an angle because this makes it easy to under- or over-estimate the measurement.

There is a general rule of thumb for non-digital apparatus that the accuracy can be taken to be half a division of the marked scale.

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QUANTITATIVE ANALYSIS

TEMPERATURE RECORDINGS

for exothermic reactions:

	experiment 1	experiment 2
initial temperature / °C		
highest temperature / °C		
ΔT / °C		

for endothermic reactions:

	experiment 1	experiment 2
initial temperature / °C		
lowest temperature / °C		
ΔT / °C		

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QUANTITATIVE ANALYSIS