CONTENTS

1. VOLUMETRIC ANALYSIS (VA)

Prerequisite Knowledge from Mole Concept lecture notes (non-exhaustive):

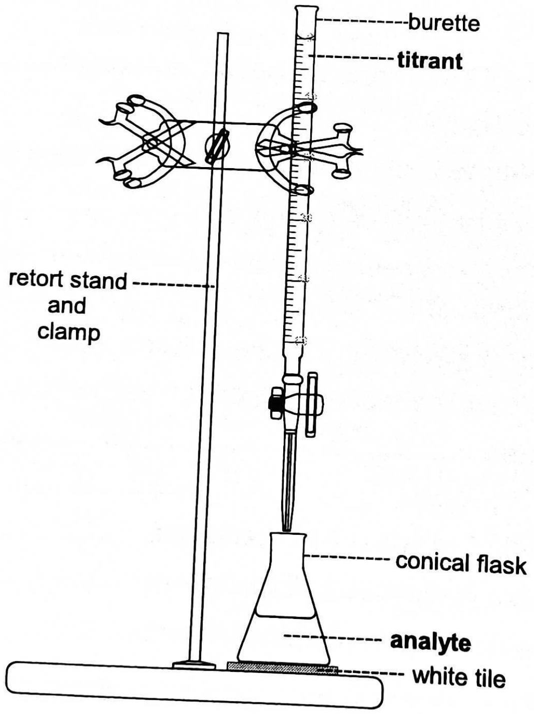
- Construct balanced equations
- Determine mole ratio and limiting reagent
- Calculate volume and concentration of solutions, including those involving dilution process and back titration

Prerequisite Knowledge from Redox Chemistry lecture notes (non-exhaustive):

- Construct half equations and overall equations in acidic and alkaline medium
- Calculations involving redox titrations (Potassium Manganate(VII) & Iodometric titration)

1.1 Introduction

- Volumetric analysis (VA) is a method of **quantitative analysis** which depends essentially on the **accurate measurements** of the **volumes** of two solutions which react together completely.
- This is done through a titration process which involves gradual addition of one solution (known as the *titrant*) to a fixed volume of another (known as the *analyte*) until equivalence is reached i.e. stoichiometric amounts of the two reactants have reacted.



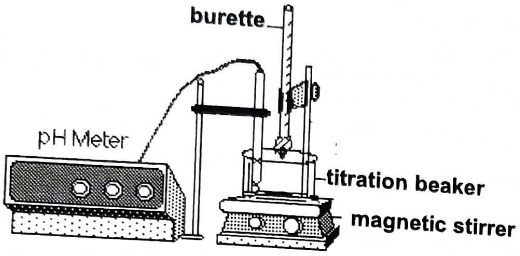
- A standard solution is used in the titration process to determine the concentration of other solution. A **standard solution is one with a known concentration** i.e. a known amount of solute in a given volume of solution (typically 1 dm^3).
- Four types of reactions that can be analysed during titration include:
  1. Acid-base reaction (more details under *Acid-Base Equilibria* in Y6)
  2. Redox reaction
  3. Precipitation (covered in *Solubility Equilibria* in Y6)
  4. Complexation (covered in *Chemistry of Transition Elements* in Y6)

This handbook will focus primarily on acid-base and redox reactions.

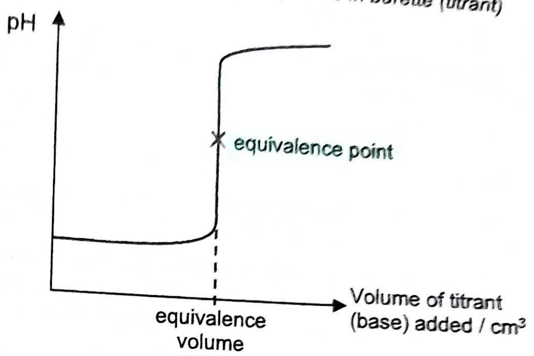
1.2 Acid-Base Titration

- In an acid-base titration, the equivalence point corresponds to the point when stoichiometric amounts of acid and base have been reacted. It generally involves a **rapid pH change** over this point.

- The pH changes during an acid-base titration may be monitored using a pH meter and a datalogger, following which a titration curve can be plotted to determine the equivalence point.



acid in conical flask (analyte); base in burette (titrant)



- Alternatively, a pH indicator can be used in an acid-base titration. An indicator is a solution which undergoes a distinct colour change. When the indicator **first changes colour permanently**, it marks the **end-point** of the titration process.

- Each pH indicator changes colour over its characteristic pH transition range in solution. Some common indicators are given below.

indicator	pH range	colour		
		in acid	at end point	in alkali
methyl orange	3.2 – 4.4	red	pale orange	yellow
screened methyl orange	3 – 5	magenta	grey	green
methyl red	4 – 6	red	pale orange	yellow
thymolphthalein	9.4 – 10.6	colourless	pale blue	blue
thymol blue *(commonly used for double indicator titration)*	1.2 – 2.8 from red to yellow 8.0–9.6 from yellow to blue	red	yellow or pale blue	blue

- The choice of pH indicator is considered appropriate if its pH transition range **lies within** the range of rapid pH change over the equivalence point of the titration (shown as a 'step' in the titration curve). It will give a **sharp end-point in which a colour change occurs distinctly when one drop of titrant is added.**

The table below lists some common indicators used for different types of acid-base titration.

type of titration	suitable indicator
strong acid – strong base	methyl orange, screened methyl orange, methyl red, thymolphthalein
strong acid – weak base	methyl orange, screened methyl orange, methyl red
weak acid – strong base	thymolphthalein, thymol blue
weak acid – weak base	no suitable indicator

1.3 Redox Titration

Two common redox titrations are the potassium manganate(VII) and iodometric titrations.

1.3.1 Potassium Manganate(VII) ($KMnO_4$) Titration

- $KMnO_4$ is a strong oxidising agent commonly used in redox titrations to react with a wide range of reducing agents. Titration is carried out in **acidic medium using dilute H_2SO_4**.

$$\underset{\textit{purple}}{MnO_4^-(aq)} + 8H^+(aq) + 5e^- \rightarrow \underset{\textit{colourless}}{Mn^{2+}(aq)} + 4H_2O(l)$$

- Other acids such as dilute HCl and HNO_3 are not suitable.
  - Dilute HCl can be oxidised by MnO_4^- to give Cl_2.
  - Dilute HNO_3 is also a strong oxidising agent and may interfere with the oxidising action of MnO_4^-.

- **No indicator is required** since there is a distinct colour change at the end of reaction.

- $KMnO_4$ is usually the titrant i.e. placed in the burette. It is usual to **read the top**, rather than the bottom, **of the meniscus** in the burette because the purple colour obscures the reading at the bottom.

- During titration, the conical flask is **continuously swirled** to avoid the formation of brown MnO_2(s) which makes determination of end-point difficult. The end-point of the titration is taken when permanent pale pink (or pale orange if Fe^{2+} is involved) first appears by one excess drop of $KMnO_4$ added into the conical flask.

- Some examples of redox titration involving $KMnO_4$ are shown below.

reducing agent	**half–equation**	**mole ratio**	**end-point**
$FeSO_4$ $Fe(NH_4)_2(SO_4)_2$	$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$	$MnO_4^- \equiv 5Fe^{2+}$	pale orange
$Na_2C_2O_4$ $H_2C_2O_4$ $(NH_4)_2C_2O_4$	$C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e^-$ (involves heating at ~60°C)	$2MnO_4^- \equiv 5C_2O_4^{2-}$	pale pink
H_2O_2	$H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$	$2MnO_4^- \equiv 5H_2O_2$	

1.3.2 Iodometric Titration (I_2–$S_2O_3^{2-}$)

- The iodometric titration involves I_2 as the oxidising agent and $S_2O_3^{2-}$ (thiosulfate ion) as the reducing agent.

reduction half equation:	$I_2(aq) + 2e^- \rightarrow 2I^-(aq)$
oxidation half-equation:	$2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2e^-$
overall redox equation:	$\underset{\textit{brown}}{I_2(aq)} + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + \underset{\textit{colourless}}{S_4O_6^{2-}(aq)}$

- An experiment involving iodometric titration typically involves two steps:
  1. A substance is added to acidified potassium iodide solution to liberate iodine.
  2. The liberated iodine is then estimated by titrating against standard sodium thiosulfate.

  Hence, the iodometric titration enables one to analyse the concentration of an oxidising agent in a sample. Some examples are listed below.

oxidising agent	overall equation for oxidation of iodide	mole ratio
$KMnO_4$	$2MnO_4^- + 10I^- + 16H^+ \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O$	$2MnO_4^- \equiv 5I_2 \equiv 10S_2O_3^{2-}$
$K_2Cr_2O_7$	$Cr_2O_7^{2-} + 6I^- + 14H^+ \rightarrow 2Cr^{3+} + 3I_2 + 7H_2O$	$Cr_2O_7^{2-} \equiv 3I_2 \equiv 6S_2O_3^{2-}$
$Fe_2(SO_4)_3$	$2Fe^{3+} + 2I^- \rightarrow 2Fe^{2+} + I_2$	$2Fe^{3+} \equiv I_2 \equiv 2S_2O_3^{2-}$
$K_2S_2O_8$	$S_2O_8^{2-} + 2I^- \rightarrow 2SO_4^{2-} + I_2$	$S_2O8^{2-} \equiv I_2 \equiv 2S_2O_3^{2-}$

- Starch indicator is used to detect the end-point of the titration more accurately since the colour change from brown to pale yellow to colourless is less distinct. The colour changes during the titration are as follows:

$$\underset{(I_2)}{\text{Brown}} \xrightarrow{\text{add } S_2O_3^{2-}} \text{yellow} \xrightarrow{\text{add } S_2O_3^{2-}} \text{pale yellow} \xrightarrow{\boxed{\textbf{add starch}}} \text{blue-black} \xrightarrow{\text{add } S_2O_3^{2-}} \underset{\text{(end-point)}}{\text{colourless}}$$

- Starch is added only when the solution is pale yellow. Otherwise, more iodine molecules will be strongly adsorbed on the starch molecules to form the blue-black complex. As the release of iodine molecules from the complex to react with $S_2O_3^{2-}$ takes time, this causes the colour change to occur less readily and reduces the accuracy of the titration.

- **1 cm³ (10 drops) of starch indicator** is typically used. The blue-black complex formed will turn colourless when one excess drop of $S_2O_3^{2-}$ is added to the conical flask.

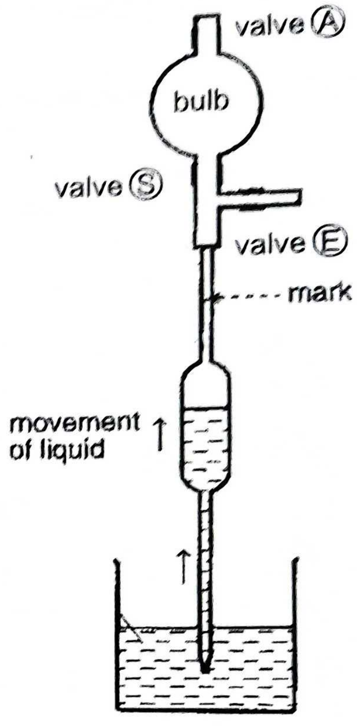
- After the titration, the mixture in the flask will slowly return from colourless to blue-black. This is due to atmospheric oxidation of excess I^- to I_2 and this I_2 formed should be ignored.

1.4 Experimental Techniques in Volumetric Analysis & Data Presentation

1.4.1 Use of Pipette and Pipette Filler

Filling a Pipette using a Pipette Filler

Tip: When using a pipette filler, do not push it too far onto the pipette as it will fail to suck up efficiently.



filling a pipette

- Rinse pipette with deionised water followed by the solution it is to be filled with.
- Squeeze the bulb of the pipette filler and valve **A** to let air out.
- Immerse the tip of pipette into the solution. Squeeze valve **S** to suck up the solution to a level **above** the graduation mark.
- Raise the pipette such that the tip is above the surface of the solution. Squeeze valve **E** under control to expel solution until the meniscus just touches the graduation mark.

Delivering Solution into a Conical Flask

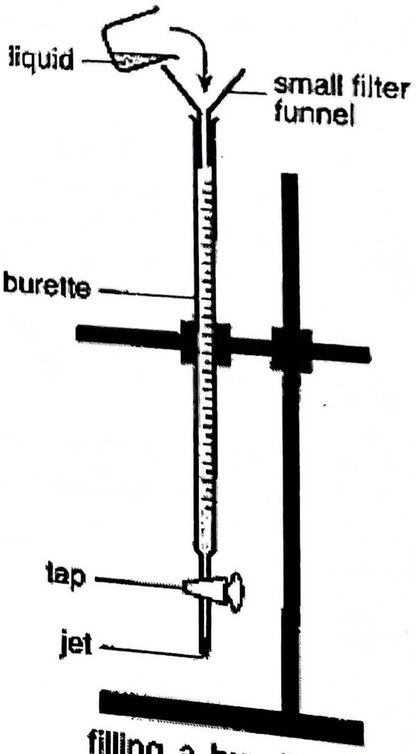
- Remove the pipette filler and allow the solution to run out into a clean conical flask. Rinse dirty conical flasks only with deionised water if needed.
- When almost all the solution has drained out of the pipette, tilt the conical flask, and **gently** tap the tip of the pipette against its base.

  For your close attention: Do not attempt to force out the last drop in the pipette as this is part of its calibration during manufacture to give the required volume. Doing so will result in more solution transferred than required.

- Clamp the pipette to the retort stand after use.

1.4.2 Use of Burette

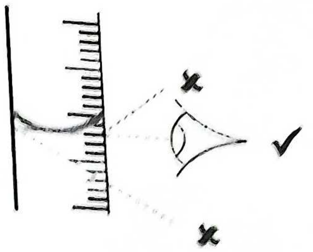
- Rinse burette with deionised water followed by the solution it is to be filled with.
- Ensure that the burette is clamped vertically.
- Place filter funnel and fill the burette with solution above the zero mark.
- Remove the filter funnel. Open the stopcock (tap) to fill the burette jet with solution and remove any trapped air bubbles in the burette.
- Adjust the meniscus level to zero or any other convenient mark.



filling a burette

- Take the burette reading at eye level with the bottom of the meniscus.

  If the solution has an intense colouration, e.g. potassium manganate(VII) which is dark purple, you may instead take the burette reading the top of the meniscus.



1.4.3 Performing Titration

Before Titration

- Ensure **filter funnel is removed** from the burette.
- Check that the **burette jet is filled** with the required solution with no air bubbles.
- Obtain the **initial burette reading** and record to 2 decimal places (d.p.).
- Ensure that the **appropriate indicator** (where required) is added into the conical flask. *Just one to two drops will suffice. Do not add excessively.*
- Place a **white tile** below the burette and place the conical flask on it.

During Titration

- Add the solution from the burette into the conical flask **with continuous swirling** to ensure even mixing.

  Note: If small quantities of solution from the burette remain as droplets on the sides of the conical flask, rinse these droplets down with deionised water.

- When nearing the end-point, **add the solution** from the burette **dropwise** into the conical flask. Swirl the flask right after adding each drop of solution.
- Stop titration when the **first drop** of solution from the burette causes a **permanent colour change** in the conical flask.

After Titration

- Obtain the **final burette reading** and record to 2 d.p..
- Calculate the volume of solution used (i.e. titre value) to 2 d.p..
- Repeat the titration process until you obtain two titre values which are consistent i.e. within ± 0.10 cm^3 of each other.
- Calculate the average titre value based the consistent results of two accurate titrations. If a rough titration was performed, the titre value obtained **cannot** be included in the average calculation.

Presenting Titration Results

Any data collected during volumetric analysis should be recorded to an appropriate level of precision.

- Pipette volumes should be recorded to 1 decimal place, such as 10.0 cm^3 and 25.0 cm^3.
- Burette readings should be recorded to 2 decimal places. The last decimal place should be a **0** or **5**.

Your titration results should be presented *clearly* and *neatly* by drawing a table if it is not given. Some sample titration results are shown below for your reference.

Example 1:

Remarks:
- Follow the order of items presented here.
- Adhere to the term 'burette reading'.
- Include **units**!
- **"FA 1"** is the titrant used here. *Adapt* your label according to the procedure given.

Remarks:
- Record burette readings to 2 decimal places.
- Last decimal place is either 0 or 5.
- Calculation:

  Volume of **FA 1** = Final burette reading – Initial burette reading
- Titration results are complete since the two titre values here are within ± 0.10 cm^3. Hence, a third titration is not required.
- Place ticks below to indicate the values to be used in average titre volume calculation.

	1	2	
Final burette reading / cm^3	15.40	15.30	
Initial burette reading / cm^3	0.00	0.00	
Volume of **FA1** / cm^3	15.40	15.30	

✓ ✓

Average volume of **FA 1** required $= \frac{(15.40+15.30)}{2} = 15.35\ cm^3$

Remarks:
- Working statement is expected.
- Note the term 'average volume' is used here.
- **"FA 1"** is the titrant used here. *Adapt* your label according to the procedure given.

Remarks:
- Working **must** be shown.
- Answer in **2 d.p.**.
- Include **units** (cm^3)!

Example 2:

Remarks:

- A 3rd titration was required as the first two titre volumes were **not** consistent i.e. within ± 0.10 cm^3 of each other.
- Only the last two titre volumes are selected since results are consistent i.e. within ± 0.10 cm^3 of each other.

	1	**2**	**3**
Final burette reading / cm^3	13.00	13.20	13.25
Initial burette reading / cm^3	0.00	0.00	0.00
Volume of **FA 3** / cm^3	13.00	13.20	13.25
		✓	✓

Average volume of **FA 3** required = $\frac{(13.20 + 13.25)}{2} = 13.23\ cm^3$

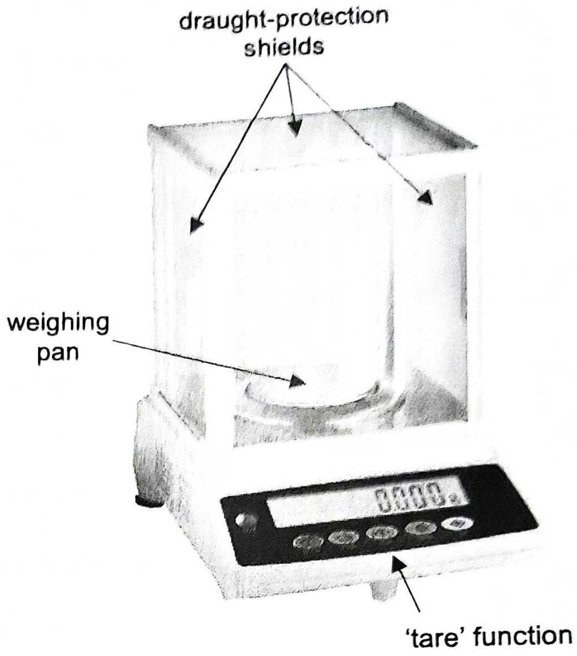
Remarks:

- The last decimal place for the average titre volume need not be 0 or 5.

1.4.4 Using an Electronic Balance

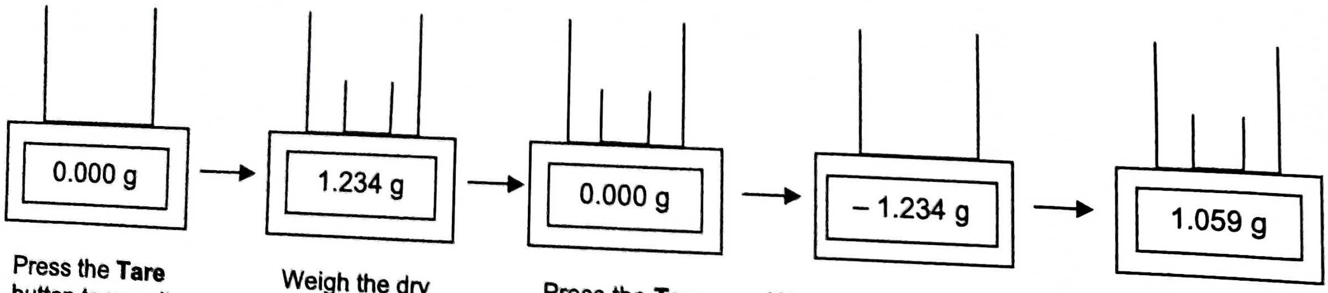
- The electronic balance in the laboratory is capable of reading to ±0.001 g. Hence, mass readings are to be **recorded to 3 decimal places**.
- A weighing bottle is used to contain the sample to be weighed. It should be placed on the centre of the weighing pan.
- The draught-protection shields will ensure that the reading is not affected by wind or movement.
- The 'tare' function resets the balance reading to zero and thus the tare facility allows the mass of the sample to be read directly i.e. excluding the mass of the weighing bottle.

  Note: The 'tare' function cannot be used in some cases, such as if we need to determine the mass of residue after transferring the solid or in a gravimetry experiment.



Weighing a Solid Sample into the Weighing Bottle

1. Ensure all the doors of the shield are fully closed. Press the 'tare' function to set the balance reading to zero.
2. Place a **dry and clean** empty weighing bottle on the centre of the weighing pan and close the door of the shield. Once the reading stabilises, record the mass of the empty weighing bottle.
3. With the empty weighing bottle on the weighing pan, press the 'tare' function to set the balance reading to zero.
4. Remove the weighing bottle from the weighing pan. Add the required mass of solid sample into the weighing bottle and put it back on the weighing pan. Close the door of the shield.
   (Do not add solid to the weighing bottle when it is on the weighing pan. This is to ensure that no solid will spill onto the pan.)
5. Once the reading stabilises, record the mass of the solid sample.
6. To obtain the mass of the solid sample and weighing bottle, add up the mass readings obtained in steps 2 and 5.



Press the **Tare** button to zero it.

Weigh the dry and clean empty bottle.

Press the **Tare** button to zero it.

Weighing balance will show negative sign when bottle is taken out.
(ignore this reading)

Place the solid in bottle and weigh.

Presenting Mass Readings

- Adapt your presentation of data according to the context of the practical task. Note the **difference in data presentation** between the two examples below.

Example 1:
This task requires students to weigh accurately between 4.50 g and 5.00 g of solid **FA 1** into the weighing bottle.

(In other words, students are provided with empty weighing bottles. They are required to weigh the solid sample on their own.)

	mass of **FA 1** + weighing bottle / g	10.418
note →	mass of empty weighing bottle / g	5.672
	mass of **FA 1** used / g	4.746

Example 2:
This task requires students to find out the mass of solid **FA 1** used in the experiment when a sample of **FA 1** is provided in a weighing bottle.

*(In other words, students are provided with weighing bottles that contain **FA 1**. After use, there will be residual **FA 1** that inevitably remains in the weighing bottle.)*

	mass of weighing bottle + **FA 1** / g	9.309
note →	mass of weighing bottle + residual **FA 1** / g	5.686
	mass of **FA 1** used / g	3.623

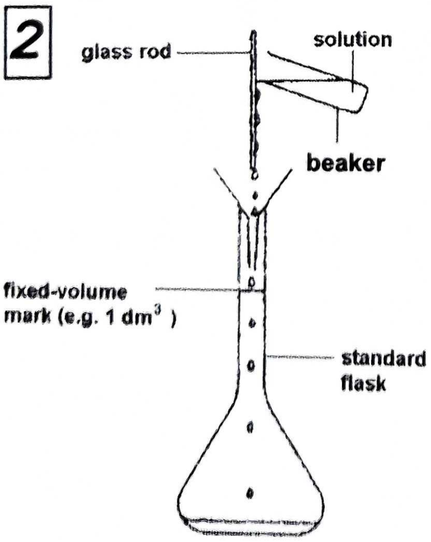
1.4.5 Using a Volumetric (or Standard) Flask

- The volumetric flask is a calibrated piece of glassware that can be used to prepare a fixed volume of solution. A typical volumetric flask in the laboratory has a capacity of 250 cm^3. It is often used for dilution and preparation of standard solutions from a solid.

Preparing Standard Solution from Solids

1. Transfer the weighed solid sample into a clean small beaker containing a little deionised water or other specified solvents.
2. Rinse out the remaining solid from the weighing bottle with some deionised water and transfer the washings into the small beaker. Rinse the weighing bottle a second time and transfer the washings into the small beaker.
3. Use a glass rod to stir the solution until all solids have completely dissolved. Add more deionised water if necessary.
4. Pour the concentrated solution into the volumetric flask via a filter funnel, using a glass rod to direct the solution into the flask.
5. Using deionised water, rinse the beaker, glass rod followed by the filter funnel and ensure that all the washings go into the volumetric flask. Always keep track of the solution level during rinsing. Rinse slowly and sparingly to ensure that the solution level does not exceed the graduation mark.
6. Remove the filter funnel from the volumetric flask. Continue to add deionised water until the level is about 1 cm below the graduation mark.
7. Pour some deionised water into a clean small beaker. Using a dropper (*pre-rinsed with deionised water*), add deionised water dropwise to bring the bottom of the meniscus to the mark.
8. Insert the stopper of the volumetric flask (*ensure it is tightly fitted*), invert and shake thoroughly (*~10 times*) to obtain a homogeneous solution.

2



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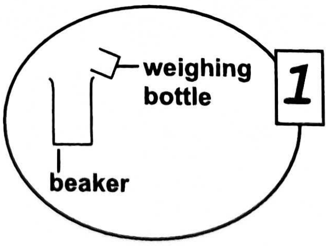
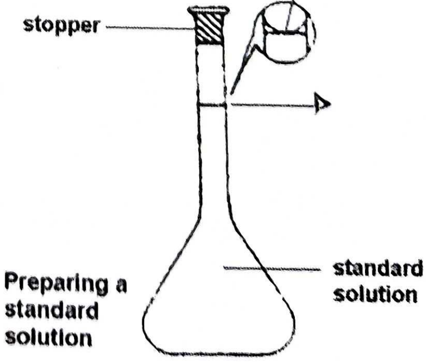


3

1-dm^3 mark



Diluting a Stock Solution

- The procedure to dilute a solution of known concentration is like that of solids described above, except that a fixed volume of solution is added into the volumetric flask *using a pipette or burette.*
- The table below shows how data for dilution should be presented for a sample solution, **FA 1**. Adapt your presentation of data according to the context of the practical task.

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

2. ERRORS IN EXPERIMENTAL CHEMISTRY

This section covers some basic knowledge about handling errors in chemical experiments. The content covered here will be applicable to other types of chemistry experiments not covered in this handbook. Sections 2.1 and 2.2 are not directly examinable but underpins the remaining sections.

2.1 Classes of Errors

- An error refers to the difference between a measured value and the true value of a physical quantity being measured. Experimental errors are inevitable, and the known errors are corrected where possible.

- In general, errors associated with a measurement can be classified as systematic errors and random errors.

systematic error	random error
• A constant error in all measured readings • Cannot be analysed statistically • Cannot be eliminated by averaging Examples: • Instrumental error – faulty calibrations, usage under inappropriate conditions • Method error – non-ideal chemical or physical behaviour, such as slowness of reaction, side reactions, instability of species	• Measured readings are scattered about the mean value with no fixed pattern • Can be analysed statistically • Can be reduced by averaging over many measurements or data

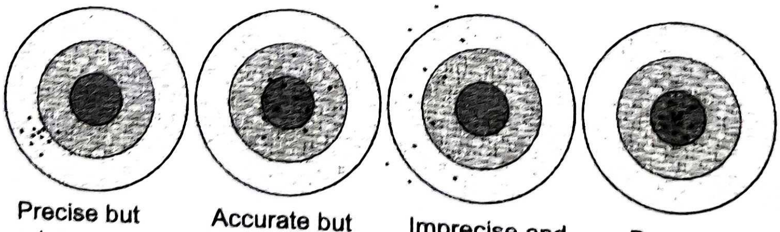
2.2 Accuracy vs. Precision

- The terms *accuracy* and *precision* need to be distinguished when discussing errors.

- Accuracy refers to the closeness of a measured or derived data value is to its 'true' value. The 'true' value of a measurement is the value that would be obtained by a perfect measurement, i.e. in an ideal world. Hence, the true value is not known. Good accuracy is associated with small systematic errors.

- Precision refers to the closeness of agreement among repeated measurements of a given sample. Good precision is associated with small random errors.

Target Analogy for Accuracy and Precision:



Precise but not accurate

Accurate but not precise

Imprecise and inaccurate

Precise and accurate

- For example, an uncalibrated electronic balance will give precise but inaccurate results. This means that the random errors associated with the mass measurement is small (little scattering among mass readings) but has a large systematic error (constantly deviates from the 'true' value).

2.3 Error Calculations

- Every measuring apparatus or equipment has an uncertainty and error associated with its use.

  (Uncertainty is the range of values on both sides of a measurement in which the 'true' value of the measurement is expected to lie.)

  Example:
  A 50 cm^3 measuring cylinder has an uncertainty of ±0.5 cm^3. When it is used to measure 10 cm^3 of water, it will give a volume ranging between 9.5 cm^3 and 10.5 cm^3.

- Percentage (%) error can be calculated using the general formula:

$$\boxed{\%\ \text{error} = \pm\frac{\text{uncertainty of apparatus} \times \text{no. of readings taken using apparatus}}{\text{measured quantity}} \times 100\%}$$

  Food for thought: How many readings on a burette are taken to measure 10 cm^3 of water?

- While the uncertainty associated with a calibrated apparatus is the same, the percentage error is larger when it is used to measure a smaller quantity.

  Example:
  A 50 cm^3 measuring cylinder to measure 10 cm^3 of water gives a larger percentage error than compared to measuring 40 cm^3 of water.

  Uncertainty of 50 cm^3 measuring cylinder = ±0.5 cm^3

for 10 cm^3 of water	**for 40 cm^3 of water**
$\%\ \text{error} = \frac{0.5}{10} \times 100\% = \pm 5\ \%$	$\%\ \text{error} = \frac{0.5}{40} \times 100\% = \pm 1.25\ \%$

  To obtain more accurate measurement for 10 cm^3 of water, an apparatus with a higher precision could be used, such as 10 cm^3 measuring cylinder or a 50 cm^3 burette. (The term *precision* here is used to describe the range of uncertainty in an instrument's scale.)

- The total apparatus error is the sum of the percentage errors associated with each piece of apparatus.

  Example:
  To prepare a solution from a solid sample, a 50 cm^3 burette was used to measure 20.00 cm^3 of water and an electronic balance precise to 2 decimal places was used to weigh 0.50 g of solid.

% error using 50 cm^3 burette	**% error using electronic balance**
$\%\ \text{error} = \frac{0.05 \times 2}{20.00} \times 100\% = \pm 0.50\ \%$	$\%\ \text{error} = \frac{0.01}{0.5} \times 100\% = \pm 2\ \%$

  Total apparatus error = ±(0.50 + 2) % = ±2.5 %

  The electronic balance has a larger contribution to the total apparatus error. An improvement to the process could be to use an electronic balance that is more precise i.e. to ±0.001 g.

2.4 Instrument Precision & Uncertainty

- The uncertainty in a single scale reading of an instrument is typically taken as half the smallest division. (Exceptions apply, such as digital meters.)

- The table below summarises the uncertainties for apparatus commonly used in H2 Chemistry.

apparatus	smallest division	uncertainty	remarks
electronic balance	0.001 g	±0.001 g	record to 3 d.p.
10 cm^3 pipette	-	±0.02 cm^3	record to 1 d.p.
25 cm^3 pipette	-	±0.03 cm^3	
50 cm^3 burette	0.1 cm^3	±0.05 cm^3	record to 2 d.p., last decimal either 0 or 5
10 cm^3 measuring cylinder	0.2 cm^3	±0.1 cm^3	record to 1 d.p.
25 cm^3 measuring cylinder	0.5 cm^3	±0.25 cm^3	record to 2 d.p.
50 cm^3 measuring cylinder	1 cm^3	±0.5 cm^3	record to 1 d.p. last decimal either 0 or 5
0.2°C thermometer	0.2 °C	±0.1 °C	record to 1 d.p.
1 °C thermometer	1 °C	±0.5 °C	record to 1 d.p., last decimal either 0 or 5
stopwatch	0.01s	1 s (human reaction time)	Format for recording depends on context. *Example*: 135s (nearest second) = 2 min and 15 s (in min and s) = 2.25 min (in decimal place)

2.5 Anomalous Results

- Anomalous results refer to data which are inconsistent or irregular. Anomalous results are likely to be present when readings do not fall within a certain range or that the distribution of measurements do not follow a certain trend.

- For the case of volumetric analysis, anomalous results occur when titre values are not consistent i.e. not within ±0.10 cm^3.

  Possible reasons:
  1. The titrant was not added dropwise near the end-point, making end-point determination difficult to ascertain.
  2. Solution in the conical flask was not swirled continuously to ensure complete mixing.
  3. The standard solution prepared was not mixed thoroughly to ensure a homogeneous mixture.

3. QUALITATIVE ANALYSIS (QA)

Prerequisite Knowledge from O Level Chemistry or equivalent (non-exhaustive):
- Colour and Solubility of Salts
- Identification of Cations, Anions and Test for Gases

3.1 Introduction

Qualitative Analysis (QA) is an experimental method used to identify unknown chemical samples. Chemical tests involving a set of chemical reagents are systematically and sequentially conducted on the unknown samples. By comparing the observations of each test, the possible identities of the unknown samples are deduced and narrowed down methodically to reach a conclusion.

This section of the handbook focuses primarily on the analysis of *inorganic* compounds. Nevertheless, the experimental techniques covered here are also applicable to analysis of organic compounds.

3.2 General Experimental Techniques in Qualitative Analysis

3.2.1 Quantity of Chemicals

The following quantities should be used unless the given procedure specifies otherwise.

For Solids
- Sufficient to fill the hemisphere at the bottom of a **clean and dry** test-tube.

For Solutions
- Sufficient to fill one-fifth the length (~1 cm depth, unless otherwise specified) of a **clean** test-tube.
- In cases where test on a gas evolved is required, a larger volume of solution may be required for significant observation.

Note: Do not waste chemicals, litmus papers, aluminium foil, filter papers, etc. Use sufficient quantities but not in excess.

3.2.2 Handling Reagents

Safety:
- ❖ *Do not touch chemicals with your bare hands. In case of skin contact with chemicals, wash off immediately under running water.*
- ❖ *Concentrated acids or alkalis should be diluted before disposal. Flush the sink with plenty of water after disposal. Follow the instructions given to you for disposal of these solutions.*
- ❖ *Use a test-tube holder when handling concentrated acids, alkalis and flammable reagents. Carry out the experiments in a fume cupboard.*

Handling Solids

- Transfer solid from stock bottle into a test-tube using a **clean and dry** spatula.

- If more than one solid sample is involved, use one spatula only for one type of solid. Do not cross-contaminate the stock bottles!

- Dispose solids into the waste bin. Do not throw into the sink.

Handling Solutions

- To avoid contamination of the given reagent bottles, do not allow the tip of the bottle to touch the sides of the test-tubes! The tip of bottle should be held just above the mouth of the test-tube.

- When adding reagents using a dropper (or teat pipette), the dropper should be held just above the mouth of the test-tube. The tip of dropper should not touch the sides of the test-tube.

  Use one dropper for only one type of reagent to avoid contamination. Do not leave the dropper on the bench or allow the tip to touch anything else except the intended reagent.

adding reagent from bottle:

adding reagent from dropper (teat pipette):

How to use a dropper (teat pipette) correctly?
Squeeze the bulb of the dropper **BEFORE** immersing the tip into the solution. Release to draw up the solution into the dropper. Squeeze the bulb again to dispense the solution.

- Solutions should always be added dropwise. Shake the test-tube to mix the chemicals and observe the mixture. Repeat the process until no further change is observed.

Mixing Solids and Solutions

- For small volumes of mixture in a test-tube
  - Hold the test-tube near its mouth with one hand.
  - Tap the bottom of the test-tube against your palm OR use your index finger to tap near the bottom of the test-tube.

- For large volumes of mixture in a test-tube
  - Stir the contents gently using a clean glass rod.

3.2.3 Heating and Warming

Safety:

❖ *Always use a **test-tube holder** when heating or warming chemicals.*

❖ *Direct the mouth of the test-tube **away from you and others** during heating or warming.*

❖ *Operate a Bunsen burner correctly and safely. If unsure, clarify with your tutor. Turn down the flame to luminous (orange flame) when not in use. Turn it off when no longer required.*

❖ *If needed, note the smell of a gas by wafting it gently to the nose. Do not inhale deep breaths.*

- Use the correct apparatus (test-tube or boiling tube) for heating based on the procedure given.
- Ensure that the glassware is clean and dry.
- During warming or heating, the test-tube or boiling tube should be held at angle of about 45° or lower using a test-tube holder. Point the mouth of the test-tube away from you and others.

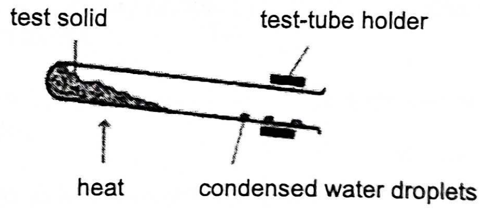
Heating Solids

- Heat **gently initially** to drive off water vapour then heat **strongly subsequently** to ensure complete decomposition of the solid or until no further changes take place.

  For gentle heating, ensure the airhole of the Bunsen burner is half-open. Fully open the airhole for strong heating. Adjust the size of the airhole where necessary.

- If a solid gives off water when heated, hold the test tube **slightly sloping downwards**. This prevents the condensed water at the mouth of the test tube from running back to the hot part of the test tube and cracking the glass.

heating a solid that gives off water:



- Most solids undergo changes when heated. These changes often provide a clue to identity of the cation or anion present in the solid.
  - Consider testing for gases that could possibly be liberated during heating.
  - Note the colour of the residue when it is hot and when it is cold.

Some examples are shown in Tables 1 and 2 for your reference (good to know).

Table 1: Examples of Gas Evolved on Heating & Deduction

observation	deduction	
Gas rekindles a glowing splint.	O_2 evolved	PbO_2 or oxy–salts like NO_3^- and MnO_4^-
Gas produces a white ppt with calcium hydroxide.	CO_2 evolved	HCO_3^-, CO_3^{2-}; $C_2O_4^{2-}$ and organic compounds
Gas turns moist red litmus paper blue.	NH_3 evolved	Ammonium salts (NH_4^+)
Gas turns purple acidified $KMnO_4$ solution colourless.	SO_2 evolved	SO_3^{2-}; $S_2O_3^{2-}$ and certain SO_4^{2-} e.g. $FeSO_4$

Table 2: Examples of Coloured Residue & Deduction

observation	deduction
Residue appears reddish–brown when hot. Residue appears brown when cold.	Fe compounds present. Fe_2O_3 formed.
Black residue obtained after heating blue or green solids.	Cu or Ni compounds present. CuO or NiO formed.
Black residue obtained after heating pink solids.	Mn compounds present. Oxidation of Mn^{2+} to higher oxidation states.
Residue appears yellow when hot. Residue appears white when cold.	Zn compounds present.

Heating Solutions

- Solutions should be warmed or heated under gentle heating.

- When a test-tube containing a liquid, heat just below the level of the meniscus while agitating the liquid at the same to ensure even heating.

- Do not hold the test-tube at a fixed position or concentrate heating at the bottom of the test-tube as it may cause the hot contents to spurt out of the test-tube unexpectedly.

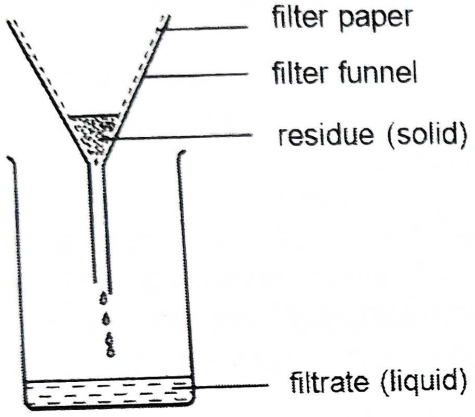
- If the volume of solution in the test-tube is too large (more than half of the test-tube), transfer a portion of the solution into a clean test-tube and carry out heating.

3.2.4 Filtration

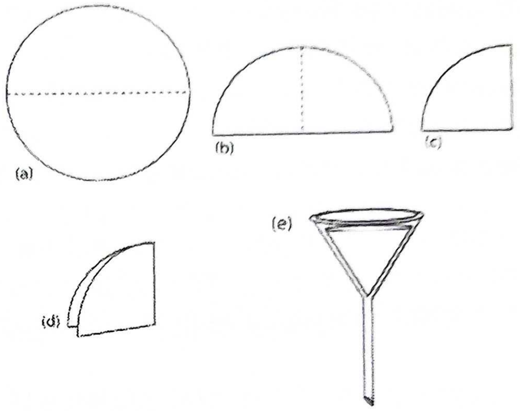
- Gravity filtration is used to separate a mixture consisting of liquids and insoluble solids.

  The mixture is passed through a filter funnel that is lined with filter paper. The filter paper is semi-permeable i.e. only particles whose sizes fit into the pores of the paper can pass through it. Hence, the larger solid particles are trapped in the filter paper as residue. The smaller liquid particles are collected as the filtrate.

A Filtration Set-up:



Steps to fold a filter paper (a) to (e):



- Before pouring the mixture into the filter funnel, wet the filter paper with some deionised water. This is typically done to reduce the loss of liquid mixture as some of it will be absorbed by the filter paper.

- When almost all of liquid has been filtered, wash the residue with some deionised water to remove any trace amounts of filtrate from the residue.

  For organic experiments which require dry conditions i.e. no water present, the residue obtained is washed with an appropriate solvent.

- A fluted filter paper may quicken the process of filtration as it allows for faster equilibration of air pressure through the folded sides.

  Scan the QR code to watch how to fold a fluted filter paper.

3.3 Tests in Qualitative Analysis

The following list of chemical reagents (or bench reagents) are typically used for qualitative analysis:

- aqueous sodium hydroxide, $NaOH(aq)$
- aqueous ammonia, $NH_3(aq)$
- hydrochloric acid, $HCl(aq)$
- nitric acid, $HNO_3(aq)$
- sulfuric acid, $H_2SO_4(aq)$
- aqueous silver nitrate, $AgNO_3(aq)$
- aqueous barium nitrate, $Ba(NO_3)_2(aq)$
- limewater (a saturated solution of calcium hydroxide, $Ca(OH)_2(aq)$)
- aqueous potassium manganate(VII), $KMnO_4(aq)$
- aqueous potassium iodide, $KI(aq)$
- aluminium foil, $Al(s)$
- red and blue litmus paper or Universal Indicator (UI) paper

It is imperative that you understand the purpose behind the use of each chemical reagent listed above. In other words, what kind of information can you gain when conducting chemical tests using each reagent? What is the type of reaction expected when using each reagent?

Points to Note for Practical Assessment

- Candidates should be familiar with Qualitative inorganic analysis involving an element, a compound or a mixture. Systematic analysis will **not** be required.

- Candidates should be familiar with the reactions of cations, reactions of anions and test for gases as detailed in the Qualitative Analysis Notes.

- Candidates would **not** be required to carry out tests involving hexane, sulfur dioxide gas, nitrite ions or sulfite ions.

- Reactions involving ions not included in the Qualitative Analysis Notes may be tested: in such cases, candidates will **not** be expected to identify the ions but only to draw conclusions of a general nature.

- Candidates should **not** attempt tests, other than those specified, on substances, except when it is appropriate to test for a gas.

3.3.1 Test for Cations

The following aqueous cations are listed in the QA notes used for practical examinations:

Al^{3+} NH_4^+ Ba^{2+} Ca^{2+} Cr^{3+} Mn^{2+} Fe^{2+} Fe^{3+} Cu^{2+} Mg^{2+} Zn^{2+}

- NaOH(aq) and NH_3(aq) are typically used to test for cations in QA. They produce OH^- ions which react with the metal cations to form metal hydroxides (observed as precipitates). In excess volumes of NaOH(aq) and NH_3(aq), some of these metal hydroxide precipitates remain insoluble while others may dissolve to form soluble complex ions.

NaOH is a strong base:	$NaOH(aq) \rightarrow Na^+(aq) + OH^-(aq)$
NH_3(aq) is a weak base:	$NH_3(aq) + H_2O(l) \rightleftharpoons NH_4^+(aq) + OH^-(aq)$

- Testing for cations involves performing the following steps and making observations in each step.

step	what to look out for?
1. To 1 cm depth of sample in a test-tube, add NaOH(aq) (or NH_3(aq)) **dropwise with shaking**.	• Colour of precipitate, if any. [For NaOH(aq) test only]: If no precipitate seen, warm solution and test for NH_3(g).
2. Add NaOH(aq) (or NH_3(aq) **in excess or until no further change**.	• Solubility of precipitate – Is it soluble in excess to give a solution? If yes, what is the colour of the solution?

- The colours of the transition metal cations are listed below. The rest of the cations are colourless.

cation	colour	remarks
Cu^{2+}	blue	-
Fe^{2+}	pale green	-
Fe^{3+}	pale yellow	orange-brown at higher concentrations
Cr^{3+}	green	-
Mn^{2+}	colourless	pale pink at higher concentrations

Practical Tip: Knowing the colours of these cations will provide you some preliminary clues about the identity of the unknown sample if it is coloured.

- Al^{3+}, Cr^{3+}, Fe^{3+} and NH_4^+ are acidic cations.

cation	nature of cation	explanation
Al^{3+} Cr^{3+} Fe^{3+}	cations of high charge density (triply charged and small cationic size)	The cations undergo **hydrolysis** with water to release H_3O^+, resulting in an acidic solution. $[M(H_2O)_6]^{3+} + H_2O \rightleftharpoons [M(H_2O)_5(OH)]^{2+} + H_3O^+$
NH_4^+	conjugate acid of $NH_3(aq)$	NH_4^+ undergoes **hydrolysis** with water to release H_3O^+, resulting in an acidic solution. $NH_4^+ + H_2O \rightleftharpoons NH_3 + H_3O^+$

Given the acidic property of these cations, **adding aqueous sodium carbonate, $Na_2CO_3(aq)$, is another useful cation test in QA** that allows these ions to be distinguished from the rest.

Example:
Addition of Na_2CO_3 to solution containing Al^{3+} results in an **acid-base reaction** with effervescence of CO_2 gas and formation of a white precipitate, $Al(OH)_3(s)$.

$$[Al(H_2O)_6]^{3+}(aq) + H_2O(l) \rightleftharpoons [Al(H_2O)_5(OH)]^{2+}(aq) + H_3O^+(aq) \quad \text{---- (1)}$$
$$CO_3^{2-}(aq) + 2H_3O^+(aq) \rightarrow CO_2(g) + 3H_2O(l)$$

In case you are wondering: Why is the precipitate $Al(OH)_3$ and not $Al_2(CO_3)_3$?

By Le Chatelier's Principle, the removal of H_3O^+ by CO_3^{2-} causes the position of equilibrium (1) to shift to the right, producing more H_3O^+ and $[Al(H_2O)_5(OH)]^{2+}$. Continual removal of H_3O^+ results in positions of equilibrium (2) and (3) to shift to the right, eventually forming $Al(H_2O)_3(OH)_3$, which is equivalent to $Al(OH)_3$.

$$[Al(H_2O)_5(OH)]^{2+}(aq) + H_2O(l) \rightleftharpoons [Al(H_2O)_4(OH)_2]^{+}(aq) + H_3O^+(aq) \quad \text{---- (2)}$$
$$[Al(H_2O)_4(OH)_2]^{+}(aq) + H_2O(l) \rightleftharpoons [Al(H_2O)_3(OH)_3](s) + H_3O^+(aq) \quad \text{---- (3)}$$

Note: The above explanation can also be extended to Cr^{3+} and Fe^{3+}. For all other metal cations listed in the QA note, metal carbonates are formed as the precipitate.

- Al^{3+}, Cr^{3+} and Zn^{2+} ions form **amphoteric metal hydroxides** with OH^-. The metal hydroxides can dissolve further in excess NaOH to form soluble complex ions. They can also undergo acid-base reactions with acids.

cation	metal hydroxide	metal complex ion	equations (formation of metal hydroxide & complex ion)
Al^{3+}	$Al(OH)_3$	$[Al(OH)_4]^-$	$Al^{3+}(aq) + 3OH^-(aq) \rightleftharpoons Al(OH)_3(s)$ $Al(OH)_3(s) + OH^-(aq) \rightleftharpoons [Al(OH)_4]^-(aq)$
Cr^{3+}	$Cr(OH)_3$	$[Cr(OH)_6]^{3-}$	$Cr^{3+}(aq) + 3OH^-(aq) \rightleftharpoons Cr(OH)_3(s)$ $Cr(OH)_3(s) + 3OH^-(aq) \rightleftharpoons [Cr(OH)_6]^{3-}(aq)$
Zn^{2+}	$Zn(OH)_2$	$[Zn(OH)_4]^{2-}$	$Zn^{2+}(aq) + 2OH^-(aq) \rightleftharpoons Zn(OH)_2(s)$ $Zn(OH)_2(s) + 2OH^-(aq) \rightleftharpoons [Zn(OH)_4]^{2-}(aq)$

- Cu^{2+} and Zn^{2+} form metal hydroxides which are soluble in excess NH_3(aq). Under excess conditions, NH_3 functions as a ligand which can bond to these cations. Ligand exchange reaction occurs where NH_3 displaces H_2O, forming soluble Cu^{2+} and Zn^{2+} complex ions. Details about ligands and ligand exchange reactions will be covered under *Chemistry of Transition Elements* in Y6.

cation	Cu^{2+} (or $[Cu(H_2O)_6]^{2+}$)
metal hydroxide	$Cu(OH)_2$
metal complex ion	$[Cu(NH_3)_4(H_2O)_2]^{2+}$
equations	$[Cu(H_2O)_6]^{2+}(aq) + 2OH^-(aq) \rightleftharpoons Cu(OH)_2(s) + 6H_2O(l)$ $[Cu(H_2O)_6]^{2+}(aq) + 4NH_3(aq) \rightleftharpoons [Cu(NH_3)_4(H_2O)_2]^{2+}(aq) + 4H_2O(l)$

cation	Zn^{2+} (or $[Zn(H_2O)_6]^{2+}$)
metal hydroxide	$Zn(OH)_2$
metal complex ion	$[Zn(NH_3)_4]^{2+}$
equations	$[Zn(H_2O)_6]^{2+}(aq) + 2OH^-(aq) \rightleftharpoons Zn(OH)_2(s) + 6H_2O(l)$ $[Zn(H_2O)_6]^{2+}(aq) + 4NH_3(aq) \rightleftharpoons [Zn(NH_3)_4(H_2O)_2]^{2+}(aq) + 4H_2O(l)$

- Potassium iodide, KI(aq), can be used to test for Cu^{2+} and Fe^{3+} ions. It acts as a reducing agent.

cation	**product**	**observation**	**equation**
Cu^{2+}	CuI and I_2	white precipitate in brown solution formed	$2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$
Fe^{3+}	Fe^{2+} and I_2	brown solution formed	$2Fe^{3+}(aq) + 2I^-(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$

3.3.2 Precipitate Reformation from Soluble Complex Ions [To be covered in Y6]

Precipitate reformation is a test in QA that is typically performed on colourless filtrates after filtering a mixture. The reformation of a precipitate in the filtrate enables one to ascertain whether specific cations, such as Al^{3+}, Zn^{2+} and Ba^{2+}, are present or absent.

Dilute acid is usually used in precipitate reformation from soluble complex ions such as $[Al(OH)_4]^-$, $[Zn(OH)_4]^{2-}$, $[Cr(OH)_6]^{3-}$ and $[Cu(NH_3)_4(H_2O)_2]^{2+}$.

<u>Experimental Technique for Precipitate Reformation</u>

Example: Reforming $Zn(OH)_2$ from $[Zn(OH)_4]^{2-}$

Given procedure:
To 2 cm depth of the filtrate in a test-tube, add dilute nitric acid dropwise until no further change.

steps	explanation
Place test-tube containing filtrate on test-tube rack. Add 2 to 3 drops of nitric acid. Do not shake the test-tube. Observe for precipitate reformation. (Observation: Some white precipitate reforms)	This avoids the test-tube from shaking and allows observation of any precipitate reformed. Adding nitric acid provides H^+ which removes OH^-. By Le Chatelier's Principle, position of equilibrium shifts left, reforming $Zn(OH)_2(s)$. $Zn(OH)_2(s) + 2OH^-(aq) \rightleftharpoons [Zn(OH)_4]^{2-}(aq)$ Shaking the test-tube initially will prevent clear observation of the reformed precipitate since there is excess OH^- present which will dissolve the reformed precipitate.
Continue to add more nitric acid to the filtrate until no further change. Observe the solution followed by shaking the test-tube. (Observation: More white precipitate reforms. After shaking, the white precipitate dissolves in excess acid to give a colourless solution)	As more nitric acid is added, excess OH^- present will be removed (neutralisation occurs). More $Zn(OH)_2(s)$ will be reformed. In excess volumes of nitric acid, $Zn(OH)_2(s)$, being amphoteric, undergoes **acid-base reaction** to form $Zn(NO_3)_2(aq)$, which is colourless. $Zn(OH)_2(s) + 2HNO_3(aq) \rightarrow Zn(NO_3)_2(aq) + 2H_2O(l)$

3.3.3 Test for Anions

The following aqueous anions are listed in the QA notes used for practical examinations:

CO_3^{2-} Cl^- Br^- I^- NO_3^- NO_2^- SO_4^{2-} SO_3^{2-}

- Dilute acids, HCl(aq), H_2SO_4(aq) and HNO_3(aq) are typically used to test for CO_3^{2-}(aq), NO_2^-(aq) and SO_3^{2-}(aq).

- You will **not** be required to test for NO_2^- (nitrite) and SO_3^{2-} (sulfite) ions during practical examinations.

What to look out for: **effervescence (of CO_2)**

anion	**equation**
CO_3^{2-}	$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l)$
NO_2^-	$2NO_2^-(aq) + 2H^+(aq) \rightarrow NO_2(g) + NO(g) + H_2O(l)$
SO_3^{2-}	$SO_3^{2-}(aq) + 2H^+(aq) \rightarrow SO_2(g) + H_2O(l)$

- Aqueous silver nitrate, $AgNO_3$(aq), together with NH_3(aq), is typically used to test for the halides, Cl^-, Br^- and I^-.

What to look out for: **precipitate and solubility in NH_3(aq)**

anion	**precipitate**	**colour**	**equation**	**solubility in NH_3(aq)**
Cl^-	$AgCl$	white	$Ag^+(aq) + Cl^-(aq) \rightleftharpoons AgCl(s)$	soluble
Br^-	$AgBr$	pale cream	$Ag^+(aq) + Br^-(aq) \rightleftharpoons AgBr(s)$	partially soluble
I^-	AgI	yellow	$Ag^+(aq) + I^-(aq) \rightleftharpoons AgI(s)$	insoluble

$AgCl$ and $AgBr$ can be distinguished more easily by adding NH_3(aq) as the colour of these two precipitates may be difficult to differentiate during practical.

NH_3(aq) forms a soluble complex ion with Ag^+.

$$Ag^+(aq) + 2NH_3(aq) \rightleftharpoons [Ag(NH_3)_2]^+(aq)$$

Note: The explanation for the different solubilities of the precipitates in NH_3(aq) can be found in *Solubility Equilibria* lecture notes (Y6).

- Aqueous barium nitrate, $Ba(NO_3)_2$(aq), is typically used to test for SO_4^{2-} and SO_3^{2-}.

What to look out for: **white precipitate**

anion	**precipitate**	**equation**
SO_4^{2-}	$BaSO_4$	$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightleftharpoons BaSO_4(s)$
SO_3^{2-}	$BaSO_3$	$Ba^{2+}(aq) + SO_3^{2-}(aq) \rightleftharpoons BaSO_3(s)$

$BaSO_3$ and $BaSO_4$ can be distinguished by adding dilute strong acids. No reaction occurs for $BaSO_4$ while $BaSO_3$ undergoes acid-base reaction.

$$BaSO_3(s) + 2H^+(aq) \rightarrow Ba^{2+}(aq) + SO_2(g) + H_2O(l)$$

- Aluminium foil (Al) and NaOH(aq) are used to test for NO_3^- and NO_2^-. Warming the mixture is required. NH_3(g) is evolved, and its presence is confirmed by using moist red litmus paper which will turn blue.

  Two reactions occur during the test:
  1. Reduction of NO_3^- and NO_2^- by Al to NH_4^+(aq)
  2. Acid-base reaction between NH_4^+(aq) and OH^-(aq) to form NH_3(g)

  Overall equation:

$$3NO_3^-(aq) + 8Al(s) + 5OH^-(aq) + 18H_2O(l) \rightarrow 3NH_3(g) + 8[Al(OH)_4]^-(aq)$$

$$NO_2^-(aq) + 2Al(s) + OH^-(aq) + 5H_2O(l) \rightarrow NH_3(g) + 2[Al(OH)_4]^-(aq)$$

Practical Tip:
While effervescence is observed during heating, do **not** record your observation as 'effervescence seen'. The effervescence observed is H_2(g) that is produced from the reaction between Al(s) and NaOH(aq).

$$2Al(s) + 2OH^-(aq) + 6H_2O(l) \rightarrow 2[Al(OH)_4]^-(aq) + 3H_2(g)$$

The gas to be tested here is NH_3(g) which is soluble in water. Hence, warming the mixture will reduce the solubility of NH_3(g), causing it to be evolved. The expected observation is 'Gas **evolved** turns moist red litmus paper blue. Gas is ammonia'.

- Acidified aqueous potassium manganate(VII), $KMnO_4$(aq), can oxidise with the following anions listed in the QA notes: NO_2^-, SO_3^{2-}, Cl^-, Br^- and I^-.

  Note that in the laboratory, the $KMnO_4$ solution provided in the set of bench reagents is **not** acidified. An equal depth of dilute sulfuric acid must be added to acidify the solution.

3.3.4 Test for Gases

The following gases can be found in the QA notes used for practical examinations:

NH_3 CO_2 Cl_2 H_2 O_2 SO_2

- You will **not** be required to test for SO_2 gas during practical examinations.
- Testing for each gas in QA typically requires multiple steps with the use of a few reagents and apparatus. Conducting tests for gas blindly can be inefficient, potentially frustrating, and misleading when performing QA.

  Hence, it is important to understand the given procedure and **infer possible types of reaction occurring from the given reagents** before deciding the appropriate test for gas. If more than one possible test is considered, it may help to conduct the simpler test first.

  Examples (non-exhaustive):

reagents and procedure	purpose of test	gas to be tested
$NaOH(aq)$ and warm	acid-base reaction, test for NH_4^+	$NH_3(g)$
$Al(s)$ and $NaOH(aq)$, heat	test for NO_2^- and NO_3^-	$NH_3(g)$
dilute acid (may include warming)	acid-base reaction, test for CO_3^{2-}, NO_2^- and SO_3^{2-}	$CO_2(g)$
solid and heat	thermal decomposition of ammonium, nitrate or carbonate salt	1. $NH_3(g)$ 2. $CO_2(g)$ 3. $O_2(g)$ (with brown NO_2)
solid (dull-looking) and acid	acid-metal (redox) reaction	$H_2(g)$
$H_2O_2(aq)$	redox reaction / oxidation	$O_2(g)$
HCl and oxidising agent	redox reaction	$Cl_2(g)$

Some Remarks (good to know)

- NH_3 is the only alkaline gas in the list. CO_2, Cl_2 and SO_2 are acidic while O_2 and H_2 are neutral.
- $CO_2(g)$ forms a white precipitate of $CaCO_3$ when bubbled into limewater. If excess CO_2 is bubbled into limewater, $CaCO_3$ will dissolve to form calcium hydrogencarbonate, $Ca(HCO_3)_2$.

$$CO_2(g) + Ca(OH)_2(aq) \rightarrow CaCO_3(s) + H_2O(l)$$

$$CO_2(g) + CaCO_3(s) + H_2O(l) \rightarrow Ca(HCO_3)_2(aq)$$

- $H_2(g)$ burns with oxygen in air explosively with a lighted splint, resulting in a 'pop' sound.

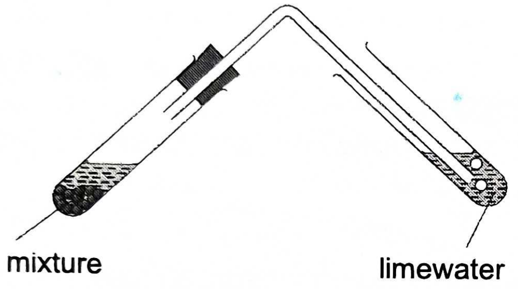
$$2H_2(g) + O_2(g) \rightarrow 2H_2O(l)$$

- $O_2(g)$ supports combustion and hence can relight a glowing splint.
- SO_2 is a reducing agent. It undergoes redox reaction with acidified $KMnO_4(aq)$.

$$2MnO_4^-(aq) + 5SO_2(g) + 2H_2O(l) \rightarrow 2Mn^{2+}(aq) + 5SO_4^{2-}(aq) + 4H^+(aq)$$

Experimental Techniques

- If a gas is expected to be produced, **prepare** the necessary reagents and apparatus **BEFORE** starting the test!

gas	technique	observation
Ammonia, NH_3	• Moisten red paper with deionised water. • Place paper at the mouth of the test-tube without touching the sides	**Gas** evolved turned **moist red litmus paper blue**. Gas is **NH_3**.
Carbon dioxide, CO_2	• Deliver gas into the limewater (about 1 cm depth in a test−tube) as shown.  • Alternatively, use the dropper method to suck gas produced into a test−tube with 1 cm depth of limewater.	**Effervescence seen**. **Gas** formed **white ppt with limewater**. Gas is **CO_2**.
Chlorine, Cl_2	• Moisten blue paper with deionised water. • Place paper at the mouth of the test-tube without touching the sides	**Effervescence seen.** **Gas** turned **damp blue litmus paper red** then **bleached** it. Gas is **Cl_2**.
Hydrogen, H_2	• Place a **lighted** splint (burning with visible flame) at the mouth of the test-tube.	**Effervescence seen.** **Gas "pops"** with a **lighted splint**. Gas is **H_2**.
Oxygen, O_2	• Light a wooden splint and put out the flame. Observe for a glow at the tip of the splint. • Insert the **glowing** splint into the test-tube.	**Effervescence seen.** **Gas relights a glowing splint**. Gas is **O_2**.
Sulfur dioxide, SO_2	• Fill a test-tube with 1 to 1.5 cm depth of containing **equal** volumes of aqueous potassium manganate(VII), $KMnO_4$(aq), and dilute sulfuric acid • Immerse the long end of the delivery tube into acidified $KMnO_4$(aq). • Connect the rubber stopper to the mouth of the test-tube producing the gas. Deliver gas into acidified $KMnO_4$(aq).	**Gas** evolved turned acidified $KMnO_4$(aq) **from purple to colourless**. Gas is **SO_2**.

3.4 Performing QA Tests, Recording Observations and Making Deductions

Before performing any tests during QA, it is important to **first understand the intent of the test** based on the given procedure. In other words, **analyse the chemical reagent added**. What does the specified reagent test for? What should one be looking out when performing the test?

Knowing the intent of test will provide guidance as to what observations should be recorded, which will in turn enable one to make the correct deductions in QA.

A list of reagents commonly used in Qualitative Analysis is summarised in *Section 3.5*.

3.4.1 Recording Observations for Different Tests in QA

- As covered in Section 3.3, tests in QA generally include those for cations, anions and gases.
- Record observations **in ink immediately after each test**, **alongside the given procedures** to indicate clearly at which stage a change occurs during the tests.
- Chemical equations are not required for observations.
- In general, observations in QA include details such as:
    - Colour of precipitate (abbreviated as 'ppt') and its solubility in excess reagent
    - Colour change of solution and/or solid (include ppt)
    - Test for gas (exact details depend on the specific gas tested)
    - Colour of residue and filtrate (if filtration is performed)
- For negative results i.e. tests with no observable changes, record observation as the negative statement of the expected observations.

  Example: No ppt formed.
  No NH_3 gas evolved.

  Note: You are to write "**no observable change**" instead of the negative result if the question requires you to do so.

Formation of Precipitate and its Solubility in Excess Reagent

scenario 1: **ppt formed is *insoluble* in excess reagent**	**scenario 2:** **ppt formed is *soluble* in excess reagent**
What to record: ✓ <state colour> ppt formed, ✓ Insoluble in excess <state reagent>.	*What to record:* ✓ <state colour> ppt formed, ✓ soluble in excess <state reagent> to give a <state colour> solution.
Example: Green ppt formed, turning brown on contact with air. Green ppt is insoluble in excess aqueous NaOH.	*Example*: Blue ppt formed, soluble in excess $NH_3(aq)$ to give a dark blue solution.

Colour Change of Solution and/or Precipitate

What to record: <state initial colour> solution/ppt turned <state final colour>.

Examples: Blue solid turned black.
Blue solution turned yellow-green.

Test for Gas

What to record:

✓ Effervescence observed. (applicable to gases **except** for NH_3 and SO_2)
✓ Chemical test and test result for the gas (see QA notes)
✓ Gas is <state name/formula of gas>.

See *Section 3.3.4 Test for Gases – Experimental Techniques* for observations specific to each gas.

3.4.2 How to Approach QA: A Worked Example

The worked example below illustrates some guiding points for beginners when performing QA. Note that it is non-exhaustive. Expect that the instructions given in QA questions will differ – always read them carefully before starting to attempt the question.

FA 1 is a solid containing two cations and two anions. One anion is known to be Cl^-.

Carry out the following tests. Carefully record your observations in Table 1.

Practical Tip:
- Read and understand each test in terms of *what the reagents test for* and *what to look out for* before performing each test.

Remarks:
- Observations recorded must be **aligned alongside** the instructions given in the tests.

Table 1

	tests	observations
1	Add a spatula load of **FA 1** into a clean boiling tube. Add 3 cm depth of aqueous nitric acid. Warm the contents in the boiling tube to dissolve **FA 1**. Allow the solution to cool. The solution obtained is **FA 2**. *Remarks*: • Add nitric acid ⇒ test for CO_3^{2-}, NO_2^- and SO_3^{2-}. Look for effervescence. • Prepare apparatus and chemical required for CO_2 test **before adding nitric acid**: test-tube containing limewater, delivery tube with rubber bung.	*Effervescence seen.* *Gas formed white ppt with limewater. Gas is CO_2.* *Blue solution is obtained.*
2	Add 2 cm depth of **FA 2** into a clean test-tube. Add aqueous sodium hydroxide slowly, with shaking, until 4 cm depth of aqueous sodium hydroxide has been added. Filter the mixture into a clean boiling tube. *Remarks*: • Add NaOH(aq) ⇒ test for cations. Look for colour of ppt & its solubility in excess. • "slowly, with shaking," ⇒ **always add dropwise first, <u>observe</u>, then shake and observe then add in excess.**	*Pale blue ppt formed, insoluble in excess NaOH(aq).* *Residue is pale blue.* *Filtrate is colourless.* *Remarks*: • Colour of residue and filtrate must be identified for filtration process.

	tests	observations
3	Add 2 cm depth of the filtrate into a clean test-tube. Add dilute sulfuric acid slowly, with shaking, until no further change is seen. *Remarks*: • Add sulfuric acid to filtrate ⇒ reformation of ppt. Look for ppt and its solubility in excess acid. • Note that addition of sulfuric acid (H_2SO_4) can also ascertain whether Ba^{2+} is present since $BaSO_4$ is a white ppt. • "until no further change is seen" means add reagent until in *excess*. • If procedure is worded as "add an equal depth of dilute sulfuric acid", you should **add dropwise first, <u>observe</u>, shake & observe then add in excess**.	*No ppt is seen.* *Remarks*: • Write "no observable change" if question requires you to do so for negative test results.
4	Add 2 cm depth of the filtrate into a clean boiling tube. Warm gently. *Remarks*: • Filtrate contains NaOH(aq) that was added in test 2. Warming gently with NaOH(aq) ⇒ test for NH_4^+ since all NH_4^+ salts are soluble. Look for NH_3(g). • Prepare apparatus and chemicals for NH_3(g) test: moist red litmus paper.	*Gas evolved turned moist red litmus paper blue. Gas is ammonia.*
5	Transfer a spatula load of the residue in test 2 to a clean test-tube and heat strongly. *Remarks*: • Strong heating ⇒ thermal decomposition. • Look for colour change in solid.	*Blue residue turned black.*

Conclusion:

ion	evidence
Cu^{2+}	*Pale blue ppt formed is insoluble in excess NaOH(aq).* *Blue residue decomposed to a black solid upon strong heating.*
NH_4^+	*Ammonia gas evolved when filtrate was warmed with NaOH(aq).*
CO_3^{2-}	*Effervescence observed. Gas gives white ppt in limewater.*

3.5 List of Reagents used in Qualitative Analysis

This section summarises the common reagents used in QA. Note that this list is non-exhaustive. Tests involving NO_2^-, SO_3^{2-} and SO_2 are not required to be carried out but knowledge of the tests is expected for QA planning.

S/N	Reagent	Test for?	What to look out for during the test?	Observations (<u>positive</u> test)	Key Products	Type of Reaction & Relevant Equations
1	NaOH(aq)	NH_4^+	Gas evolved during heating: $NH_3(g)$	Gas evolved turned moist red litmus paper blue. Gas is NH_3.	$NH_3(g)$	Acid-base reaction $NH_4^+(aq) + OH^-(aq) \rightarrow NH_3(aq) + H_2O(l)$
		Metal cations (M^{2+} or M^{3+})	1. Colour of ppt 2. Solubility of ppt in excess 3. Colour of solution formed if ppt is soluble in excess	Refer to QA table for observations specific to each metal cation. Examples: 1. <u><state colour></u> ppt formed, insoluble in excess NaOH(aq). 2. <u><state colour></u> ppt formed, soluble in excess NaOH(aq) to give a <u><state colour></u> solution.	$M(OH)_2(aq)$ or $M(OH)_3(aq)$ For soluble complexes: $[Al(OH)_4]^-(aq)$ $[Cr(OH)_6]^{3-}(aq)$ $[Zn(OH)_4]^{2-}(aq)$	Precipitation General equation: $M^{x+}(aq) + xOH^-(aq) \rightleftharpoons M(OH)_x(s)$ Complexation (for formation of hydroxide complex) Refer to *Section 3.3.1 Test for Cations* for specific equations.
2	NaOH(aq) + Al(s) + heat	NO_3^- NO_2^- (in absence of NH_4^+)	Gas evolved during heating: $NH_3(g)$	Gas evolved turned moist red litmus paper blue. Gas is NH_3. *Do not record 'effervescence observed' as it is due to $H_2(g)$ produced, not due to $NH_3(g)$.*	$NH_3(g)$	Redox reaction $3NO_3^-(aq) + 8Al(s) + 5OH^-(aq) + 18H_2O(l) \rightarrow 3NH_3(g) + 8[Al(OH)_4]^-(aq)$ $NO_2^-(aq) + 2Al(s) + OH^-(aq) + 5H_2O(l) \rightarrow NH_3(g) + 2[Al(OH)_4]^-(aq)$
3	$NH_3(aq)$	Metal cations (M^{2+} or M^{3+})	1. Colour of ppt 2. Solubility of ppt in excess 3. Colour of solution formed if ppt is soluble in excess	Refer to QA table for observations specific to each metal cation. Examples: 1. <u><state colour></u> ppt formed, insoluble in excess NaOH(aq). 2. <u><state colour></u> ppt formed, soluble in excess NaOH(aq) to give a <u><state colour></u> solution.	$M(OH)_2(s)$ or $M(OH)_3(s)$ For soluble complexes: $[Cu(NH_3)_4(H_2O)_2]^{2+}(aq)$ $[Zn(NH_3)_4]^{2+}(aq)$	Precipitation General equation: $M^{x+}(aq) + xOH^-(aq) \rightleftharpoons M(OH)_x(s)$ 9 b? Ligand Exchange Reaction (for formation of ammine complex) Refer to *Section 3.3.1 Test for Cations* for specific equations.

S/N	Reagent	Test for?	What to look out for during the test?	Observations (<u>positive</u> test)	Key Products	Type of Reaction & Relevant Equations
4	$Na_2CO_3(aq)$	H^+ (include organic acids)	Effervescence of $CO_2(g)$	Effervescence observed. Gas formed white ppt with limewater. Gas is CO_2.	$CO_2(g)$	Acid-base reaction $CO_3^{2-}(aq) + 2H_3O^+(aq) \rightarrow CO_2(g) + 3H_2O(l)$
		Al^{3+} Cr^{3+} Fe^{3+}	1. Colour of ppt 2. Effervescence of $CO_2(g)$	<u><state colour></u> ppt formed. Effervescence observed. Gas formed white ppt with limewater. Gas is CO_2.	white $Al(OH)_3(s)$ green $Cr(OH)_3(s)$ red-brown $Fe(OH)_3(s)$ $CO_2(g)$	Hydrolysis (by M^{3+}) $[M(H_2O)_6]^{3+} + H_2O \rightleftharpoons [M(H_2O)_5(OH)]^{2+} + H_3O^+$ Acid-base reaction $CO_3^{2-}(aq) + 2H_3O^+(aq) \rightarrow CO_2(g) + 3H_2O(l)$
		M^{2+}	Colour of ppt	<u><state colour></u> ppt formed. For Mn^{2+} and Fe^{2+}: ppt turns brown on standing.	white Grp 2 $MCO_3(s)$ white $ZnCO_3(s)$ off-white $MnCO_3(s)$ green $FeCO_3(s)$ pale-blue $CuCO_3(s)$	Precipitation General equation: $M^{2+}(aq) + CO_3^{2-}(aq) \rightleftharpoons MCO_3(s)$
5	Dilute acids: HCl H_2SO_4 HNO_3	CO_3^{2-}	Effervescence of $CO_2(g)$	Effervescence observed. Gas formed white ppt with limewater. Gas is CO_2.	$CO_2(g)$	Acid-base reaction $CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l)$
		NO_2^-	Gas evolved during heating: $NO(g) \rightarrow NO_2(g)$	Brown gas evolved. Gas is NO_2.	$NO(g) \rightarrow NO_2(g)$	Redox reaction $2NO_2^-(aq) + 2H^+(aq) \rightarrow NO_2(g) + NO(g) + H_2O(l)$
		SO_3^{2-}	Gas evolved during heating: $SO_2(g)$	Gas evolved turned acidified $KMnO_4(aq)$ from purple to colourless. Gas is SO_2.	$SO_2(g)$	Redox reaction $SO_3^{2-}(aq) + 2H^+(aq) \rightarrow SO_2(g) + H_2O(l)$
6	$AgNO_3(aq)$ followed by $NH_3(aq)$	Cl^-	1. Colour of ppt 2. Solubility of ppt in $NH_3(aq)$	White ppt formed, soluble in $NH_3(aq)$	AgCl(s)	Precipitation $Ag^+(aq) + Cl^-(aq) \rightleftharpoons AgCl(s)$
		Br^-		Cream ppt formed, partially soluble in $NH_3(aq)$	AgBr(s)	Precipitation $Ag^+(aq) + Br^-(aq) \rightleftharpoons AgBr(s)$
		I^-		Yellow ppt formed, insoluble in $NH_3(aq)$.	AgI(s)	Precipitation $Ag^+(aq) + I^-(aq) \rightleftharpoons AgI(s)$
7	$Ba(NO_3)_2(aq)$ OR $BaCl_2(aq)$	SO_4^{2-}	1. Colour of ppt 2. Solubility of ppt in dilute HCl or HNO_3	White ppt formed, insoluble in dilute HCl (or HNO_3).	$BaSO_4(s)$	Precipitation $Ba^{2+}(aq) + SO_4^{2-}(aq) \rightleftharpoons BaSO_4(s)$
		SO_3^{2-}		White ppt formed, soluble in dilute HCl (or HNO_3).	$BaSO_3(s)$	Precipitation $Ba^{2+}(aq) + SO_3^{2-}(aq) \rightleftharpoons BaSO_3(s)$ Acid-base reaction $BaSO_3(s) + 2H^+ \rightarrow Ba^{2+}(aq) + SO_2(g) + H_2O(l)$

S/N	Reagent	Test for?	What to look out for during the test?	Observations (positive test)	Key Products	Type of Reaction & Relevant Equations
8	Cu^{2+} salts (aq) e.g. $CuSO_4$	I^-	1. Colour of ppt 2. Colour change of solution	White ppt in brown solution formed.	$CuI(s)$ $I_2(aq)$	Redox reaction $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$
9	Acidified $KMnO_4(aq)$ (Oxidising agent)	Reducing Agents, e.g. NO_2^-, SO_3^{2-}, Cl^-, Br^- and I^-	1. Colour change of $KMnO_4(aq)$ 2. Gas evolved, ppt or other colour changes (if any)	Decolourisation of purple $KMnO_4$ solution	$Mn^{2+}(aq)$	Redox reaction $MnO_4^-(aq) + 8H^+(aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$
10	$KI(aq)$ (Reducing agent)	Cu^{2+}	1. Colour of ppt 2. Colour change of solution	White ppt in brown solution formed.	$CuI(s)$ $I_2(aq)$	Redox reaction $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(aq)$
		Fe^{3+}	Colour change of solution	Brown solution formed.	$Fe^{2+}(aq)$ $I_2(aq)$	Redox reaction $2Fe^{3+}(aq) + 2I^-(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$
11	$H_2O_2(aq)$ (Reducing agent OR oxidising agent)	Oxidising agents e.g. Fe^{3+} Reducing agents e.g. Cl^-	1. Effervescence of $O_2(g)$ 2. Gas evolved, ppt or other colour changes (if any)	Effervescence observed. Gas relights a glowing splint. Gas is O_2.	$O_2(g)$	Oxidation (half-equation) $H_2O_2(aq) \rightarrow O_2(g) + 2H^+(aq) + 2e^-$ Reduction (half-equation) $H_2O_2(aq) + 2H^+(aq) + 2e^- \rightarrow 2H_2O(l)$
12	$FeCl_3(aq)$	Reducing agents e.g. I^-	1. Colour change 2. Gas evolved, ppt or other colour changes (if any)	Yellow solution turned pale green.	$Fe^{2+}(aq)$	Redox reaction $2Fe^{3+}(aq) + 2I^-(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$
13	Concentrated HCl	Cu^{2+}	Colour change of solution	Blue solution turned yellow-green.	$[CuCl_4]^{2-}(aq)$	Ligand Exchange $[Cu(H_2O)_6]^{2+}(aq) + 4Cl^-(aq) \rightleftharpoons [CuCl_4]^{2-}(aq) + 6H_2O(l)$

4. GRAVIMETRIC ANALYSIS (GA)

4.1 Introduction

Gravimetric analysis (GA) is used to accurately quantify the masses of solid samples formed via thermal decomposition or precipitation. It enables the determination of information such as relative molecular mass (M_r) and amount of water of crystallisation of a salt.

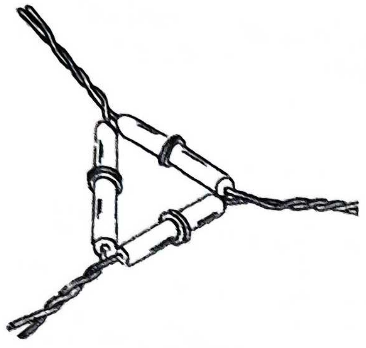
4.2 Common Substances involved in Thermal Decomposition

The table below shows common solid compounds which decompose upon heating.

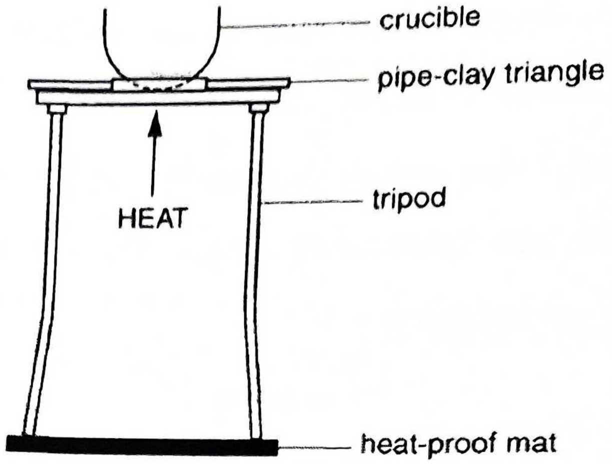
substance	effect upon heating
bicarbonate	Some bicarbonates thermally decompose to metal carbonates, water and carbon dioxide gas. *Example*: $2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$
carbonate	Some carbonates thermally decompose to metal oxides and carbon di *Example*: $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$
sulfite	Some sulfites thermally decompose to oxides and sulfur dioxide gas. *Example*: $CaSO_3(s) \rightarrow CaO(s) + SO_2(g)$
nitrate	Group I nitrates thermally decompose to nitrite and oxygen gas. *Example*: $KNO_3(s) \rightarrow KNO_2(s) + \frac{1}{2}O_2(g)$ Group II nitrates thermally decompose to give metal oxides, nitrogen dioxide and oxygen gas. *Example*: $2Mg(NO_3)_2(s) \rightarrow 2MgO(s) + 4NO_2(g) + O_2(g)$
hydroxide	Group II hydroxides thermally decompose (dehydrates) to oxides and water. *Example*: $Be(OH)_2(s) \rightarrow BeO(s) + H_2O(g)$
salts with water of crystallisation (hydrates)	Water molecules are lost from a salt with water of crystallisation. The anhydrous salt itself is stable to heating. *Example*: $CuSO_4.5H_2O(s) \rightarrow CuSO_4(s) + 5H_2O(g)$

4.3 General Procedure for Thermal Decomposition

- A crucible is typically used and is supported on a pipe-clay triangle and tripod.



pipe-clay triangle



General steps

1. Weigh a dry empty crucible using an electronic balance and record its mass.
2. Add a fixed mass of solid sample to the crucible. Weigh and record the total mass and mass of solid sample used.
3. **Heat** the crucible and its contents **gently** for one minute and then **strongly** for three minutes.
4. Allow to the crucible and its contents to **cool to room temperature**.
5. **Reweigh** the crucible and its contents using an electronic balance and record the mass.
6. **Repeat the heat-cool-reweigh** process (steps 3 to 5) until three **constant** mass readings are obtained. Record this final mass.

Results Presentation

Mass of crucible / g	28.504
Mass of crucible + sample / g	32.506
Mass of sample used / g	4.002
Mass of crucible and contents after 1st heating / g after 2nd heating / g after 3rd heating / g	 31.564 31.559 31.556
Mass of residue / g	3.052

Remarks:
- *Adapt* your label for "sample" according to the chemical given.

Remarks:
- Sample results shown.
- Mass recordings to 3 decimal places (follow exactly what is stated in the electronic balance).

5. INTRODUCTION TO PLANNING

This section will cover some basic guidelines when planning a scientific investigation in H2 Chemistry. Content related to planning experiments for Volumetric Analysis, Qualitative Analysis and Gravimetric Analysis will be also covered.

5.1 Introduction

The Planning skill area requires one to apply and integrate knowledge and understanding from different sections of the H2 Chemistry syllabus. As such, it is one of the most difficult skills to master.

The following competencies are expected in this skill area:

- define the question/problem using appropriate knowledge and understanding
- give a clear logical account of the experimental procedure to be followed
- describe how the data should be used in order to reach a conclusion
- assess the risks of the experiment and describe precautions that should be taken to keep risks to a minimum

Unlike the other skill areas (MMO, PDO and ACE) which typically require students to execute a defined procedure, the Planning skill area requires students to *design* a procedure. Students also need to propose how the expected data can be evaluated to achieve the aim of the investigation.

In some ways, designing a procedure to complete a scientific investigation is like designing a recipe for a new dish. Whether writing a recipe or procedure, there are three big questions to consider:

	Recipe for Cooking	Procedure for Experiment
Q1: Regarding **Method** *(March HBL: Intro to Planning 1)*	What is the main method to cook this dish? Steam, deep fry, bake etc.?	What is the main method to achieve the aim of the experiment? Titration, gas collection, etc.?
Q2: Regarding **Quantities** *(March HBL: Intro to Planning 2)*	How much of each ingredient should I use? How long should I cook each ingredient?	How much of each starting reagent should I use? What calculations can I do? How do I analyse my data?
Q3: Regarding **Procedure** *(March HBL: Intro to Planning 3)*	What are the steps needed to cook this dish? What are the details to be mindful about?	What is the logical flow of steps needed? What are the details required?

Consider the following actions to improve your concept of designing experimental procedures:

- Look back at the procedure given in the practical work you have done in the laboratory.
  - Understand the intent of each step and the sequence of the steps in the procedure.
  - How should the procedure be changed if the given reagents were changed?
- Visualise in your mind how the experiment will look like based on your proposed procedure during planning. Will there be any loopholes or gaps that will limit the validity of your procedure?

5.1.1 Guiding Questions for Planning an Experiment

The table below lists some guiding questions which need to be considered when planning a scientific investigation. A detailed application of the guiding questions will be covered in *Introduction to Planning: March Home-based Learning (HBL)*.

components	questions for consideration
Aim	• What is the aim of this experiment?
General Method	• How can this aim be achieved? • What is the general method? *Look at the apparatus and chemicals provided – does the question suggest what method should be used?* • Is there a chemical equation or formula that will be helpful? • What would be a logical flow of steps in this method? List the general steps.
Measurements	• What measurements do I need to take? • What apparatus should I use to take these measurements? • How should I record and present my data using tables?
Calculations	• Are there restrictions in the measurement instrument that limits the quantity of starting reagent? • Are there restrictions imposed by the question (e.g. limiting reagent)? • Do I need to perform a dilution step? • What calculations needs to be done in light of the previous 3 points?
Data Analysis *(if required by question)*	• How will the measurements that I have taken allow me to achieve the aim? • Use unknowns (e.g. x) or graph to help show how the aim is achieved.
Apparatus and reagents	• What apparatus do I need for the method? • What is the setup for the experiment? • Can I use a more precise apparatus?
Procedure (Achievable, Logical and Efficient)	• What is a logical flow for my procedure? • What specific data do I need to obtain? • How should the experiment end? • Have I considered accuracy and precision? • Have I considered safety *(if question requires)*?

5.1.2 General Format of Writing Procedures

- Number and write each step on a new line.

- Do not show calculation steps in the procedure unless required by the question.

- Phrase your steps as instructions i.e. use imperatives (command words).

  Example:

  Remarks:
  - Specify key apparatus (e.g. burette and volumetric flask).
  - Specify its capacity when the apparatus is first introduced.
  - For apparatus with different capacities, e.g. pipette and measuring cylinders, specify their capacities in every step for clarity.

1. Using a $50.00\ cm^3$ burette, add $50.00\ cm^3$ of $0.5\ mol\ dm^{-3}$ HCl to a $100\ cm^3$ volumetric flask.

Remarks:
- Use imperative (command words) e.g. add.
- Include adverbs if required (e.g. dropwise, slowly, quickly, immediately).

Remarks:
- Specify quantity with appropriate precision of the apparatus.
- Specify concentration of solution if given.

- At times, it may be necessary to specify the **purpose** (e.g. "to ensure solids dissolve completely") or **expected outcome/observation** (e.g. "until solution changes from yellow to orange").

- For time-based procedures, indicate the time at which the required action is to be executed (e.g. "At t = 5 min, pipette …").

- A list of verbs (action word) and adverbs typically used in chemical procedures are shown below for your reference. This list is non-exhaustive.

	verbs			**adverbs**	
add	weigh	heat	slowly	continuously	gently
fill	reweigh	cool	quickly	carefully	gradually
pour	empty	repeat	immediately	thoroughly	strongly
transfer	pipette	start	dropwise		
rinse	titrate	stop			
shake	filter	pause			
stir	dry	calculate			
swirl	wet	record			
invert	wash	draw			
place	stopper	cap			

5.2 Planning Volumetric Analysis (VA) Experiments

5.2.1 Approaching VA Planning Questions

- Planning VA experiments typically involve the following components:
    - Preparation of standard solution (from solid sample or dilution of liquid sample)
    - Titration

- The following are some considerations when planning VA experiments.

consideration	description
Type of reaction and end-point determination	• Identify from the information given whether the reaction is acid-base, redox, precipitation or complexation, etc. ○ Write chemical equations to help you if not given. • If a pH indicator is given in the list of chemicals and apparatus, it typically hints the use of acid-base titration. ○ Ensure you remember the colour of typical pH indicators under acidic, neutral and alkaline conditions. ○ If a pH indicator is not provided and you have determined the reaction to be acid-base by nature, decide on your own pH indicator. • Redox titrations are typically self-indicating, except for iodometric titration which requires the use of starch indicator. • **In short, if a colour change is not obvious at end-point, then an indicator is necessary.**
Use of volumetric flask	• A standard solution preparation requires a volumetric flask with a capacity of 100 cm^3 or 250 cm^3 typically. • As titration involves the reaction between two solutions, solid samples, if given, need to be dissolved or reacted first. • For solid compounds which are **soluble** in water: ○ A standard solution is directly prepared by dissolving the solid and are used in fixed portions for titration. • For solid compounds which are **insoluble** in water: ○ Back (or indirect) titration is required. ○ The solid is typically reacted with another compound to form an aqueous solution. ○ Transfer the aqueous solution into a volumetric flask to achieve an appropriate concentration. Use fixed portions of the diluted solution for titration. • If the concentration of a solution given in the question is relatively too high for appropriate use in titration, perform dilution by preparing a standard solution. ○ Use $C_oV_o = C_dV_d$ to calculate the volume of original solution (V_o) required. (V_d = capacity of volumetric flask) ○ Use a burette or pipette to transfer the solution as they are more accurate than a measuring cylinder.
Accuracy of titre volume	• The volume ratio between the titrant and analyte in a titration is typically about 1 : 1 to ensure minimum error of the titre volume. • If the volume of analyte (in conical flask) used is 25 cm^3, a suitable titre volume to assume will be between 20 – 25 cm^3 for pre-calculations. ○ A titre volume that is too small (e.g. 5.00 cm^3) will result in large percentage error. ○ A titre volume that is too large (e.g. 60.00 cm^3) necessitates refilling the burette, which is inefficient and increases error.

- The table below illustrates some key elements which should be covered in your procedure. Please note that it is **not meant to be prescriptive. Adapt your procedure according to the context of the experiment.** Add steps where necessary.

method	general key elements
Preparation of Standard Solution (from solid)	1. Weigh a weighing bottle containing <state solid> **using an electronic balance** and **record the mass**. 2. **Empty the contents** of the weighing bottle into a 100 cm^3 beaker. 3. **Reweigh** the weighing bottle containing any residual <state solid>. **Record the mass** and calculate the mass of <state solid> used. 4. Add about 50 cm^3 of deionised water to the beaker containing the <state solid>. Stir with a glass rod to **ensure that the solid dissolves completely**. 5. Transfer this solution into a 250 cm^3 volumetric flask. 6. **Rinse the beaker thoroughly** with deionised water **and transfer the washings into the volumetric flask**. 7. Add deionised water to the volumetric flask **until the liquid level reaches the graduation mark**. 8. **Stopper** the flask and **invert a few times to ensure a homogeneous solution**.
Preparation of Standard Solution (dilution)	1. **Using a burette (or pipette)**, transfer 25.00 (or 25.0 cm^3) of <state solution and its concentration> into a 100 cm^3 **volumetric flask**. 2. [For burette] Record **initial and final burette readings**. 3. **Add deionised water** to the volumetric flask until the **water level reaches the mark**. 4. **Stopper** the flask and **invert it a few times to ensure a homogeneous solution**.
Titration	*Example shown for acid-base titration. Adapt accordingly for redox titration.* 1. Fill a 50.00 cm^3 **burette** with <state titrant and its concentration>. Record the **initial burette reading**. 2. **Pipette** 25.0 cm^3 of <state analyte> into a 250 cm^3 conical flask placed on a white tile and add **2 drops of <state indicator>**. 3. Add <state titrant> from the burette into the conical flask with **continuous swirling**. 4. Add <state titrant> **dropwise towards the end-point and swirl**. Stop the addition when one drop of <state titrant> turns the solution from **<initial colour of indicator> to <final colour of indicator>**. Record the **final burette reading** and calculate the titre volume of <state titrant>. 5. **Repeat** the titration until **two titre volumes within ±0.10 cm^3 of each other** are obtained.