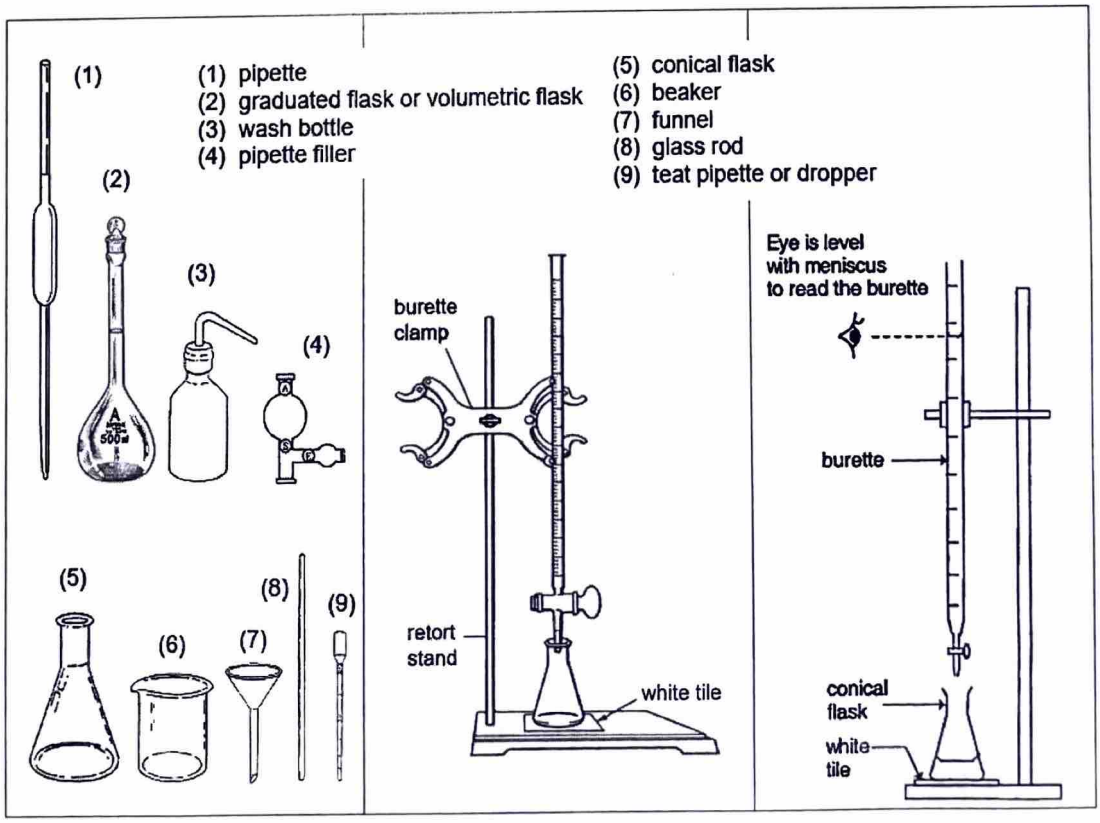
H2 Chemistry Practical	Raffles Institution Year 5 H2 Chemistry 2022	Feb 2022
	Guide to Volumetric Analysis	

Contents

- **Volumetric analysis** (or titrimetric analysis) is a method of quantitative analysis which depends essentially on the **accurate measurements of volumes** of two solutions which react together completely.
- In volumetric analysis, a **standard solution** (i.e. a solution of known concentration) is used to determine the concentration of another solution.
- This is done through a **titration** process which involves the gradual addition of one solution (from a burette) to a fixed volume of another solution (in a conical flask) until stoichiometric amounts of the two reactants have reacted.
  - The solution that is placed in the burette is sometimes referred to as the **titrant**.
  - The solution that is placed in the conical flask is less commonly referred to as the **titrand**.
- In practice, the completion of an **acid-base titration** is usually detected by a distinct colour change brought about by the use of a suitable **indicator**. The point at which this distinct colour change occurs is called the **end-point** of the titration.
- In an acid-base titration, the indicator used is usually added in a small quantity. Common indicators include methyl orange, screened methyl orange, thymol blue and thymolphthalein. Each indicator has a pH range over which it changes colour.

The common apparatus used in a titration are shown below.



1. Manipulation of Apparatus and Materials

1.1 Using the Pipette

- The pipette commonly used for 'A' level practical work is the "volumetric" or "transfer" pipette. It is used to measure out a specified volume of liquid (e.g. 25.0 cm^3) accurately.

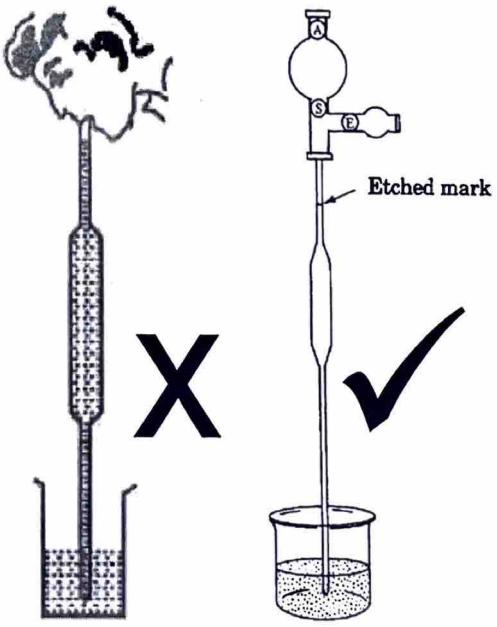
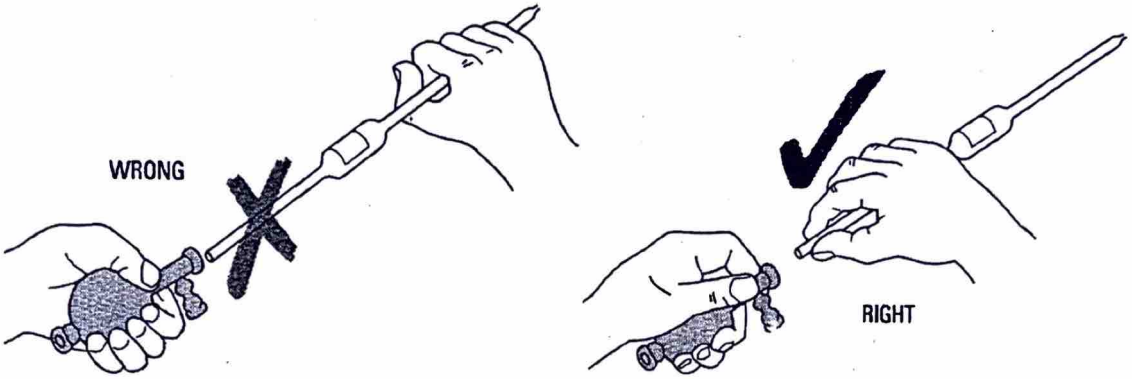
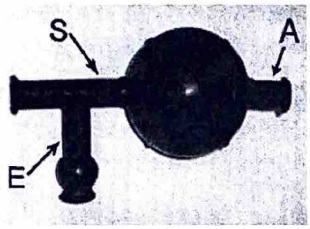
Do's	Don'ts
Rinsing the pipette 1. With the aid of the pipette filler, rinse the pipette with deionised water first. 2. Dry the outside of the pipette (if necessary) and also the region near the pipette tip with tissue paper. 3. Next, rinse the pipette with the solution which it is going to contain. Draw some solution into the pipette, and then tilt and rotate the pipette until **all** of the inner surfaces have been rinsed by the solution. 4. Repeat the rinsing with another small portion of the solution (if necessary). 5. Dry the pipette stem and pipette tip with tissue paper. **Filling the Pipette** 1. With the aid of the pipette filler, draw the solution up to about 1 cm above the calibration mark. 2. Ensure that there are no air bubbles trapped in the solution in the pipette. 3. Lift the pipette **above the surface of the solution** and slowly adjust the solution level in the pipette until the **meniscus just touches the calibration mark**. Do this adjustment at eye-level to avoid parallax error. **Delivering the solution** 1. Hold the pipette vertically with the pipette tip inside a conical flask (i.e. the receiving vessel) to get ready to drain the solution out. Ensure that the **pipette stem and tip are not in contact with any parts of the conical flask**. Use the pipette filler to eject some liquid till the level drops to the bulb region of the pipette. 2. **Remove the pipette filler gently** and hold the pipette vertically to allow further draining of the solution into the conical flask. 3. **Tilt the conical flask slightly** and allow the **pipette tip to touch the bottom** of the conical flask **without touching the solution** (see **Figure 3**). Rotate the pipette tip to draw out some more of the remaining solution in the pipette. Then remove the pipette. **At the end of the experiment** 1. Rinse the pipette with water thoroughly. Solutions should not be allowed to dry inside a pipette because removal of internal deposits is difficult. 2. Inflate the pipette filler when it is not in use.	1. **Do not** use your mouth to draw any solution into a pipette. There could be serious health consequences! 2. **Do not** insert the pipette too far into the pipette filler. This could damage the pipette filler (See **Figure 1** on page 3). 3. **Do not** allow any solution to enter the pipette filler. This contaminates the pipette filler for subsequent use and could also damage it. 4. **Do not** adjust the liquid level in the pipette to the calibration mark when the pipette tip is still immersed in the solution. 5. **Do not** tap the base of the conical flask with the pipette tip to force the last drop of solution out. **Note:** Volumetric pipettes are designed and calibrated such that after the required volume of a liquid is dispensed, a very small volume of liquid will remain in the tip. This last drop should not be blown out! 

Figure 1: Always carry out pipetting with a pipette filler. Care must be exercised when inserting the upper end of the pipette into the pipette filler.





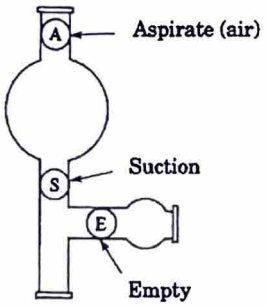
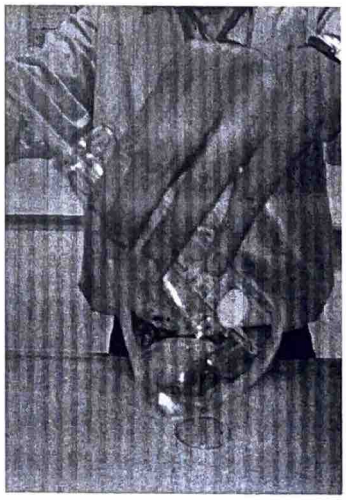


Figure 2: Using the pipette filler

1. Use your thumb and forefinger to press on valve "A" and squeeze the bulb with other fingers to produce a vacuum for aspiration. Release valve "A" once the bulb is completely deflated.
2. Hold the pipette close to its upper end and insert into the bottom of the pipette filler (see **Figure 1** above).
3. Insert the pipette into the liquid to be transferred. Press on valve "S". Suction will draw liquid up into the pipette. Continue pressing valve "S" until the liquid reaches the desired level. Carefully adjust the liquid level so that the bottom of the meniscus coincides with the calibration line on the pipette.
4. Press on valve "E" to expel liquid.
5. Carefully remove the pipette filler from the pipette and allow the liquid to drain freely into the receiving flask.

Figure 3: Tilt the conical flask slightly and allow the pipette tip to touch the bottom of the conical flask without touching the solution. Rotate the pipette tip a few times to draw out some more of the remaining solution in the pipette. Do not blow out the last drop!



1.2 Using the Burette

- The burette is used for measuring out the volume of a liquid (maximum volume: 50.00 cm^3) accurately and is commonly used in volumetric analysis.

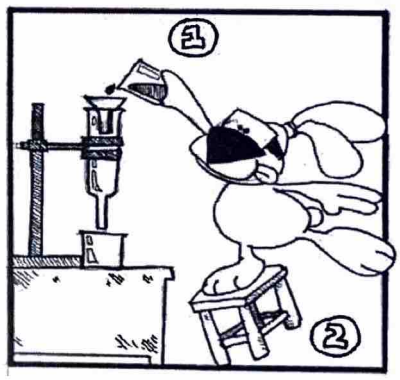
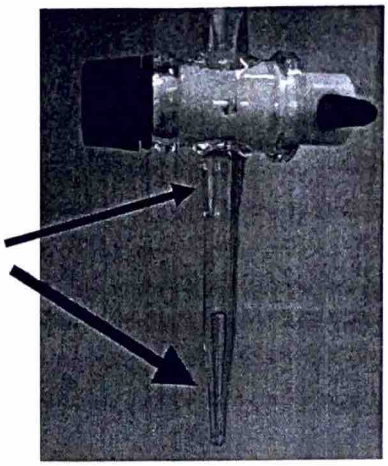
Do's	Don'ts
Rinsing the burette 1. Rinse the burette with deionised water and dry the outside if necessary. 2. Introduce a small portion of the required reagent into the burette with the aid of a funnel. Rinse the burette with this small portion of solution. Tilt the burette and rotate it such that all of the inner surfaces (including the burette tip) have been rinsed by the solution. **Note:** Rinse the burette (and funnel) at least once with a small portion of the required reagent.	1. Do not close the top end of the burette with your fingers while rinsing the burette. Avoid contact with chemicals at all times! 2. Do not climb onto a stool to fill up a burette. Bring the retort stand with the burette down to the stool if necessary.
Filling the burette 1. Clamp the burette vertically using a retort stand. 2. Fill the burette with the required solution from a beaker with the **aid of a funnel** (see **Figure 4** on page 5). 3. **Remove the funnel** after filling up the burette with the solution. 4. **Check that no air bubbles are trapped** in the region near the burette tip (see **Figure 5** on page 5). Flush out any air bubble by opening the tap to allow some solution to drain out. **Note:** If an air bubble is present in a burette at the start of a titration, its volume may change during the course of titration, thereby causing an error in reading the volume of liquid delivered. 5. Adjust the initial volume of the solution in the burette to a convenient value (e.g. 1.00 cm^3). Do the adjustment at eye-level to avoid parallax error (see **Figure 7** on page 5). **Note:** • For clear (i.e. light-coloured) solutions, the bottom of the meniscus is used to read the burette readings (see **Figure 6** on page 5). • For opaque (i.e. dark-coloured) solutions (e.g. purple $KMnO_4$), the bottom of the meniscus may be difficult to see. For such solutions, the top of the meniscus can be used to read the burette readings. 6. Record the initial burette reading.	Lab safety is important. You do not need to learn about safety from accidents!  

Figure 4: Fill a burette with the aid of a funnel. You may need to lift up the funnel slightly, to allow the solution to flow in freely.

Figure 5: Always check that there is no air bubble trapped in the burette. Flush out any air bubble by opening the tap to allow some solution to drain out.



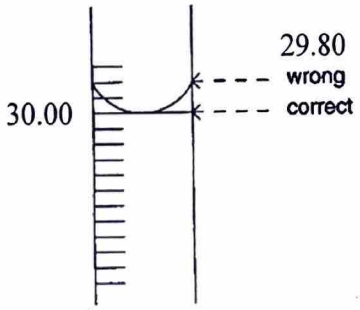


Figure 6: For **clear solutions**, the **bottom** of the meniscus should be read at eye-level. In this case, the burette reading is 30.00 cm^3.

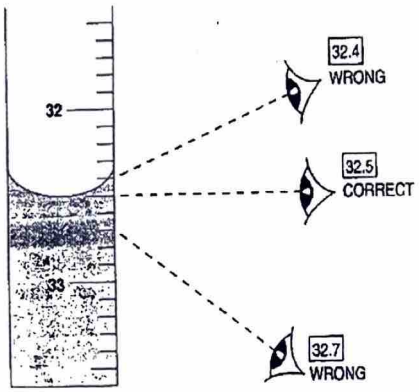


Figure 7: Always take readings at eye-level to avoid parallax errors.

1.3 Titration

Do's

1. Follow the instructions given in **Section 1.2** to get your burette ready for titration. **Record the initial burette reading**.
2. Place a piece of white tile on the base of the retort stand holding the burette.
3. Adjust the burette (on the bench top) to a comfortable height for titration (e.g. with the burette tip slightly inside the conical flask but not in contact with any parts of the flask).
4. Carry out the titration with the addition of the solution from the burette (see **Figure 8**), with **continuous swirling** of the solution in the conical flask.
5. Add the solution from the burette **drop-wise when approaching the end-point** of the titration. Also **rinse the inner walls of the conical flask with deionised water** when approaching the end-point to wash down any adhering solution.

   Note:
   It is common to carry out a rough titration to determine the approximate volume of the solution required to reach the end-point.

6. **Stop** the titration when the end-point is reached i.e. when **one drop of the solution** added from the burette **causes a distinct colour change** of the solution in the conical flask.
7. Read the final burette reading at eye-level and record it.
8. Repeat the titration to get at least **two consistent readings** (i.e. the two titre values are within 0.10 cm^3 of each other).

Don'ts

1. Do not rinse the conical flask with the titrand solution. Rinse the conical flask only with deionised water.
2. Do not release solution continuously from the burette without swirling the conical flask even if the approximate end-point is known.
3. Do not carry out titration with the funnel still resting on the top of the burette.

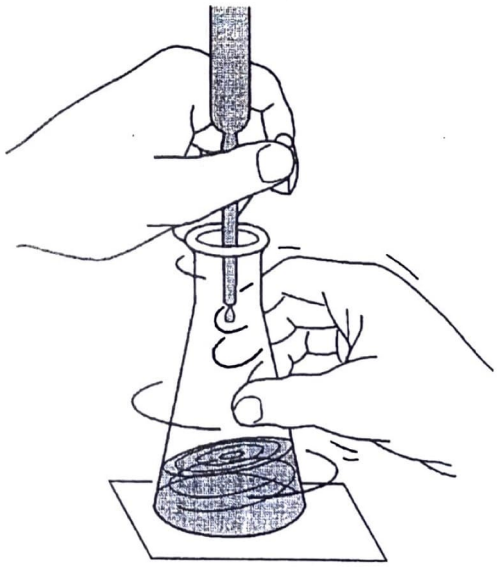
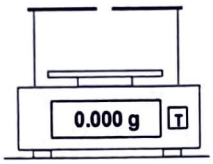


Figure 8: The correct way of holding the burette during titration.

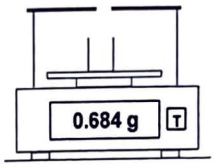
1.4 Weighing a sample using an analytical balance

- Ensure a draught-free environment before weighing a sample (e.g. switch off the fans near the balance) using an analytical balance.
- Check that the units displayed on the weighing balance are correct i.e. in grams (g) (see **Figures 10a** and **10b** on page 8).

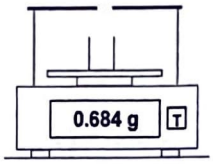
(A) To find the mass of an empty weighing bottle



1. Press the tare button to zero the electronic balance.

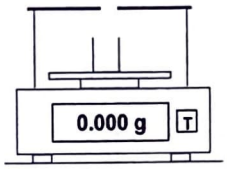


2. Place the clean and dry weighing bottle (without the cap) onto the weighing pan of the balance.

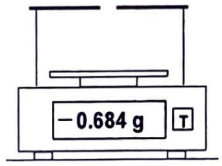


3. Record the mass reading of the empty weighing bottle.

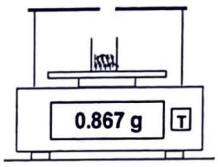
(B) To find the mass of a chemical X (direct weighing)



1. Place the clean and dry weighing bottle (without the cap) onto the weighing pan of the balance.
2. Press the tare button to zero the electronic balance with the weighing bottle.
   Note: The reading shown should be 0.000 g.



3. Take out the empty weighing bottle.
   Note: The balance will now show a negative reading.
4. Add the chemical X into the weighing bottle.
   Note: The addition or removal of X must be done outside the weighing balance to avoid any accidental spillage of X on the weighing pan (see **Figure 11** on page 8).



5. Place the weighing bottle containing X onto the weighing pan of the electronic balance.
   Note: The balance will now show the mass of X present.
6. Record the mass of X as shown by the electronic balance.

(C) To find the total mass of weighing bottle and chemical X

Method 1

1. Obtain the mass of the empty weighing bottle by carrying out steps (A1) to (A3).
2. Obtain the mass of X required by carrying out steps (B1) to (B6)
3. Add up the two mass readings to get the total mass of weighing bottle and X.

Method 2

1. Obtain the mass of the empty weighing bottle by carrying out steps (A1) to (A3).
2. Introduce the mass of X required by carrying out steps (B1) to (B5).
3. Take out the weighing bottle containing X from the balance.
4. Tare the balance to zero it.
5. Place the weighing bottle containing X back into the balance.
   Note: The balance will now show the total mass of the weighing bottle and X.

Figure 9a: A top-loading electronic balance commonly used in our chemistry laboratories.

Figure 9b: Another type of analytical balance commonly used for weighing chemicals in the laboratory.

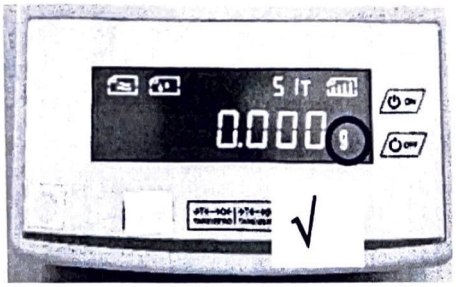
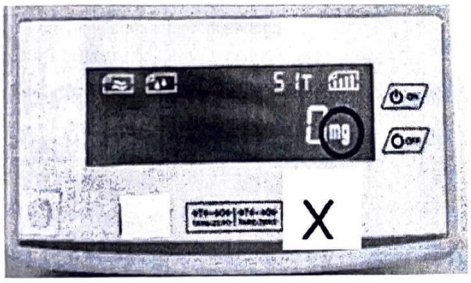


Figure 10a: The display on the analytical balance should show "g" as the units.



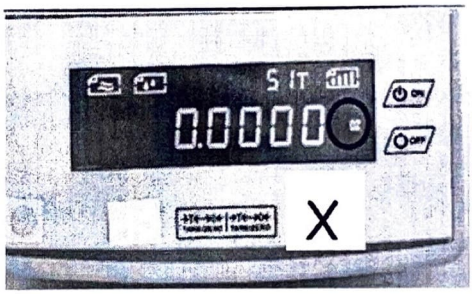


Figure 10b: Examples of inappropriate units being shown on the display on the analytical balance.

Figure 11: The transfer of a chemical into the weighing bottle should be done outside of the weighing balance to avoid any spillage on the weighing balance pan.

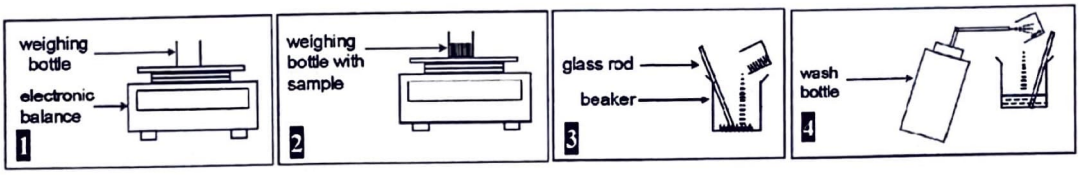
Don'ts

1. **Do not** attempt to clean the weighing balance pan yourself unless instructed to do so.
2. **Do not** transfer chemicals into a weighing bottle inside an analytical balance. This is to avoid spillage of chemicals on the weighing balance pan.
3. **Do not** press other buttons on the panel except for the TARE button. Ask your teacher for help if needed, for example, if the wrong units are displayed.

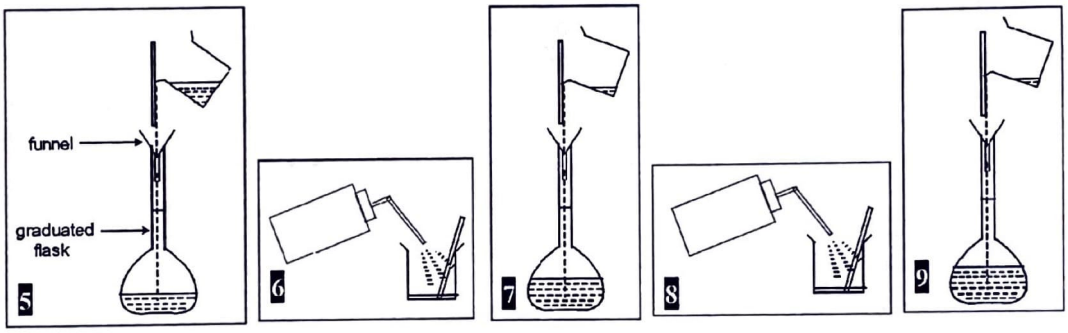
1.5 Preparing a Standard Solution

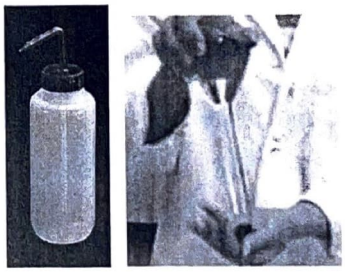
- A **volumetric flask (or graduated flask)** is used to prepare a solution of **known volume**. It is commonly used to prepare a **standard solution** (i.e. a solution of known concentration).
- Volumetric flasks are available in various capacities. For 'A' level experiments, **250 cm^3 volumetric flasks** are commonly used.
- The main steps involved in the preparation of a standard solution from a solid reagent are shown diagrammatically below.

1. The solid reagent is weighed using a weighing bottle, transferred quantitatively into a 100 cm^3 beaker and then dissolved in some deionised water.

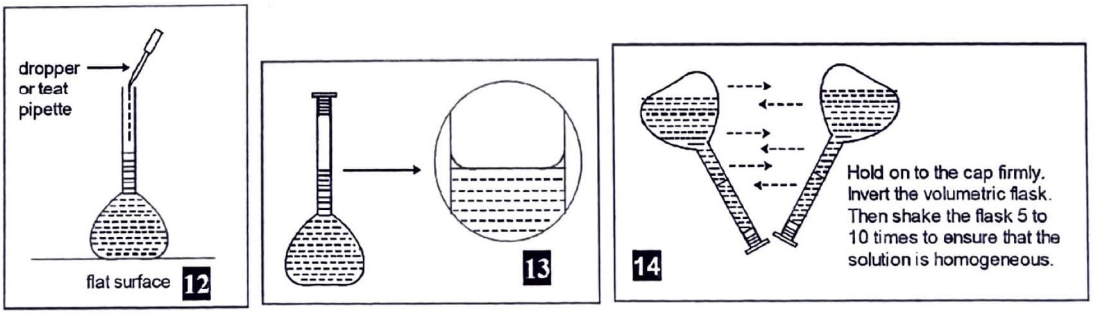


2. The resultant solution in the beaker is transferred quantitatively into a 250 cm^3 volumetric flask with aid of a glass rod and a funnel.

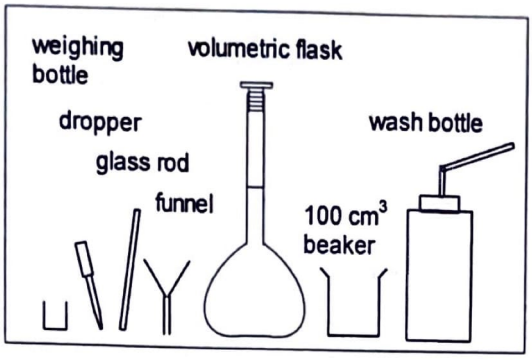




3. More deionised water is added into the volumetric flask until the 250 cm^3 mark is reached. The volumetric flask is then stoppered and the solution shaken to ensure that it is homogeneous.



- Common apparatus used for the preparation of a standard solution:



Do's

1. Wash the following apparatus with water and then rinse them with deionised water: volumetric flask, 100 cm^3 glass beaker, glass rod, funnel and dropper.
2. Place the **funnel** onto the volumetric flask.
3. **Transfer as much solid as possible** from the weighing bottle into the beaker carefully.
4. **Rinse the weighing bottle** with some deionised water (from the wash bottle) to help 'wash' and transfer the remaining small amount of solid from the weighing bottle into the beaker.
5. Rinse the weighing bottle a few times with deionised water and transfer all the washings into the beaker **with the aid of the glass rod**. Lean the weighing bottle against the glass rod and allow the washings to flow down the glass rod into the beaker.
6. Dissolve as much of the solid in the beaker by **stirring with the glass rod**. Add more deionised water if necessary. Ensure that the beaker is at most ¾ full to avoid spillage while stirring.
7. Transfer the solution from the beaker into the volumetric flask carefully with the **aid of the funnel and the glass rod**. Rest the spout of the beaker on the glass rod and pour the solution out carefully, using the glass rod to direct the solution towards the funnel. Ensure that any undissolved solid remains in the beaker.
8. Add more deionised water to dissolve the remaining solid in the beaker. Then transfer the solution into the volumetric flask as in step 7.
9. **Rinse the beaker, glass rod and funnel repeatedly with deionised water** and pour all the washings into the volumetric flask to ensure a complete transfer of the solid weighed out initially.
10. Remove the funnel from the volumetric flask.
11. **Fill the volumetric flask with deionised water** (from the wash bottle) **until the solution level is about 1 – 3 cm away from the mark** at the neck of the volumetric flask.
12. Add deionised water **drop-wise using a dropper** into the volumetric flask (placed on the bench) until the mark is reached. Check at eye-level that the bottom of the meniscus is just touching the mark.
13. Stopper the volumetric flask. Hold the stopper firmly, **invert the volumetric flask and shake the solution**. **Repeat** the inversion and shaking about 10 times to ensure that the solution is **homogeneous**. Label the solution (if necessary).

Don'ts

1. **Do not** add deionised water to the solid in the weighing bottle before transferring as much of the solid as possible into the small beaker.
2. **Do not** place the glass rod or the funnel onto the bench top unless they have been rinsed with deionised water and the washings transferred into the volumetric flask.
3. **Do not** use more deionised water than necessary for rinsing to avoid overshooting the required mark in the volumetric flask.
4. **Do not** lift the volumetric flask away from the bench top while filling it up to the mark with deionised water from the dropper. The volumetric flask should be placed on a flat surface, e.g. the bench top, when filling up to the mark.

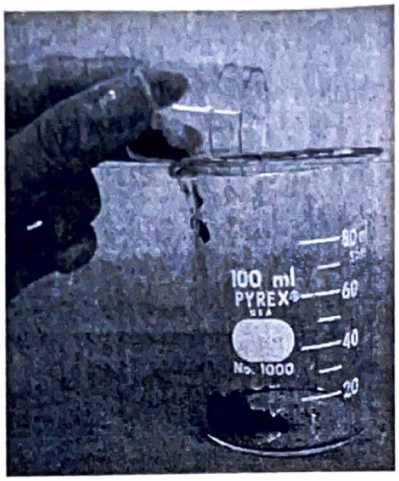


Figure 12: The solid in the weighing bottle is to be transferred **quantitatively** into a 100 cm^3 glass beaker and then dissolved in deionised water.

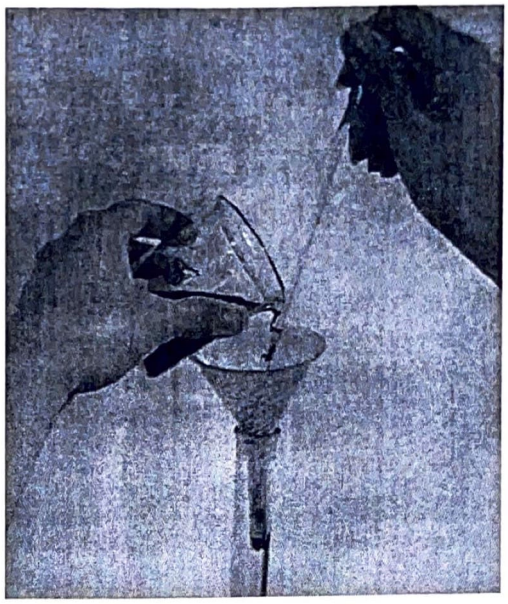


Figure 13: The transfer of the solution from the beaker to the volumetric flask is done with the aid of the glass rod and the funnel.

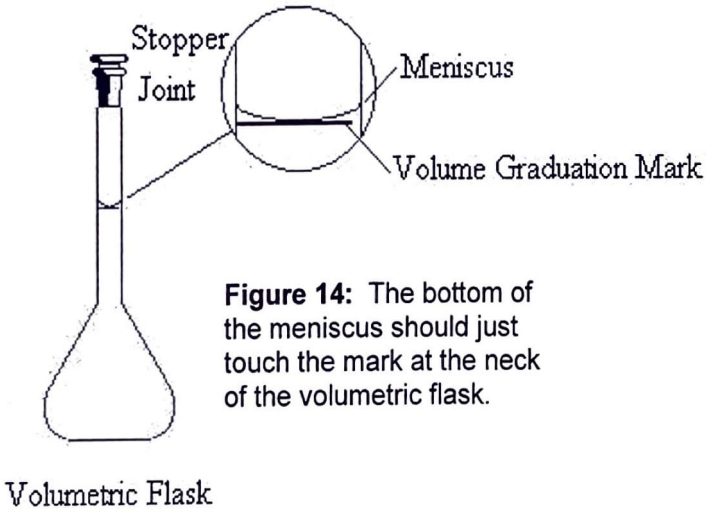


Figure 14: The bottom of the meniscus should just touch the mark at the neck of the volumetric flask.

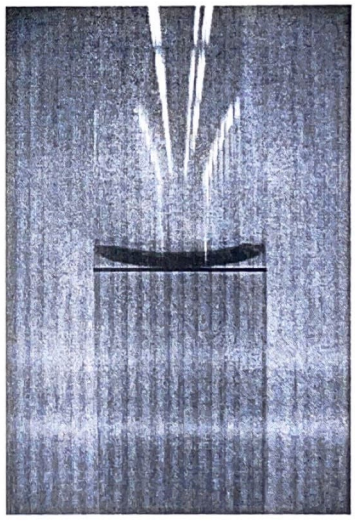


Figure 15: The bottom of the meniscus should just touch the mark at the neck of the volumetric flask. This should be checked at eye-level.

1.6 Summary on Manipulative Skills

(a) Checklist of manipulative skills

- The list below shows some of the manipulative skills that are needed when carrying out an experiment involving volumetric analysis.
- Go through the list and tick to indicate that you have taken note and can perform each of them.

		Action	Check (✓)
Standard solution preparation	1	Ensures that the weighing bottle is clean and dry.	
	2	Adds solid sample to the weighing bottle outside of analytical balance to avoid spillage.	
	3	Dissolves solid sample with deionised water in a small beaker.	
	4	Rinses volumetric flask with deionised water.	
	5	Transfers the solution into volumetric flask using a glass rod and filter funnel without spillage (include washings).	
	6	Adds deionised water drop-wise into the volumetric flask when near the mark.	
	7	Stoppers the volumetric flask and shakes the solution thoroughly.	
Use of pipette	8	Rinses all titration glassware with deionised water.	
	9	Rinses the pipette with the correct solution and dries the tip.	
	10	Handles the pipette safely when fitting the pipette filler.	
	11	Adjusts the liquid level in the pipette with the pipette tip above the liquid surface.	
	12	Removes the pipette filler without solution drawn/sucked into the pipette filler.	
	13	Drains out the solution in the pipette correctly.	
Use of burette	14	Rinses the burette with the correct solution and dries the tip.	
	15	Clamps the burette vertically.	
	16	Fills the burette at eye level or lower without climbing onto a stool.	
	17	Fills the burette with the aid of a filter funnel.	
	18	Ensures that there is no air bubble anywhere in the burette.	
	19	Removes the filter funnel from the burette after use.	
	20	Records the initial burette reading at eye-level before starting to titrate.	
Titration	21	Adds a few drops of the correct indicator at the start of the titration, except for iodometric titration.	
	22	Titrates with continuous swirling.	
	23	Ensures no spillage of solution during titration.	
	24	Ensures no solution added from the burette adheres to the inner walls of the conical flask during the titration by washing the inner walls with deionised water from the wash bottle whenever necessary.	
	25	Uses a white tile to help in colour change observation.	
	26	Adds the solution from the burette drop-wise near the end-point.	
	27	Stops titration at the correct end-point (e.g. when the solution turns from colourless to a permanent pale pink).	
	28	Takes burette reading at eye-level.	

(b) Discussion Questions

A student carried out a titration experiment in which solution **FA1** is titrated against solution **FA2** placed in the burette.	
1	Before filling up the burette with solution **FA2**, the student rinsed the burette first with deionised water and then with solution **FA2**. Explain the need for each rinsing.
2	Before the start of the first titration, the student rinsed the pipette with deionised water but did not rinse it with solution **FA1**. Explain the impact of this on the first titre volume.
3	Before the start of the first titration, the student rinsed the conical flask with deionised water but did not rinse it with solution **FA1**. Explain why the conical flask should not be rinsed with solution **FA1**.
4	After rinsing the conical flask with deionised water, the student proceeded to pipette 25.0 cm^3 of **FA1** into it. Explain why the presence of water droplets in the conical flask would not affect the titration result.
5	Explain why a conical flask and not a beaker was used for titration.
6	The student swirled the conical flask continuously during the titration. Explain the need for him to do so.
7	During titration, the student occasionally used the wash bottle to add deionised water to rinse the inner walls of the conical flask. Explain the need for him to do so.

1.7 Acid-base indicators

- The indicators used in acid-base titrations are generally organic dyestuffs which are weak acids or weak bases.

- In an acid-base titration, the indicator used is usually added in a small quantity to help to determine the end-point of the titration. Each indicator has a pH range over which it changes colour.

- The choice of an indicator for an acid-base titration depends on: (i) the type of titration carried out and (ii) the pH range of the indicator.

- Essential characteristics of a good indicator:

1) The colour change of the indicator must occur when the **correct volume of titrant** is added from the burette, i.e. the colour change takes place only when the reaction is complete.

2) The indicator must give a **sharp end-point** in the titration, i.e. it must change colour distinctly upon the addition of one drop of the titrant from the burette. It would be useless if 2 or 3 cm^3 of the titrant were necessary to bring about the colour change.

- Colour changes and pH ranges of selected indicators:

Indicator	Approximate pH range	Colour in 'acid' solution	Colour in 'alkaline' solution	Colour at the end-point
methyl orange	3.1 – 4.4	red	yellow	orange
screened methyl orange	3.1 – 4.4	violet	green	grey
thymol blue	1.2 – 2.8 8.0 – 9.6	red yellow	yellow blue	green
thymolphthalein	9.3 – 10.5	colourless	blue	light blue (if titrant is alkali) colourless (if titrant is acid)

2. Uncertainty (or error) in Measurement

2.1 Uncertainty in Measurement

- A measurement gives **quantitative information**. The result of nearly every measurement is **a number** and **a unit**.
- There is no such thing as a perfect measurement. Every measurement contains a degree of **uncertainty** (also called **error**). No device or instrument is capable of producing an exact result – a result with no uncertainty.
- Recording a measurement provides information about both its magnitude and uncertainty. When a scientist reports the result of a single measurement, he also reports the uncertainty in the result, a number indicating the reliability of the measuring device used.
- For example, the mass of a sample obtained using an electronic balance that weighs to the nearest 0.01 g may be reported as **32.46 ± 0.01** g. This means that the actual mass will be in the range **32.45 g to 32.47 g**.
  - Scientists sometimes report the above mass as **32.46 g**. Reporting the mass to 2 decimal places implies an uncertainty of 0.01 g.
- In science, measured values are reported in terms of **significant figures**.

  - A mass that is reported as **32.4 g** (to 3 sf) has an uncertainty of ± **0.1** g.
  - A mass that is reported as **32.40 g** (to 4 sf) has an uncertainty of ± **0.01** g.

- **Significant figures** in a measurement consist of all the digits known with certainty plus one final digit, which is somewhat **uncertain or is estimated**.
- **Note:**

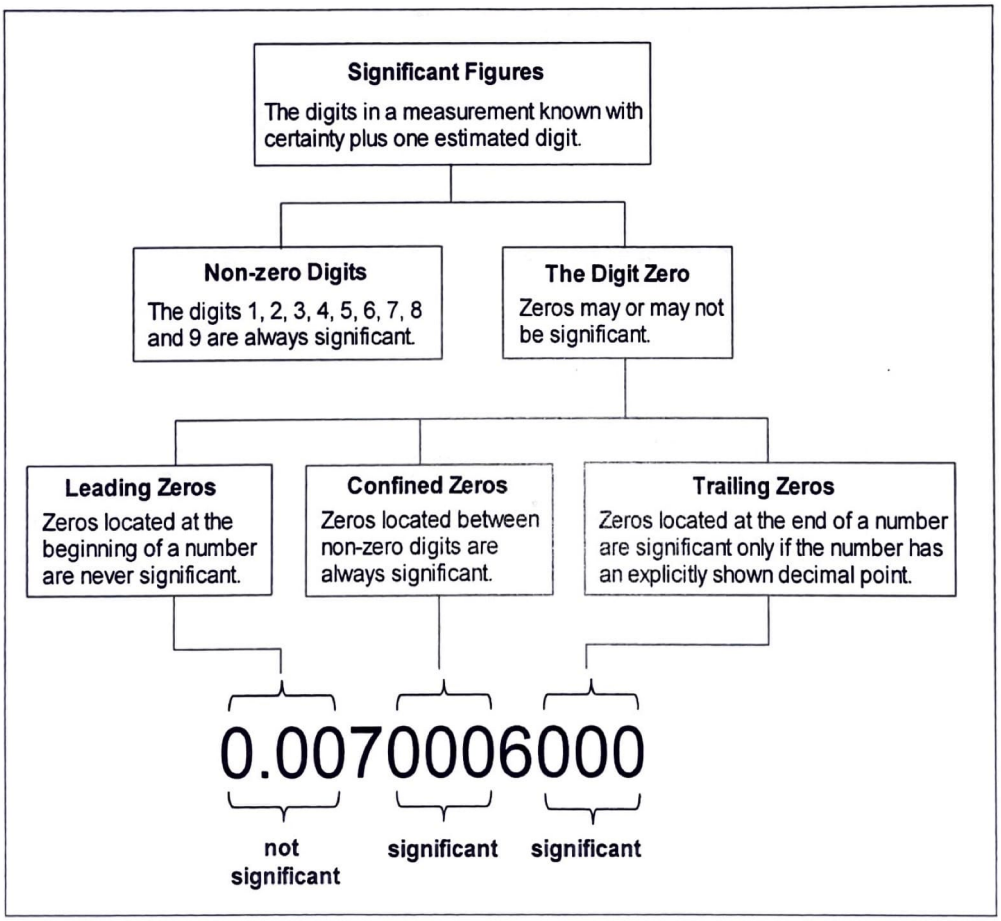
  - Analytical chemists make a distinction between **error** and **uncertainty**.
  - **Error** is the difference between a single measurement or result and its true value (or accepted value or literature value).
  - **Uncertainty** is an estimate that expresses the range of possible values that a measurement or result might reasonably be expected to have.
  - For simplicity at 'A' level, we will treat **uncertainty as being the same as error** when dealing with calculations involving **equipment/measurement error**. In this case, equipment/measurement error refers to the uncertainty that is associated with measurements made with that piece of equipment (i.e. the error that is intrinsic in the use of a particular piece of equipment).

2.2 Significant Figures

- Recording a measurement to the correct number of **significant figures** is important because the number of significant figures in a recorded reading is an indication of the degree of uncertainty in the reading.
- The general rules for counting the number of significant figures are shown below.

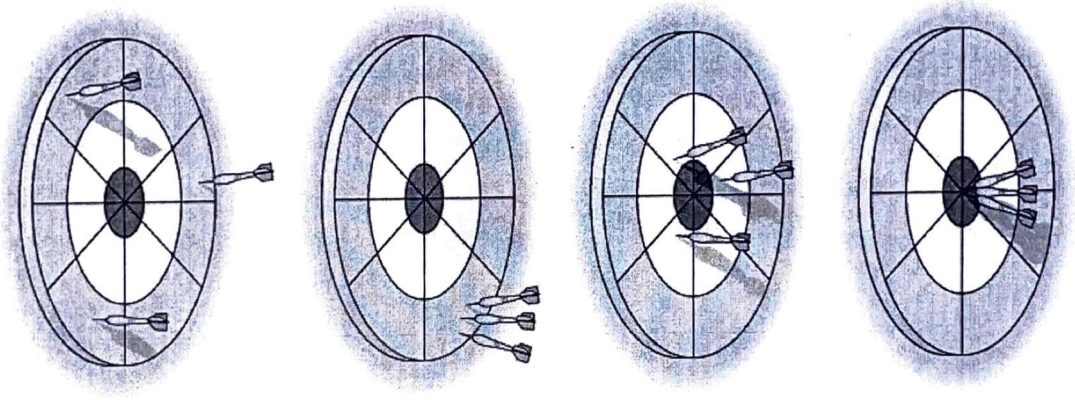
General Rules	Examples
1. All **non-zero digits** are significant.	54.3 (3 significant figures)
2. **Zeros between non-zero digits** are significant.	5009 (4 significant figures)
3. Zeros that are **simultaneously** to the right of the decimal point and at the end of the number are significant.	57.00 (4 significant figures)
4. Zeros which are to the left of a written decimal point and are in a number more than or equal to 10 are significant.	10.5 (3 significant figures)
5. Zeros that do not fulfil rules 2, 3 and 4 are **NOT** significant.	0.001 (1 significant figure)
Note: • For a number with zeros at the end and without a decimal point, the zeros are ambiguous when the counting of significant figures is concerned. • The ambiguity can be removed by reporting such numbers in **scientific notation**.	**Example: 3000** 3.000×10^3 (4 significant figures) 3.00×10^3 (3 significant figures) 3.0×10^3 (2 significant figures) 3×10^3 (1 significant figure)

- Another example:



2.3 Accuracy and Precision

- Two concepts that have to do with measurements are **accuracy** and **precision**.
- The **accuracy** of a measurement refers to how close the measured value is to the true, accepted or literature value. Measurement error is the amount of inaccuracy.
- **Precision** refers to how close together several measurements of the same quantity are to each other. It is the degree of consistency and agreement among several measurements made in the same manner.
  - Precision is commonly divided into two categories: **repeatability** and **reproducibility**.
  - **Repeatability** is the precision obtained when one experimenter carries out several measurements of the same quantity and they are in close agreement with each other.
  - **Reproducibility** is the precision obtained when different experimenters carry out the measurement of the same quantity and obtain readings that are in close agreement with each other.
- A dartboard analogy can be used to illustrate the difference between accuracy and precision.



(a) Low accuracy Low precision | (b) Low accuracy High precision | (c) High accuracy Low precision | (d) High accuracy High precision

  - Measurements that are close to the 'true' or 'correct' value are accurate.
  - Measurements that are close to each other are precise.

- Measurements can be
  - accurate and precise as in (d)
  - precise but inaccurate as in (b)
  - neither accurate nor precise as in (a)
- You must strive for both accuracy and precision in all of your laboratory experiments. Make sure that you understand the operation of each instrument, take each measurement carefully, and check to make sure that you have precision.

2.4 Precision of Instrument

- The **smallest division** of a measuring instrument determines the **precision** of the instrument.

Examples	Uncertainty of measurement	Recording
burette, thermometer	± (½ of the smallest division of the instrument)	Record all the digits that are known for certain plus one that is estimated.

- The following table summarises the precision of some equipment and apparatus commonly used in the chemistry laboratory.

No.	Apparatus	Smallest division	Uncertainty	Examples of recording	Number of decimal places
1	Analytical balance	0.001 g	±0.0005 g	3.456 g	3
		0.01 g	±0.005 g	3.46 g	2
2	Burette (50 cm^3)	0.1 cm^3	±0.05 cm^3 but may depend on the grade of the apparatus	25.55 cm^3, 20.50 cm^3	2
3	Pipette	– (no divisions)	depends on the grade of the apparatus	10.0 cm^3, 25.0 cm^3	1
4	Measuring cylinder (50 cm^3)	1 cm^3	±0.5 cm^3	22.0 cm^3	1
5	Stopwatch (digital)	0.01 s	±0.01 s	35.15 s (BUT commonly recorded as 35 s)	2
6	Thermometer (–5 °C to 50 °C)	0.2 °C	±0.1 °C	53.3 °C, 54.2 °C	1
7	Thermometer (–10 °C to 100 °C)	1 °C	±0.5 °C	53.0 °C, 53.5 °C	1
8	Volumetric Flask	– (no divisions)	depends on the grade of the apparatus	100 cm^3, 250 cm^3	0

2.5 Absolute Uncertainty and Percentage Uncertainty

- Consider the following quantity that is measured: **28.50 ± 0.01 g**

Absolute uncertainty	• The **absolute uncertainty** is the actual uncertainty in the value. • In this case, absolute uncertainty = ± 0.01 g
Percentage uncertainty	• The **percentage uncertainty** is the absolute uncertainty expressed as a percentage of the measured value i.e. $\text{Percentage uncertainty} = \frac{\text{absolute uncertainty}}{\text{measured value}} \times 100\ \%$ • In this case, percentage uncertainty = $\frac{(\pm 0.01)}{28.50} \times 100\ \%$ $= \pm 0.0351\ \%$ (3 s.f.)

- An uncertainty of ±0.01 g is **more significant** for a mass measurement of 1.00 g than it is for 100.00 g. This is shown by the different percentage uncertainties for the two measurements using the same analytical balance.

For 1.00 ± 0.01 g, the percentage uncertainty = $\frac{(\pm 0.01)}{1.00} \times 100\ \% = \pm 1.00\ \%$

For 100.00 ± 0.01 g, the percentage uncertainty = $\frac{(\pm 0.01)}{100.00} \times 100\ \% = \pm 0.0100\ \%$

2.6 Combining Uncertainties

- In an experiment, it is common that a number of measurements using different apparatus are made and a final result is calculated from these measurements.

- **Question**: How do we combine the uncertainties in the individual measurements to work out the overall uncertainty in the final result?

- The simplified approach in combining uncertainties shown on the next page depends on the mathematical operations involved in calculating the results.

(a) Addition and Subtraction of Readings

Consider the volume of a solution **FA1** measured using a burette.

Final burette reading /cm^3	37.20
Initial burette reading /cm^3	15.05
Volume of **FA1** /cm^3	22.15

What is the uncertainty associated with the volume of **FA1** measured?

Uncertainty in volume of **FA1** measured
= uncertainty in final burette reading + uncertainty in initial burette reading
= (±0.05) + (±0.05)
= ±0.10 cm^3

Note: For measurements involving 2 readings:
Uncertainty in measurement = uncertainty of final reading + uncertainty of initial reading

Proof

- The smallest division in a burette is 0.10 cm^3.
  Each burette reading has an uncertainty of ±0.05 cm^3.

  The final burette reading is in the range of 37.15 – 37.25 cm^3.
  The initial burette reading is in the range 15.00 – 15.10 cm^3.

- Maximum volume calculated = Maximum final reading – Minimum initial reading
  = 37.25 – 15.00
  = 22.25 cm^3

- Minimum volume calculated = Minimum final reading – Maximum initial reading
  = 37.15 – 15.10
  = 22.05 cm^3

- Therefore **volume of FA1 = 22.15 ± 0.10 cm^3**
- Hence the uncertainty associated with the volume of **FA1** is **2(± 0.05) = ± 0.10 cm^3**

- **Note:**
  The volume of **FA1** calculated depends on the difference between the two burette readings and the uncertainty is **the sum of the two absolute uncertainties of the two measurements.**

- **Note:**

When adding or subtracting measurements, the uncertainty in the calculated result is **the sum of the absolute uncertainties of the individual measurements**.

(b) Multiplication and Division of Readings

- Consider the calculation of density from mass and volume measurements.

	Value	Absolute uncertainty	% uncertainty
Mass/ g	24.0	(2)(±0.05) = ±0.10	$\frac{(\pm 0.1)}{24.0} \times 100\ \% = \pm 0.417\ \%$
Volume/ cm^3	2.00	±0.10	$\frac{(\pm 0.10)}{2.00} \times 100\ \% = \pm 5.00\ \%$

Density = 24.0 / 2.00 = 12.0 g cm^{-3}

Total % uncertainty = Sum of % uncertainties of mass and volume = (±0.417) + (±5.00) = ±5.42 % (3 s.f.)

Absolute uncertainty of density = ±5.42 / 100 x 12.0 = ±0.650 g cm^{-3} (3 s.f.)

- **Note:**

- When multiplying or dividing measurements, **the total percentage uncertainty is the sum of the individual percentage uncertainties**.
- The **absolute uncertainty** in the result can then be calculated from the percentage uncertainty.

(c) Summary – Combining uncertainties

- The absolute uncertainties in individual measurements can be combined to calculate the uncertainty in the final value of the quantity being determined.
- The following simple rules should be applied:

To find the absolute uncertainty in a calculated value, **(*a*+*b*) or (*a*–*b*)**	• The absolute uncertainty in the calculated value is the sum of the absolute uncertainties in *a* and in *b*. o absolute uncertainty in (*a*+*b*) = absolute uncertainty in *a* + absolute uncertainty in *b* o absolute uncertainty in (*a*–*b*) = absolute uncertainty in *a* + absolute uncertainty in *b*
To find the absolute uncertainty in a calculated value, **(*a* x *b*) or (*a* ÷ *b*)**	• Find the percentage uncertainty in *a* and *b*. • Add the percentage uncertainties of *a* and *b* to find the percentage uncertainty in the calculated value. • Convert this percentage uncertainty to absolute uncertainty for the calculated value.

2.7 Uncertainties (errors) in Burette Readings

(a)	What is the smallest division in a burette?	
(b)	What is the uncertainty (or error) in a burette reading?	
(c)	What is the uncertainty in a volume of a solution measured using a burette?	
(d)	What is the percentage uncertainty associated with 24.60 cm^3 of **FA2** measured using a burette?	
(e)	Explain how a titre value may have a total error of –0.10 cm^3.	

- Consider the volume of a solution **FA2** measured using a burette. Suppose the true value for the initial burette reading is **20.00 cm^3** and the true value for the final burette reading is **45.00 cm^3** such that the true value for the volume of **FA2** is **25.00 cm^3**.

  Explain how the volume of **FA2** measured may have an error of
  (a) +0.10 cm^3, **(b)** +0.05 cm^3, **(c)** no error, **(d)** –0.05 cm^3, and **(e)** –0.10 cm^3.

Possible error made	Initial burette reading	Final burette reading	Explanation
(a) +0.10	19.95	45.05	• The initial burette reading has a **–0.05 cm^3 error** while the final burette reading has a **+0.05 cm^3 error.** • Hence the error made in the volume of FA2 is **+0.10 cm^3**. • Volume of FA2 = 45.05 – 19.95 = 25.10 cm^3 (* differs from the actual true value by +0.10 cm^3)
(b) +0.05	20.00 **OR**	45.05	• The initial burette reading has **no error** while the final burette reading has **+0.05 cm^3 error.** • Hence the error made in the volume of FA2 is **+0.05 cm^3**. • Volume of FA2 = 45.05 – 20.00 = 25.05 cm^3 (* differs from the actual true value by +0.05 cm^3)
	19.95	45.00	• The initial burette reading has **–0.05 cm^3 error** while the final burette reading has **no error.** • Hence the error made in the volume of FA2 is **+0.05 cm^3**. • Volume of FA2 = 45.00 – 19.95 = 25.05 cm^3 (* differs from the actual true value by +0.05 cm^3)
(c) no error	20.05 **OR**	45.05	• The initial burette reading has a **+0.05 cm^3 error** while the final burette reading has **+0.05 cm^3 error.** • Hence there was **no error** made in the volume of FA2. • Volume of FA2 = 45.05 – 20.05 = 25.00 cm^3 (* does not differ from the actual true value)
	20.00 **OR**	45.00	• The initial burette reading has **no error** and the final burette reading also has **no error.** • Hence there was **no error** made in the volume of FA2.
	19.95	44.95	• The initial burette reading has a **–0.05 cm^3 error** and the final burette reading has **–0.05 cm^3 error.** • Hence there was **no error** made in the volume of FA2. • Volume of FA2 = 44.95 – 19.95 = 25.00 cm^3 (* does not differ from the actual true value)
(d) –0.05	20.00 **OR**	44.95	• The initial burette reading has **no error** while the final burette reading has a **–0.05 cm^3 error.** • Hence the error made in the volume of FA2 is **–0.05 cm^3**. • Volume of FA2 = 44.95 – 20.00 = 24.95 cm^3 (* differs from the actual true value by –0.05 cm^3)
	20.05	45.00	• The initial burette reading has a **+0.05 cm^3 error** while the final burette reading has **no error.** • Hence the error made in the volume of FA2 is **–0.05 cm^3**. • Volume of FA2 = 45.00 – 20.05 = 24.95 cm^3 (* differs from the actual true value by –0.05 cm^3)
(e) –0.10	20.05	44.95	• The initial burette reading has a **+0.05 cm^3 error** and the final burette reading has a **–0.05 cm^3 error.** • Hence the error made in the volume of FA2 is **–0.10 cm^3**. • Volume of FA2 = 44.95 – 20.05 = 24.90 cm^3 (* differs from the actual true value by –0.10 cm^3)

3. Recording of Measurements

3.1 Recording Mass Readings and Calculating Percentage Uncertainty

- For 'A' level laboratory work, an analytical balance that is capable of reading to **0.001 g** is commonly used. In this case, record the mass readings to **3 decimal places**, e.g. 4.153 g.
- The common ways of recording mass readings are shown below.

<table>
<tr><th></th><th>Method of weighing</th><th>Method of recording mass readings and calculation of percentage uncertainty</th></tr>
<tr><td>(a)</td><td>Direct weighing

• direct weighing of the reagent (e.g. FA1) in a weighing bottle is carried out with the aid of the 'tare' button of the analytical balance
• the mass of the empty weighing bottle is <u>not</u> needed
• the reagent can be transferred quantitatively from the weighing bottle to the desired vessel</td><td>• Write a statement to report the mass of the reagent used:
Mass of FA1 = 4.134 g (TARE)
Uncertainty = (2)(±0.0005) = ±0.001 g
% uncertainty = $\frac{(\pm 0.001)}{4.134} \times 100\ \%$
= ±0.0242% (3 s.f.)</td></tr>
<tr><td>(b)</td><td>Weighing by difference

• the reagent (e.g. FA1) can be transferred quantitatively from the weighing bottle to the desired vessel
• the mass of the reagent is obtained by taking the difference between the first two readings
• not commonly used since direct weighing can be done
• used when the mass of the empty weighing bottle is needed</td><td>• Use a table to report the mass readings:
<table><tr><td>Mass of weighing bottle and FA1 / g</td><td>7.134</td></tr><tr><td>Mass of <u>empty</u> weighing bottle / g</td><td>3.000</td></tr><tr><td>Mass of FA1 used / g</td><td>4.134</td></tr></table>Uncertainty = (2)(±0.0005) = ±0.001 g
% uncertainty = $\frac{(\pm 0.001)}{4.134} \times 100\ \%$
= ±0.0242% (3 s.f.)</td></tr>
<tr><td>(c)</td><td>Weighing by difference

• the reagent (e.g. FA1) <u>cannot</u> be transferred quantitatively from the weighing bottle to the desired vessel
• the actual mass of the reagent (e.g. FA1) used in the experiment is obtained by taking the difference between the first two readings
• the mass of the empty weighing bottle is <u>not</u> needed
• the mass of the <u>emptied</u> weighing bottle is needed (i.e. the mass of the weighing bottle after emptying the reagent into the desired vessel)
• <u>reweighing</u> of the emptied weighing bottle is needed as some residual FA1 will stay in the emptied weighing bottle</td><td>• Use a table to report the mass readings:
<table><tr><td>Mass of weighing bottle and FA1 / g</td><td>7.134</td></tr><tr><td>Mass of weighing bottle <u>after emptying</u> FA1 / g</td><td>3.000</td></tr><tr><td>Mass of FA1 used / g</td><td>4.134</td></tr></table>Uncertainty = (2)(±0.0005) = ±0.001 g
% uncertainty = $\frac{(\pm 0.001)}{4.134} \times 100\ \%$
= ±0.0242% (3 s.f.)
Note:
• In this case, quantitative transfer of FA1 from the weighing bottle to the desired vessel cannot be carried out. Possible reasons include:
- FA1 is insoluble in water
- Design of the experiment</td></tr>
</table>

3.2 Recording Burette Readings

- Record burette readings to **2 decimal places** and to the nearest 0.05 cm^3 (i.e. the **last digit is either a '0' or '5'**).
- Ensure that a sufficient number of titrations have been carried out i.e. obtain at least two **consistent** titration results **(i.e. two titres that are within 0.10 cm^3 of each other).**
- Record burette readings using a table as shown below.

Titration results

Titration number	1	2	
Final burette reading /cm^3	24.05	48.15	
Initial burette reading /cm^3	0.00	24.05	
Volume of **FA2** used /cm^3	24.05	24.10	
Values used (✓)	✓	✓	

- Burette readings
- 2 decimal places

Average volume of **FA2** used =

- Show working
- 2 decimal places

Colour change at the end-point: _____

Volume of **FA1** pipetted = **25.0 cm^3**

- Pipette reading
- 1 decimal place

Summary:

_____ cm^3 of **FA1** required _____ cm^3 of **FA2** for complete reaction

- Do take note of the following when presenting primary and secondary data:

1. Record **mass readings** to **3 decimal places.**
2. Record **burette readings** to **2 decimal places.**
3. Record **pipette readings** to **1 decimal place.**
4. Present **intermediate answers** (i.e. part of calculation working) to **3 or 4 significant figures.**
5. Present **final answer** to each part to **3 significant figures.**
6. Present stoichiometric coefficients such as x **in $CuSO_4.xH_2O$** to the **nearest whole number.**
7. Relative molecular mass (M_r) and relative formula mass (M_r) **calculated using A_r values** from the given Periodic Table should always be quoted to **1 decimal place.**

   Examples: M_r of $CaCO_3$ = 40.1 + 12.0 + (3)(16.0) = 100.1 (1 decimal place)

   M_r of $BaCl_2$ = 137.3 + (2)(35.5) = 208.3 (1 decimal place)

   However a M_r (or A_r) value **determined experimentally** should be presented to **3 significant figures**.

4. Treatment of Anomalous Results

- In every experiment, the student should
  - identify anomalous observations/results with reasons indicated
  - repeat measurements if there are anomalous results
  - suggest possible causes for the anomaly
  - explain the impact of the anomaly on the results

- If the results are consistent, repeat measurements are **not** necessary but possible reasons for the consistent results should be given.

- Treatment of anomalous results in a titration experiment:

		Consistent results	Anomalous results
1	State and explain if the experimental results are consistent.	As titration 1 and 2 readings are within 0.10 cm^3 of each other, the readings are consistent.	As titration 1 and 2 readings are more than 0.10 cm^3 apart, the readings are not consistent.
2	State whether any result is anomalous and whether repeat measurements are required.	Since there is no anomalous result, a third titration was not attempted.	Since one of the readings is anomalous, a third titration was done.
3	If repeat measurements are made, comment if the subsequent results are consistent or anomalous.	–	Titration 1 reading is anomalous. Since titration 2 and 3 readings are within 0.10 cm^3 of each other, they are consistent.
4	Give reasons for the consistent or anomalous results. If repeat measurements are made, give reasons for the subsequent consistent results. **Note:** When commenting on anomalous results, ensure that the reasons given are relevant and contribute to the anomalous results.	Consistent readings were obtained due to proper pipetting and titration techniques being adopted. For example, the titrant (i.e. **FA1** in the burette) was added drop-wise when nearing the end-point of the titration to ensure that the latter was not exceeded. Also, swirling of the reaction mixture in the conical flask was done continuously during titration to ensure even mixing of the reactants.	• **A higher titre is obtained** Titration reading 1 **is anomalous and higher** than the other readings probably because the titrant (i.e. **FA1** from the burette) was not added drop-wise when approaching the end-point of the titration. Hence the end-point was exceeded with slightly more titrant added. This titration was meant to be a rough one to get an approximate titre value quickly. • **A lower titre is obtained** Titration reading 1 **is anomalous and lower** than the other readings probably because the pipette (initially rinsed with deionised water) was not rinsed sufficiently with the **FA2** solution when this titration was carried out such that the pipetted **FA2** solution was slightly diluted when used. Titration readings 2 and 3 are consistent because proper pipetting and titration techniques were adopted. For example, the titrant (i.e. **FA1** in the burette) was added drop-wise when nearing the end-point of the titration to ensure that the latter was not exceeded.

- **Note:** Do comment according to the data obtained!
  - As titration 1 and 2 readings are **the same**, the readings are consistent.
  - As titration 1 and 2 readings are **within 0.10 cm^3 of each other**, the readings are consistent.
  - As titration 1 and 2 readings are **are more than 0.10 cm^3 apart**, the readings are not consistent.

- **Worked Example 1:** An acid-base titration in which **FA2** is titrated against **FA1**.

Titration results

Titration number	1	2
Final burette reading /cm^3	25.05	40.10
Initial burette reading /cm^3	0.00	15.00
Volume of **FA1** used /cm^3	25.05	25.10
Values used	✓	✓

25.075, when rounded off to 2 decimal places, is 25.08

Average volume of **FA1** required = (25.05 + 25.10) / 2 = 25.08 cm^3

Volume of **FA2** pipetted = 25.0 cm^3

Summary: 25.0 cm^3 of **FA2** required 25.08 cm^3 of **FA1** for complete reaction.

Comment on the consistency of the titre values obtained. Identify, and suggest an explanation for, the presence or absence of anomalous results in these titre values.

- As titration 1 and 2 readings are **within 0.10 cm^3 of each other**, the readings are **consistent**.
- Since there is **no anomalous result**, a **third titration was not attempted**.
- Consistent readings were obtained due to proper pipetting and titration techniques being adopted. For example, the titrant (i.e. **FA1** in the burette) was added drop-wise when nearing the end-point of the titration to ensure that the latter was not exceeded. Also, swirling of the reaction mixture in the conical flask was done continuously during titration to ensure even mixing of the reactants.

- **Worked Example 2:** An acid-base titration in which **FA2** is titrated against **FA1**.

Titration results

Titration number	1	2	3
Final burette reading /cm^3	25.30	25.05	40.05
Initial burette reading /cm^3	0.00	0.00	15.00
Volume of **FA1** used /cm^3	25.30	25.05	25.05
Values used		✓	✓

Average volume of **FA1** required = 25.05 cm^3

Volume of **FA2** pipetted = 25.0 cm^3

Summary: 25.0 cm^3 of **FA2** required 25.05 cm^3 of **FA1** for complete reaction.

Comment on the consistency of the titre values obtained. Identify, and suggest an explanation for, the presence or absence of anomalous results in these titre values.

- As titration readings 1 and 2 are **more than 0.10 cm^3 apart**, the readings are **not consistent**. Since one of the readings is **anomalous**, a **third titration was done**.
- Titration readings 2 and 3 are the same and hence they are consistent. Proper pipetting and titration techniques were adopted to obtain consistent readings. For example, the titrant (i.e. **FA1** in the burette) was added drop-wise when nearing the end-point of the titration to ensure that the end-point was not exceeded.
- Titration reading 1 **is anomalous and higher** than the other readings probably because the titrant (i.e. **FA1** from the burette) was not added drop-wise when approaching the end-point of the titration. Hence the end-point was exceeded with slightly more titrant added. This titration was meant to be a rough one to get an approximate titre value quickly.

5. Examples on Uncertainty (or error) Calculations

- **Worked Example 3**

Two students used different balances to weigh the masses of two samples of lithium nitrate and recorded their readings as shown in the table below.
Student A used a balance capable of reading to 0.1 g.
Student B used a balance capable of reading to 0.01 g.

	Student A	Student B
Mass of crucible and lid /g	5.1	4.16
Mass of crucible and lid and $LiNO_3$ /g	20.9	4.86

Show by calculation, using values from the table, that any uncertainty (or error) in the mass of lithium nitrate recorded is less significant for student A than for student B.

Solution

	Student A	Student B
Mass of $LiNO_3$ /g	20.9 – 5.1 = 15.8	4.86 – 4.16 = 0.70
Uncertainty /g	(2)(±0.05) = ±0.100	(2)(±0.005) = ±0.0100
Percentage uncertainty	$\frac{(\pm 0.1)}{15.8} \times 100\ \%$ = ±0.633% (3 s.f.)	$\frac{(\pm 0.01)}{0.70} \times 100\ \%$ = ±1.43% (3 s.f.)

Since the **percentage uncertainty** associated with the mass of $LiNO_3$ weighed by student A is **smaller** than that of student B, the uncertainty in the mass of $LiNO_3$ recorded is less significant for student A.

- **Worked Example 4**

A student carried out an experiment to determine the concentration of aqueous sodium hydroxide. He weighed out a mass of 0.753 g of ethanedioic acid dihydrate, $(COOH)_2.2H_2O$. The solid was dissolved in deionised water and made up to 250 cm^3 in a graduated flask. 25.0 cm^3 of this solution was pipetted and titrated against an aqueous solution of sodium hydroxide using thymolphthalein as an indicator. A titre volume of 23.20 cm^3 of sodium hydroxide was obtained.

The errors (uncertainties) associated with each reading using a graduated flask, pipette and burette are ±0.15 cm^3, ±0.1 cm^3 and ±0.05 cm^3 respectively.

Calculate the maximum total percentage error (uncertainty) of the concentration of sodium hydroxide.

Solution

Percentage uncertainty (error) in using the **electronic balance** = $\left(\frac{\pm 0.0005 \times 2}{0.753}\right) \times 100\ \%$
= **±0.133 %** (3 s.f.)

Percentage uncertainty (error) in using the **graduated flask** = $\left(\frac{\pm 0.15}{250}\right) \times 100\ \%$
= **±0.0600 %** (3 s.f.)

Percentage uncertainty (error) in using the **pipette** = $\left(\frac{\pm 0.1}{25.0}\right) \times 100\ \%$ = **±0.400 %** (3 s.f.)

Each burette reading has an uncertainty (error) of ±0.05 cm^3.
Since the volume of NaOH measured required 2 burette readings to be taken (i.e. the initial reading and the final reading),
the total uncertainty (error) in using the burette is ±0.10 cm^3.

Percentage uncertainty (error) in using the **burette** = $\left(\frac{\pm 0.10}{23.20}\right) \times 100\ \%$ = **±0.431 %** (3 s.f.)

Hence **maximum total percentage error (uncertainty)** of the concentration of sodium hydroxide
= (±0.133) + (±0.0600) + (±0.400) + (±0.431)
= **±1.02 %** (3 s.f.)

- **Worked Example 5**

A student performed an experiment by mixing 35 cm^3 of dilute sulfuric acid and 20 cm^3 of potassium hydroxide in a Styrofoam cup, and measuring the maximum temperature of the resulting mixture.

Calculate the maximum total percentage uncertainties (errors) in the **volume of the reaction mixture** for the experiment when using the following to measure the volumes of sulfuric acid and potassium hydroxide:

(1) burettes
(2) measuring cylinders, given that the uncertainty (error) associated with each reading using a 50 cm^3 measuring cylinder is ±0.5 cm^3.

Solution

(1) When using burettes,

maximum total percentage uncertainty = $\left(\frac{(\pm 0.05)(2)+(\pm 0.05)(2)}{35.00 + 20.00}\right)(100\%)$ = **±0.364%** (3 s.f.)

(2) When using measuring cylinders,

maximum total percentage uncertainty = $\left(\frac{\pm(0.5+0.5)}{35.0 + 20.0}\right)(100\%)$ = **±1.82%** (3 s.f.)