VOLUMETRIC ANALYSIS

Students learn about	Students should be able to
4.1. Volumetric analysis	
Standardisation	
• primary standards, and standard solutions, for acid-base and redox volumetric analysis	1. recognise the importance of primary standards and standard solutions
Titrations	

• investigations, to include:
  ◦ strong acid-strong base
  ◦ strong acid-weak base
  ◦ iodine-thiosulfate
  ◦ vinegar
  ◦ iron(II)
	2. determine the concentration of analytes by titration using primary standard solutions and/or solutions standardised using primary standards

• all of the above using indicators **or pH curves**, as appropriate	3. **investigate pH titration curves, using primary data from acid-base reactions, justifying appropriate indicators for each titration**
• **issues around generation of curves for weak acid-weak base systems (limited to monoprotic and single inflection point curves)**	4. **explain how weak acid and weak base acid-base indicators function, using Le Châtelier's principle**
	5. investigate, using primary data, how to find the concentration of ethanoic acid in vinegar[EI]
	6. investigate, using primary data, the percentage of elemental iron in iron tablets[EI]
Volumetric problems	
• problem solving using varied units (see quantifying matter section)	7. solve volumetric problems
• volumetric calculations in familiar and **unfamiliar contexts** for acid-base and redox titrations	
Chemical solutions	
• chemical solutions that includes: solutions, solutes, solvents, dilution and serial dilutions, concentration, unsaturated/saturated solutions, supersaturated solutions, standard solutions, solutions made from primary standards, taking into account precision, accuracy, volumes and glassware required	5. explain and model the concept of concentration of a solution and outline how to prepare a range of solutions of different concentrations, including primary standard solutions
• units for expressing concentration: g/L, mol/L, %w/w, %v/v, %w/v and **p.p.m.**	6. convert between units of concentration

Alistair Jack: Bruce College

Volumetric analysis is a method used to find the concentration or amount of a substance in a solution. It involves reacting carefully measured volumes of solutions together. This process is called a titration.

Volumetric analysis is an important part of Leaving Certificate Chemistry because it develops skills in:

- Concentration calculations,
- Accurate measurement,
- Solution preparation,
- The correct use of laboratory glassware.
- (All of which are important for your project.)

Concentration calculations used in volumetric analysis

- In volumetric analysis, chemists and students need to know exactly how much solute is dissolved in a solution. The amount of solute present in a given volume of solution is called the concentration of the solution.
- A solution is formed when a solute dissolves in a solvent to form a homogeneous mixture.

Definition: Solution:

- **A solution is a completely perfect mixture of a solute and a solvent. In a solution, the particles of the solute are uniformly distributed throughout the solvent, i.e. the mixture is homogeneous.**

• Solute = the substance being dissolved.
• Solvent = the substance that dissolves the solute.
• Solution = a homogeneous mixture of a solute and a solvent.

Solutions can be:

- **concentrated**, meaning they contain a large amount of solute,

or

- **dilute**, meaning they contain a small amount of solute.

- The exact measure of the amount of solute in a certain volume of a solution is known as the **concentration** of a solution.

Definition: Concentration

- **The concentration of a solution is the amount of solute that is dissolved in a given volume of solution.**

There are six different ways of representing the concentration of a solute in a solution.

	CONCENTRATION (UNIT)	MEANING
% weight per weight →	% (w/w)	g per 100 g
% weight per volume →	% (w/v)	g per 100 cm^3
% volume per volume →	% (v/v)	cm^3 per 100 cm^3
Moles per litre / Molarity →	M	mol per L
Grams per litre →	g per L	g per L
Parts per million →	ppm	mg per L

Note: these type of calculations are not limited to volumetric analysis , they can make an appearance anywhere on the paper.

Calculating g/L: Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre

Calculating %(w/v): Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre $\xrightarrow{\div 10}$ %(w/v)

Calculation ppm: Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre $\xrightarrow{\times 1000}$ ppm

Percentage of solute

- Percentage concentration is used to show how much solute is present in a solution, expressed as a percentage.
- There are three main types: (%W/W), (%W/V), and (%V/V)

Weight per Weight (%W/W)

- % w/w is used when the mass of solute is compared with the total mass of solution.
- A solution labelled 5% w/w copper sulfate solution means there are 5 g of copper sulfate per 100 g of solution.

- **Formula: % w/w =** $\frac{\text{mass of solute}}{\text{total mass of solution}}$ **x 100**

Practice Questions:

A student prepares a copper sulfate solution by dissolving 18 g of $CuSO_4$ in 132 g of water.Calculate the percentage concentration of the solution in % w/w.

A copper sulfate solution is labelled 12% w/w. Calculate the mass of copper sulfate needed to prepare 250 g of this solution.

Weight per Volume, (% w/v)

- % w/v is used when the mass of solute is compared with the total volume of solution.
- A vinegar solution labelled 4% w/v ethanoic acid means there are 4 g of ethanoic acid per

- **Formula: % w/v =** $\dfrac{\textbf{mass of solute}}{\textbf{total volume of solution}}$ **x 100**

Practice Questions:

A vinegar solution contains 6 g of ethanoic acid in 150 cm^3 of solution. Calculate the concentration of the solution in % w/v.

A vinegar solution contains 0.75 mol of ethanoic acid (CH_3COOH) per litre of solution. Calculate the concentration of the solution in: g/L, and % w/v.

Volume per Volume, % v/v

- % v/v is used when a liquid solute is dissolved in a liquid solvent.
- An ethanol solution labelled 15% v/v ethanol means there are: 15 cm^3 of ethanol per 100

- **Formula: % v/v =** $\frac{\text{Volume of solute}}{\text{total volume of solution}}$ **x 100**

Practice Questions:

A solution contains 35 cm^3 of ethanol in 250 cm^3 of solution. Calculate the concentration of the solution in % v/v.

A hand sanitiser solution is labelled 70% v/v isopropyl alcohol. Calculate the volume of isopropyl alcohol needed to prepare 400 cm^3 of solution.

Parts per Million, p.p.m.

- Parts per million (p.p.m.) is used for very dilute solutions where only a very small amount of substance is present.

- It is especially important in: water analysis, pollution monitoring, environmental chemistry,

1 p.p.m. = 1 mg/L

This means:

- 1 milligram of solute per litre of solution = 1 p.p.m.

This relationship works because:

- 1 L of water has a mass of approximately 1 kg, and 1 kg = 1,000,000 mg.

Example:

- 5 p.p.m. chlorine means there are 5 mg of chlorine per litre of water.

Practice Questions:

A sample of river water contains 0.003 g of lead ions per litre. Calculate the concentration in p.p.m.

A polluted water sample contains 0.008 g of copper ions in 250 cm^3 of water. Calculate the concentration in p.p.m.

A water sample contains 0.002 mol of calcium carbonate ($CaCO_3$) in 500 cm^3 of water. Calculate the concentration of calcium carbonate in p.p.m.

Molarity, mol/L

- Molarity is one of the most important ways of expressing concentration in volumetric analysis.

Definition: Molarity.
- **The molarity of a solution is the number of moles of solute per litre of solution.**

- **Units:** M or mol/L

Example:
- 1 M HCl means 1 mole of HCl dissolved per litre of solution.

- **Formula:** $\textbf{No. of moles} = \frac{\textbf{Volume x Molarity}}{\textbf{1000}}$

Practice questions:

Calculate the molarity of a solution containing 0.50 mol of NaOH dissolved in 250 cm^3 of solution.

How many moles of HCl are present in 500 cm^3 of 1.5 mol/L HCl?

A student dissolves 11.7 g of NaCl in water and makes the solution up to 400 cm^3. Calculate the concentration of the solution in mol/L.

What is the molarity of a solution that contains 4.90 g of NaOH per litre of solution?

Calculate the concentration in moles per litre of a solution containing 36 g of sulfuric acid per 200 cm^3 of solution.

What volume of 0.20 M sodium hydroxide solution will contain 8 grams of sodium hydroxide?

Reactions Between a Solution and a Solid

- In some chemical reactions, a solution reacts with a solid substance, such as a metal reacting with an acid solution. (A balanced equation may be required)

What mass of zinc will react with 75 cm^3 of 0.40 M HCl?

What mass of magnesium will react with 80 cm^3 of 0.25 M H_2SO_4?

Dilution of Solutions

- Many chemicals in laboratories are supplied as very concentrated solutions. Before they can be used safely in experiments, they are often diluted by adding water.
- When a solution is diluted more solvent is added, the volume increases, but the amount of solute stays the same.
- Because the same number of moles is now spread throughout a larger volume, the concentration decreases.

Formula: $C_1V_1 = C_2V_2$

- C_1 = original concentration.
- V_1 = original volume.
- C_2 = new concentration after dilution.
- V_2 = final volume after dilution.

Practice questions:

What volume of 8 M HCl is required to prepare 400 cm^3 of 2 M HCl?

A student has 15 M nitric acid. What volume of this acid is needed to prepare 750 cm^3 of 3 M nitric acid?

Volumetric Titrations

Standard solutions

- In volumetric analysis, chemists need solutions with accurately known concentrations. These are called standard solutions.

Definition: Standard solution.
- **A standard solution is a solution whose concentration is accurately known.**

- Standard solutions are extremely important in titrations because they allow chemists to accurately determine the concentration of another solution.

A standard solution is prepared by:
- accurately weighing a known mass of solute,
- dissolving it in water,
- and making the solution up to an exact volume using a volumetric flask.

However, not every substance can be used to prepare a standard solution directly. Some substances:
- absorb water from the air,
- lose water to the air,
- evaporate easily,
- are not pure enough.

For this reason, chemists use substances called **primary standards.**

Definition: Primary standard.
A primary standard is a substance that can be obtained in a stable, pure and soluble solid form so that it can be weighed out and dissolved in water to give a solution of accurately known concentration.

Criteria for a Primary Standard

- Be available in a highly pure state.
- Be stable in air.
- Dissolve easily in water.
- Have a fairly high relative molecular mass.
- React completely and rapidly.
- Preferably be anhydrous, meaning it contains no

Substances that cannot be used as primary standards

Substance	Why is it not a primary standard?
Sulfuric acid	It is hygroscopic, meaning it absorbs water from the air.
Hydrochloric acid	HCl is a gas at room temperature and is difficult to weigh accurately.
Sodium hydroxide	It is deliquescent, meaning it absorbs enough water from the air to form a solution.
Hydrated sodium carbonate	It effloresces, meaning it loses water of crystallisation to the air.
Iodine	It sublimes at close to room temperature.
Potassium permanganate	It is not available in a pure state and its solutions deteriorate when exposed to light.

Primary Standards:

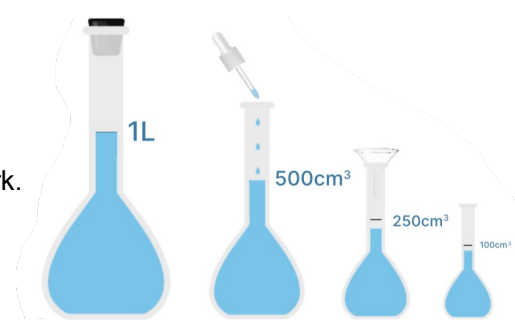
- Anhydrous sodium carbonate (Na_2CO_3).
- Sodium chloride (NaCl).
- Ammonium iron(II) sulfate.

Equipment used

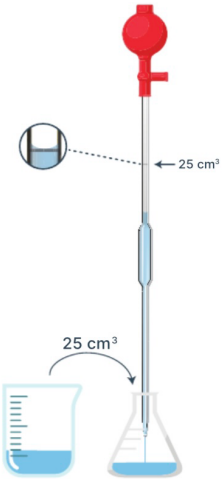
Volumetric Flask
(making and storing solutions)

Washing:
1. Rinse with deionised water (remove impurities)

Setup:
- Solution added using a **funnel**. Funnel is then removed so drops do not fall when filling to mark.
- Add water until close to the mark, then add the rest with a dropper pipette until you reach the mark
- All readings are read from the bottom of meniscus **(at eye level)**
- Stopper and invert 15 to 20 times to make homogeneous mixture.



Pipette
(measuring volume for conical flask)

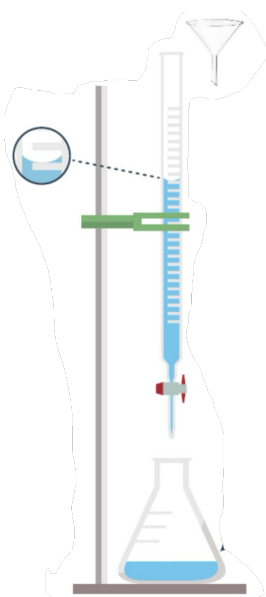


Setting up:
- Held vertically with no air bubbles (false volume)

Washing:
1. Rinse with deionised water (remove impurities)
2. Rinse with solution it will contain (remaining drops do not dilute solution)

Setup:
- Use pipette filler to fill above the mark, then allow the liquid to drain until bottom of meniscus (eye level) is on the mark
- Make sure to drain under gravity. Tip of pipette is touched against inside of conical flask to remove

Burette
(measuring volume of acid required for neutralisation)



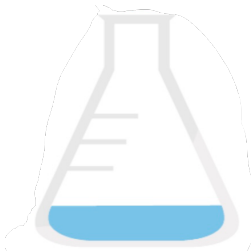
Setting up:
Clamped vertically with no air bubbles (false volume)

Washing:
1. Rinse with deionised water (remove impurities).
2. Rinse with solution it will contain (remaining drops do not dilute solution).

Usage:
- Solution added using a funnel. Funnel removed so drops do not fall during.
- All readings from bottom of meniscus (at eye level).
- Fill volume above 0 cm^3, run liquid to fill the tip of the burette (included in volume reading) and stop at 0 cm^3.

- **Both the burette and the pipette can also be used to transfer small volumes of solution because they are designed to give very accurate and precise readings.**

Conical Flask (titration flask)
(where acid base reaction happens)



Use:

- Conical shape prevents spillage when swirled

Washing:

- Rinse with deionised water only

Volumetric Analysis (Acid/Base) titrations and calculations

Volumetric analysis calculations may involve different types of titrations, such as acid base or redox, but the method used is almost always the same. All questions are solved using first principles, meaning you follow a simple logical process every time. You begin by calculating the number of moles of the known solution, use the balanced chemical equation to apply the mole ratio, and then determine the quantity of the unknown, such as its concentration or mass (M/L or G/L). The balanced equation is the key step, as it links all substances through their mole ratios. No matter how the question is presented, the same approach applies, so students should focus on understanding this one method and using it consistently.

	CONCENTRATION (UNIT)	MEANING
% weight per weight →	% (w/w)	g per 100 g
% weight per volume →	% (w/v)	g per 100 cm^3
% volume per volume →	% (v/v)	cm^3 per 100 cm^3
Moles per litre / Molarity →	M	mol per L
Grams per litre →	g per L	g per L
Parts per million →	ppm	mg per L

Note: these type of calculations are not limited to volumetric analysis , they can make an appearance anywhere on the paper.

Calculating g/L: Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre

Calculating %(w/v): Moles per litre $\xrightarrow{\times \text{RMM}}$ Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre $\xrightarrow{\div 10}$ %(w/v)

Calculation ppm: Moles per litre $\xrightarrow{\times \text{RMM}}$ Grams per litre $\xrightarrow{\times 1000}$ ppm

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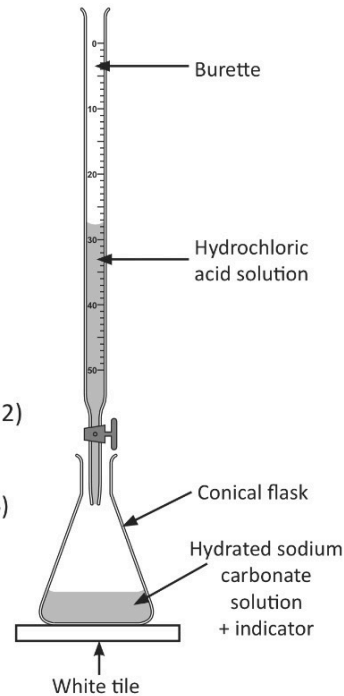
2026, Examcraft pre, Q1

1. To determine the average value of **x** in hydrated sodium carbonate (**$Na_2CO_3.xH_2O$**), 4.69 grams of the salt were weighed out and dissolved in deionised water. The solution was then made up to 250 cm^3 in a volumetric flask.

20.0 cm^3 of this solution was transferred to a conical flask and titrated against a previously standardised 0.13 M hydrochloric acid (**HCl**) solution using a suitable indicator.

The balanced equation for the titration reaction is

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2$$



(*a*) Describe the procedure for dissolving the hydrated salt and making up the solution to 250 cm^3 in the volumetric flask. (12)

(*b*) Name a suitable indicator for the titration and state the colour change observed in the conical flask at the end point. (6)

(*c*) After carrying out a number of accurate titrations using the 20 cm^3 of hydrated sodium carbonate solution, the mean titration figure was found to be 20.17 cm^3 of the 0.13 M hydrochloric acid solution.

Calculate:

(*i*) the number of moles of hydrochloric acid in 20.17 cm^3 of the standard solution.

(*ii*) the number of moles of sodium carbonate in the 20 cm^3 portion.

(*iii*) the concentration of anhydrous sodium carbonate in

(a) moles/litre,

(b) grams/litre. (21)

(*d*) Calculate the weight of water in the original sample and hence the percentage of water of crystallisation in the hydrated sodium carbonate. Calculate the value of x, the degree of hydration of the original crystals. (11)

The standard solution

The solution whose concentration you want to find in M/L

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

2020, Q1 Washing soda is a cheap, household chemical used for laundry, removing grease and softening water. Washing soda crystals are hydrated sodium carbonate ($Na_2CO_3.xH_2O$). The crystals effloresce (lose some water of crystallisation) in dry air becoming powdery in the process.

To determine the average value of ***x*** in the formula $Na_2CO_3.xH_2O$ for a sample of washing soda, 3.46 g of the crystals were dissolved in deionised water and made up to exactly 250 cm^3 of solution. 25.0 cm^3 volumes of this solution were pipetted into a conical flask and titrated with a previously standardised hydrochloric acid solution using a suitable indicator.

The balanced equation for the titration reaction is:

$$2HCl + Na_2CO_3 \rightarrow 2NaCl + H_2O + CO_2$$

On average 21.5 cm^3 of 0.12 M hydrochloric acid solution were required to completely neutralise 25.0 cm^3 of the washing soda solution.

Find by calculation

(*i*) the average number of moles of **HCl** used up in a titration,

(*ii*) the number of moles of Na_2CO_3 neutralised in each titration,

(*iii*) the number of moles of Na_2CO_3 in 250 cm^3 of the washing soda solution,

(*iv*) the mass of Na_2CO_3 in 250 cm^3 of the washing soda solution,

(*v*) the mass of water of crystallisation and hence the number of moles of water in 3.46 g of the crystals,

(*vi*) the ratio of moles of water of crystallisation to moles of Na_2CO_3 in the crystals and hence the value of ***x*** to the nearest whole number. (21)

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

TO USE A STANDARD SOLUTION OF SODIUM CARBONATE TO STANDARDISE A SOLUTION OF HCL

Purpose:

- To use a standard sodium carbonate solution to determine the concentration of a hydrochloric acid solution.

Intro:

- A titration is a laboratory technique used to determine the concentration of a solution. It involves accurately measuring the volume of one solution needed to react completely with a known volume of another solution.
- In this experiment, the concentration of the sodium carbonate solution is accurately known. A measured volume of this solution is placed in a conical flask. Hydrochloric acid of unknown concentration is gradually added from a burette until it has reacted completely with the sodium carbonate.
- Methyl orange is used to identify the end point. It changes from yellow to a faint red or pink colour when the end point is reached.
- The volume of hydrochloric acid used and the balanced equation can then be used to calculate its concentration.

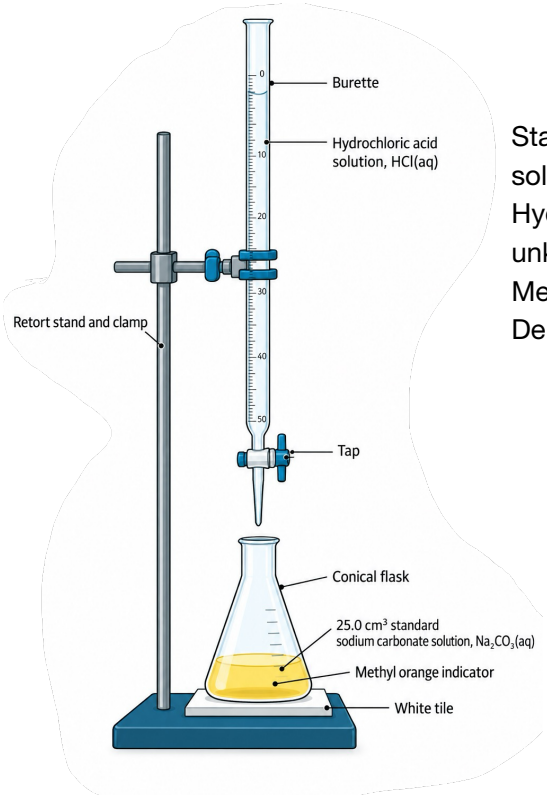
Key Info:

- To standardise a solution means to determine its concentration accurately.
- The sodium carbonate solution has a known concentration and is the standard solution.
- The sodium carbonate solution is transferred into the conical flask using a pipette.
- The hydrochloric acid has an unknown concentration and is placed in the burette.
- A few drops of methyl orange are added to the conical flask.
- Methyl orange is yellow in the sodium carbonate solution.
- At the end point, the indicator changes from yellow to a faint red or pink colour.
- One mole of sodium carbonate reacts with two moles of hydrochloric acid.
- One rough titration is completed before the accurate titrations.
- Accurate titres should agree to within 0.1 cm^3.
- The rough titre is not included when calculating the average titre.

Alistair Jack: Bruce College

Apparatus

25 cm^3 pipette.
Pipette filler.
Burette.
Funnel.
Retort stand, boss head and clamp.
Conical flask.
Beakers.
White tile.
Wash bottle.
Dropper.



Chemicals

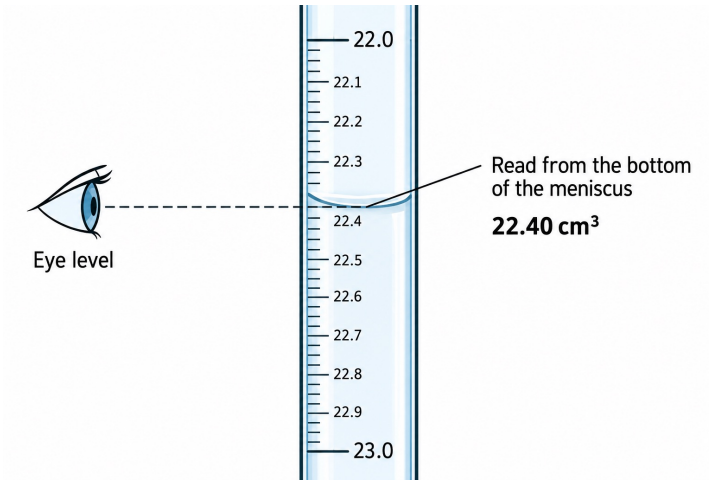
Standard sodium carbonate solution.
Hydrochloric acid solution of unknown concentration.
Methyl orange indicator.
Deionised water.

Procedure:

- Wash the pipette, burette and conical flask with deionised water.
- Rinse the pipette with the sodium carbonate solution.
- Rinse the burette with the hydrochloric acid solution.
- Using a **pipette filler,** transfer 25.0 cm^3 of sodium carbonate solution from a dry beaker into the conical flask.
- Add a few drops of methyl orange indicator. The solution should be yellow.
- **Clamp the burette vertically and use a funnel to fill it** above the zero mark with hydrochloric acid.
- Remove the funnel. Open the tap to fill the burette tip and remove any air bubbles.
- Adjust the **bottom of the meniscus to the zero mark.** Read the burette at eye level.
- Place the conical flask on a white tile beneath the burette.
- Carry out a rough titration. Add the acid fairly quickly while continuously swirling the conical flask.
- Wash down the inside walls of the conical flask occasionally using deionised water.
- Stop adding acid when the indicator changes from yellow to red or pink. Record the titre.
- Carry out an accurate titration. Add the acid quickly until close to the rough titre, then add it dropwise while swirling continuously.

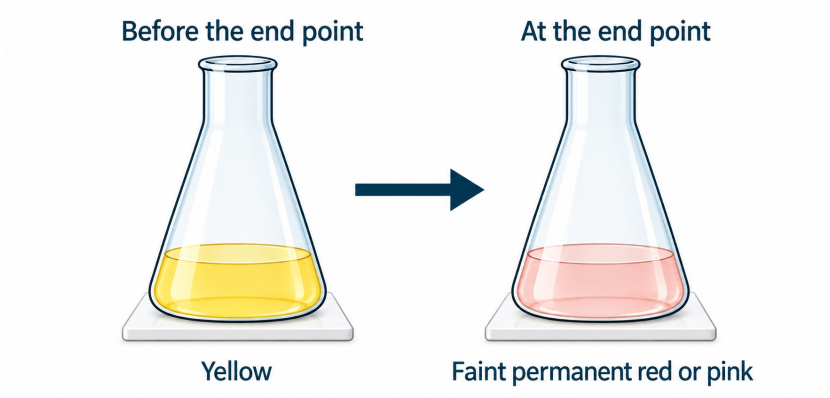
Alistair Jack: Bruce College

- Stop when one drop produces a faint permanent red or pink colour. Record the final burette reading.
- Repeat the accurate titration until two titres agree to within 0.1 cm^3.
- Calculate the average of the concordant titres. Do not include the rough titre.



Observations:

- The sodium carbonate solution is colourless.
- When methyl orange is added, the solution becomes yellow because sodium carbonate is alkaline.
- As hydrochloric acid is added, the yellow colour remains while sodium carbonate is still present.
- At the end point, one final drop of hydrochloric acid changes the solution from yellow to a faint permanent red or pink colour.
- Colour change: **Yellow** → **Faint permanent red**



Precautions and Explanations

Precaution or action	Explanation
Wash all apparatus with deionised water.	This removes impurities that could react with the solutions.
Rinse the pipette with sodium carbonate solution.	This prevents water in the pipette from diluting the sodium carbonate solution.
Rinse the burette with hydrochloric acid solution.	This prevents water in the burette from diluting the hydrochloric acid solution.
Rinse the conical flask only with deionised water.	Rinsing it with sodium carbonate solution would add an unknown number of moles.
Use a dry beaker for the sodium carbonate solution.	Water in the beaker would dilute the standard solution before it was pipetted.
Use a pipette filler.	A pipette must never be filled by mouth.
Do not blow the final drop from the pipette.	The pipette is calibrated to leave this drop inside its tip.
Add only a few drops of methyl orange.	Acid base indicators are weak acids or weak bases. Adding too much could affect the titre.
Use methyl orange as the indicator.	It is suitable for a titration between a strong acid and a weak base.
Place the acid in the burette and the base in the conical flask.	Bases such as sodium hydroxide can attack glass and absorb carbon dioxide, causing the burette tap to stick.
Remove the funnel from the burette.	Drops remaining in the funnel could enter the burette and change the reading.
Fill the burette tip with hydrochloric acid.	This removes air bubbles and ensures that all the measured acid enters the conical flask.
Clamp the burette vertically.	This allows the volume to be measured accurately.
Read the bottom of the meniscus at eye level.	This prevents parallax error.
Place the conical flask on a white tile.	This makes the indicator colour change easier to see.
Swirl the conical flask continuously.	This ensures that the solutions mix and react completely.
Wash down the sides of the flask with deionised water.	This ensures that all drops of acid reach the reaction mixture.
Add deionised water to the conical flask when required.	It changes the total volume but does not change the number of moles of sodium carbonate present.
Add the acid drop by drop near the end point.	This prevents too much acid from being added.
Carry out a rough titration first.	This gives the approximate end point and makes the accurate titrations faster.
Do not include the rough titre in the average.	It is only an approximate result.
Obtain titres that agree within 0.10 cm^3.	Concordant titres show that the results are precise and reliable.
Stop at the first faint permanent red or pink colour.	This shows that the solution has become acidic and the end point has been reached.

A student pipetted 25.0 cm^3 of 0.0500 mol L^{-1} standard sodium carbonate solution into a conical flask and titrated it with hydrochloric acid of unknown concentration, using methyl orange indicator.

(a) Write the balanced chemical equation for the reaction.

(b) Calculate the number of moles of sodium carbonate present.

(c) If the average titre was 24.8 cm^3 of hydrochloric acid, calculate the concentration of the hydrochloric acid in mol L^{-1}.

	Na_2CO_3 Reagent x	HCl Reagent y
Molarity	0.0500	
Volume	$25cm^3$	$24.8cm^3$
No. of moles	0.00125	0.0025
Ratio	1	2

$2HCl + Na_2CO_3 \rightarrow H_2O + CO_2 + 2NaCl$

$moles = \frac{25 \times 0.0500}{1000} = 0.00125\ mol/25cm^3$

$0.00125 \times 2 = 0.0025\ mol/24.8cm^3$

$\frac{0.0025 \times 1000}{24.8} = 0.1008\ M/L$

2012, Q1

A student determined the concentration of a hydrochloric acid solution by titration with 25.0 cm^3 portions of a 0.05 M primary standard solution of anhydrous sodium carbonate. The portions of sodium carbonate solution were measured into a conical flask using a 25 cm^3 pipette. The hydrochloric acid solution was added from a burette. The mean titre was 20.8 cm^3.

The balanced equation for the titration reaction was:

$$2HCl + Na_2CO_3 \longrightarrow 2NaCl + H_2O + CO_2$$

(*a*) Explain the underlined term. (5)

(*b*) Describe how the student should have prepared 500 cm^3 of the 0.05 M primary standard solution from a known mass of pure anhydrous sodium carbonate, supplied on a clock glass. (12)

Calculate the exact mass of anhydrous sodium carbonate (Na_2CO_3) required to prepare this solution. (6)

(*c*) (*i*) Describe how the liquid level in the burette was adjusted to the zero mark.

(*ii*) Why was a pipette filler used to fill the pipette with 25.0 cm^3 of the sodium carbonate solution? (6)

(*d*) Name a suitable indicator for this titration.

State the colour change observed at the end point. (9)

(*e*) Calculate, correct to two decimal places, the concentration of the hydrochloric acid solution in

(*i*) moles per litre,

(*ii*) grams per litre. (12)

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

TO MAKE UP AN APPROXIMATE SOLUTION OF SODIUM HYDROXIDE AND TO STANDARDISE IT WITH A STANDARD SOLUTION OF HCl

Purpose:

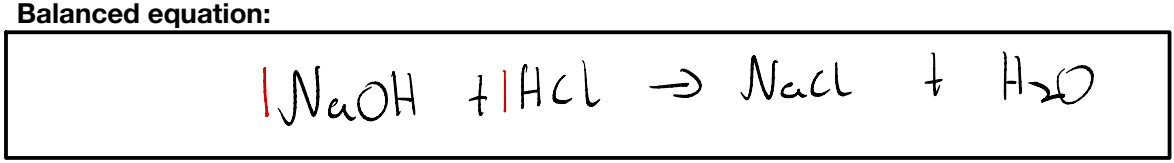
- To prepare an approximately 0.1 mol/L sodium hydroxide solution and determine its exact concentration by titrating it against a standard hydrochloric acid solution.

Introduction:

- The first titration (Na_2CO_3 vs HCl) contains the full guidance on preparing and using volumetric apparatus, reading the meniscus, carrying out rough and accurate titres. These techniques apply to every titration and will not be repeated in full. From this experiment onwards, only new information and steps specific to each titration will be included.

- Sodium hydroxide is **deliquescent**, meaning that it absorbs so much water vapour from the air that it eventually forms a solution. Therefore, an exact mass of pure sodium hydroxide cannot be weighed accurately to prepare a standard solution. An approximately 0.1 mol/L solution is prepared and its exact concentration is then determined by titrating it against hydrochloric acid of known concentration.

Balanced equation:



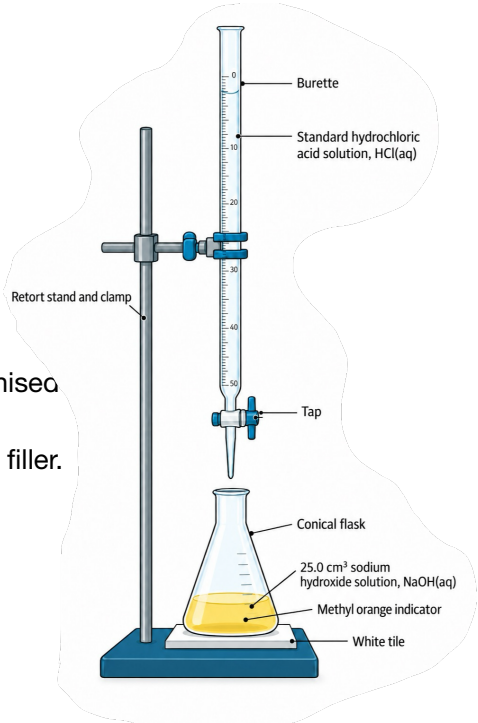
Key Info:

- Type of titration: Strong acid against strong base.
- Solution in the burette: Standard hydrochloric acid of known concentration.
- Solution in the conical flask: 25.0 cm^3 of sodium hydroxide of unknown exact concentration.
- Indicator: Methyl orange.
- Initial colour: Yellow, because the sodium hydroxide solution is alkaline.
- End point colour: The first permanent red/pink colour.
- Mole ratio: HCl : NaOH = 1 : 1.
- Safety: Sodium hydroxide and hydrochloric acid are corrosive and must not come into contact with the skin or eyes.

Alistair Jack: Bruce College

Apparatus

Electronic balance.
Spatula.
Weighing container.
Beaker.
Glass rod.
Funnel.
250 cm^3 volumetric flask.
Wash bottle containing deionised water.
25.0 cm^3 pipette and pipette filler.
Burette and burette funnel.
Retort stand and clamp.
Conical flask.
Dropping pipette.
White tile.



Chemicals

Approximately 1.0 g of solid sodium hydroxide.
Deionised water.
Standard hydrochloric acid solution of known concentration.
Methyl orange indicator.

Procedure:

A. Preparation of the Sodium Hydroxide Solution

- Weigh approximately 1.0 g of sodium hydroxide and dissolve it in deionised water.
  -**Purpose**: To prepare an approximately 0.1 mol dm^{-3} sodium hydroxide solution.
- Transfer the solution to a 250 cm^3 volumetric flask and make it up to the calibration mark using the solution preparation technique described in the first titration.
  -**Purpose**: To produce exactly 250 cm^3 of solution.
- Stopper the volumetric flask and invert it approximately 20 times.
  -**Purpose**: To ensure that the solution is completely mixed and has the same concentration throughout.

B. Standardisation of the Sodium Hydroxide Solution

- Rinse the pipette with deionised water followed by a small quantity of the sodium hydroxide solution.
  -**Purpose**: Rinsing with sodium hydroxide prevents water remaining in the pipette from diluting the solution.
- Rinse the burette with deionised water followed by a small quantity of the standard hydrochloric acid solution.
  -**Purpose**: Rinsing with hydrochloric acid prevents water remaining in the burette from diluting the acid.

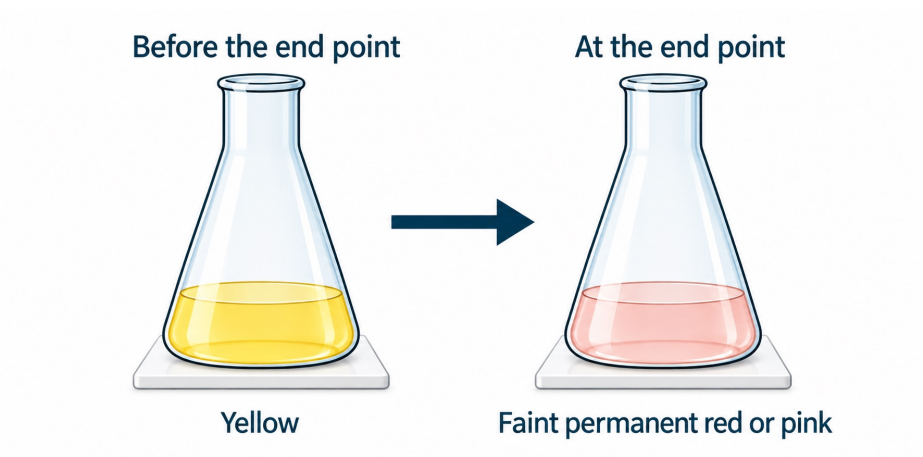
- Use the pipette to transfer 25.0 cm^3 of sodium hydroxide solution into a conical flask that has been rinsed with deionised water.
  -**Purpose**: The pipette delivers an accurate fixed volume of sodium hydroxide solution.
- Add a few drops of methyl orange indicator to the sodium hydroxide solution.
  -**Purpose**: Methyl orange shows when the end point of the titration has been reached.
- Fill the burette with the standard hydrochloric acid solution and adjust the bottom of the meniscus to the zero graduation mark.
  -**Purpose**: This allows the volume of hydrochloric acid added to be measured accurately.
- Place the conical flask on a white tile beneath the burette and titrate the sodium hydroxide solution with hydrochloric acid.
  -**Purpose**: The white tile makes the indicator colour change easier to observe.
- Carry out one rough titration followed by at least two accurate titrations that agree within 0.1 cm^3

Observation:

- The methyl orange indicator is yellow in the sodium hydroxide solution.
- At the end point, the indicator changes from yellow to red (pink).

Explanation:

- Methyl orange changes from yellow to red (pink) because the solution in the conical flask becomes acidic.



2026, Sample paper 3, Q6, (b)

(b) The team collects a 25.0 cm^3 sample of an aqueous sodium hydroxide solution to absorb CO_2. The solution is titrated against 0.100 mol L^{-1} hydrochloric acid, and the average titre is 23.6 cm^3.

(i) Write the balanced chemical equation for the titration.

$NaOH + HCl \rightarrow NaCl + H_2O$

(ii) Calculate the concentration of the sodium hydroxide solution in (a) moles/L (b) g/L.

(iii) NaOH absorbs CO_2 by forming sodium carbonate:

$$CO_2 + 2NaOH \longrightarrow Na_2CO_3 + H_2O$$

Monitoring NaOH concentration helps determine CO_2 uptake efficiency.

Using your answer from **(ii)**, calculate the **maximum mass of CO_2** that could react with the sodium hydroxide in the 25.0 cm^3 sample.

	HCl Reagent x	NaOH Reagent y
Molarity	0.100	0.0944
Volume	23.6 cm^3	25 cm^3
No. of moles	0.00236	.00236
Ratio	1	1

$$\text{moles} = \frac{0.100 \times 23.6}{1000} = 0.00236 \text{ mol}$$

HCl NaOH

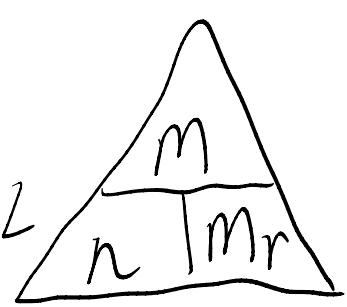
1 : 1

0.00236 0.00236

$$\frac{0.00236 \times 1000}{25}$$

$$= 0.0944 \text{ m/L}$$

$m = 0.0944 \times 40 = 3.776 \text{ g/L}$



2026, Sample paper 4, Q2, (a-c)

Question 2



A student titrated 25.0 cm^3 of 0.10 M NaOH with HCl of unknown concentration, using a pH meter.

(a) Write the balanced chemical equation.

$NaOH + HCl \rightarrow H_2O + NaCl$

(b) Calculate the moles of NaOH present.

$$\text{moles} = \frac{25 \times 0.10}{1000} = 0.0025 \text{ mol}/25 cm^3$$

(c) If the average titre was 24.8 cm^3 of HCl, calculate the concentration of HCl in mol L^{-1}.

$$\frac{0.0025 \times 1000}{24.8} = 0.1008 \text{ m/L}$$

	NaOH Reagent x	HCl Reagent y
Molarity	0.10	
Volume	25 cm^3	24.8 cm^3
No. of moles	.0025	0.0025
Ratio	1	1

TO DETERMINE THE CONCENTRATION OF A SOLUTION OF AMMONIA USING A STANDARDISED HCl SOLUTION

Purpose:

- To determine the exact concentration of a solution of ammonia by titrating it against a standardised hydrochloric acid solution of known concentration.

Intro:

- The hydrochloric acid solution was previously standardised using a standard sodium carbonate solution. It can now be used to determine the exact concentration of an ammonia solution.

Balanced equation:

Key Info:

- Type of titration: Strong acid and weak base titration.
- Solution in the burette: Standardised hydrochloric acid of known concentration.
- Solution in the conical flask: 25.0 cm^3 of ammonia solution of unknown concentration.
- Indicator: Methyl orange.
- Initial colour: Yellow.
- End point colour: Red/pink.
- Mole ratio: HCl : NH_3 = 1 : 1.
- Aqueous ammonia: A solution of ammonia in water is sometimes called ammonium hydroxide because NH_4^+ ions and OH^- ions are present in the solution.

Apparatus	Chemicals
25.0 cm^3 pipette and pipette filler. Burette and burette funnel. Conical flask. Retort stand and clamp. Dropping pipette. White tile. Wash bottle containing deionised water.	Dilute ammonia solution of unknown concentration. Standardised hydrochloric acid solution of known concentration. Methyl orange indicator. Deionised water.

Alistair Jack: Bruce College

Procedure:

- Rinse the pipette with deionised water followed by some of the ammonia solution.
  -**Purpose**: This prevents water remaining in the pipette from diluting the ammonia solution.
- Rinse the burette with deionised water followed by some of the standardised hydrochloric acid solution.
  -**Purpose**: This prevents water remaining in the burette from diluting the hydrochloric acid.
- Use the pipette to transfer 25.0 cm^3 of ammonia solution into a conical flask that has been rinsed with deionised water.
  -**Purpose**: The pipette delivers an accurate fixed volume of ammonia solution.
- Add a few drops of methyl orange indicator to the ammonia solution.
  -**Purpose**: Methyl orange shows when the end point has been reached.
- Fill the burette with the standardised hydrochloric acid solution and adjust the bottom of the meniscus to the zero graduation mark.
  Purpose: This allows the volume of hydrochloric acid added to be measured accurately.
- Place the conical flask on a white tile and titrate the ammonia solution against the hydrochloric acid using the technique described in the first experiment.
  -**Purpose**: The white tile makes the indicator colour change easier to observe.
- Carry out one rough titration followed by at least two accurate titrations that agree within 0.1 cm^3.

Observations:

- The methyl orange indicator is yellow in the ammonia solution.
- At the end point, the indicator changes from yellow to red (pink).

Explanation:

- As hydrochloric acid is added, it reacts with the ammonia in the conical flask.
- At the end point, the solution in the conical flask becomes acidic.
- Methyl orange changes from yellow to red (pink).

A student carried out a titration to determine the concentration of a dilute ammonia solution using a standardised hydrochloric acid solution.

(a) Name the indicator used in this titration.

(b) State the colour of the indicator:

(i) Before hydrochloric acid is added.

(ii) At the end point.

(c) Describe how the burette should be prepared and correctly set up before beginning the titration.

(d) Why should the funnel be removed from the burette before taking the initial reading?

(e) Why is the burette read at eye level?

(f) Why is the conical flask placed on a white tile?

(g) What is meant by stating that the hydrochloric acid solution has been standardised?

(h) Write the balanced equation for the reaction between hydrochloric acid and ammonia.

A student pipetted 25.0 cm^3 of the ammonia solution into a conical flask. The ammonia required 23.60 cm^3 of a 0.100 mol/L hydrochloric acid solution for complete reaction.

Using the balanced equation, calculate:

(i) The number of moles of hydrochloric acid used.

(ii) The number of moles of ammonia present in 25.0 cm^3 of the solution.

(iii) The concentration of the ammonia solution in mol/L

(iv) The concentration of the ammonia solution in g/L

TO DETERMINE THE CONCENTRATION OF ETHANOIC ACID IN VINEGAR

Intro:

- To determine the concentration of ethanoic acid, CH_3COOH, in a sample of vinegar by titrating the diluted vinegar against a standard solution of sodium hydroxide of known concentration.

Key Info:

- Vinegar contains ethanoic acid, **CH_3COOH**.
- Ethanoic acid is a weak acid.
- Sodium hydroxide is a strong base.
- The sodium hydroxide solution has a known concentration.
- The vinegar has an unknown concentration.
- The vinegar is diluted by a factor of x (will be told in the question) before the titration.
- **Phenolphthalein** is used as the indicator.
- Phenolphthalein changes from **pink to colourless** at the end point.
- Ethanoic acid and sodium hydroxide react in a **1:1** mole ratio.

Balanced equation:

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

Procedure:

A. Diluting the Vinegar

- Rinse a 25 cm^3 pipette with deionised water and then with a small amount of vinegar.
  -**Purpose**: Rinsing with vinegar prevents any water remaining in the pipette from diluting the vinegar sample.
- Use the pipette to transfer two 25 cm^3 samples of vinegar into a 250 cm^3 volumetric flask.
  -**Purpose**: This places exactly 50 cm^3 of vinegar in the volumetric flask.
- Add deionised water until the bottom of the meniscus is level with the graduation mark.
  -**Purpose**: This makes the total volume of the solution exactly 250 cm^3.
- Stopper the volumetric flask and invert it several times.
  -**Purpose**: This ensures that the vinegar and water are mixed thoroughly.

Alistair Jack: Bruce College

B. Carrying Out the Titration

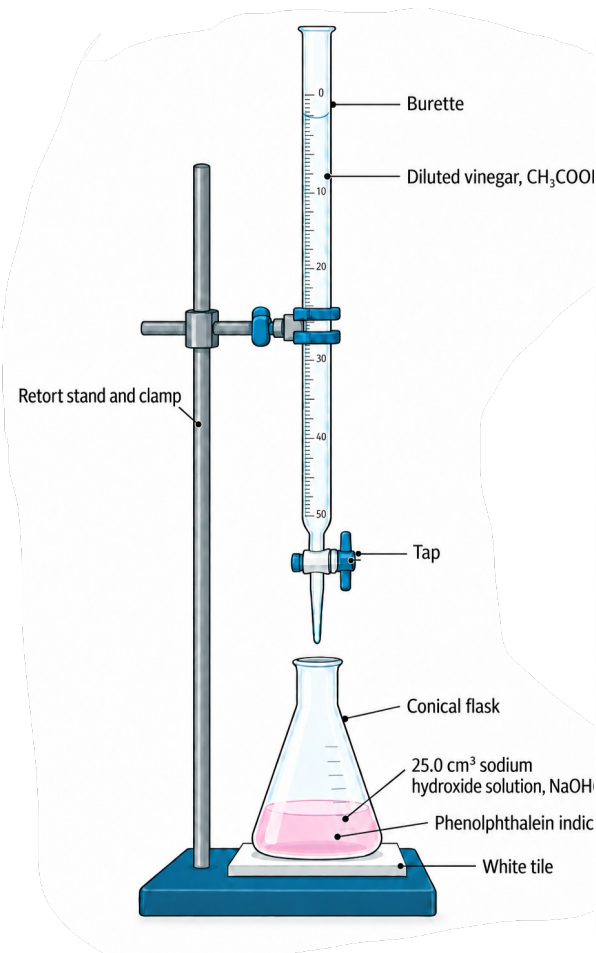
- Rinse the pipette with deionised water and then with the standard sodium hydroxide solution.
  -**Purpose**: This prevents any remaining water from diluting the sodium hydroxide solution.
- Use the pipette to transfer 25 cm^3 of sodium hydroxide solution into a conical flask that has been rinsed with deionised water.
  -**Purpose**: This transfers an accurate volume of sodium hydroxide into the flask.
- Add approximately three drops of phenolphthalein indicator to the conical flask.
  -**Purpose**: The indicator is used to detect the end point of the titration.
- Rinse the burette with deionised water and then with some diluted vinegar.
  -**Purpose**: This prevents any remaining water from diluting the vinegar.
- Fill the burette with diluted vinegar using a small funnel. Remove the funnel and record the initial burette reading.
  -**Purpose**: Removing the funnel prevents additional drops from entering the burette after the initial reading has been recorded.
- Place the conical flask on a white tile beneath the burette.
  -**Purpose**: The white background makes the colour change easier to observe.
- Add the diluted vinegar from the burette while continuously swirling the conical flask.
  -**Purpose**: Swirling ensures that the vinegar and sodium hydroxide mix completely.
- Near the end point, add the diluted vinegar one drop at a time until the pink colour just disappears.
  -**Purpose**: Adding the vinegar slowly prevents the end point from being exceeded.
- Record the final burette reading and calculate the volume of diluted vinegar added.
  -**Purpose**: This volume is the titre.
- Carry out one rough titration followed by accurate titrations until two titres agree within 0.10 cm^3.
  -**Purpose**: These concordant titres improve the reliability of the result.

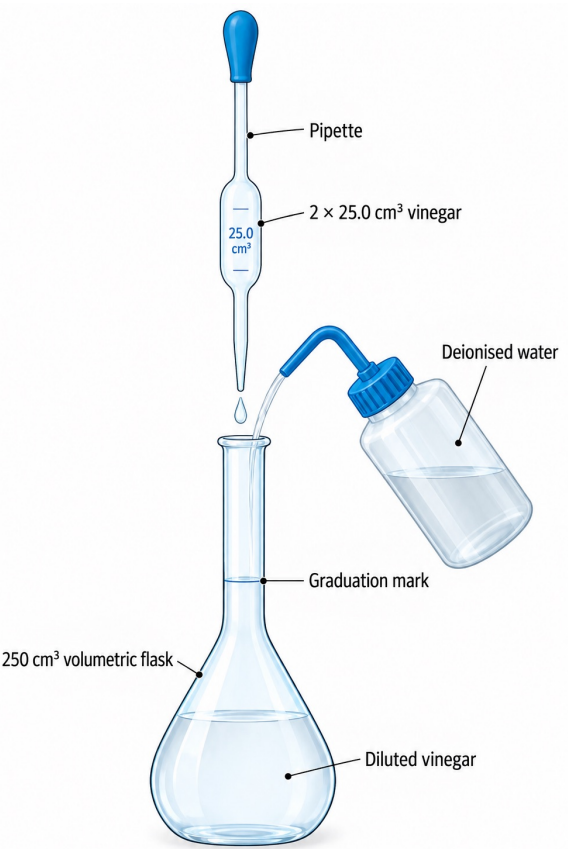
Observations:

- The sodium hydroxide solution turns pink when phenolphthalein is added.
- As diluted vinegar is added, the pink colour becomes paler.
- At the end point, the solution changes from pink to colourless.

Explanations:

- Phenolphthalein is pink in an alkaline solution, so it is pink when added to sodium hydroxide.
- As the diluted vinegar is added, ethanoic acid reacts with and neutralises the sodium hydroxide.
- At the end point, the sodium hydroxide has been neutralised and the phenolphthalein changes from pink to colourless.
- Phenolphthalein is suitable because this is a titration between a weak acid and a strong base.
- The vinegar is diluted because it is much more concentrated than the sodium hydroxide solution. Without dilution, a large volume of sodium hydroxide would be required to neutralise it. Dilution produces a suitable titre and reduces the percentage error.





Question 4

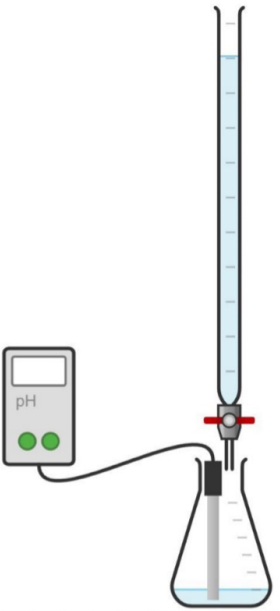
A student carried out an experiment to investigate the concentration of ethanoic acid in a bottle of vinegar.

A 25.0 cm^3 portion of the vinegar was diluted to exactly 100.0 cm^3.

The diluted vinegar solution was used to fill the burette.

The student dissolved 1.8 g of sodium hydroxide (NaOH) in enough water to make 500.0 cm^3 of solution. 25.0 cm^3 of this solution was placed in a conical flask.

The titration reaction was monitored using a pH meter.



(i) Describe the procedure for preparing the burette and then filling it with the diluted vinegar solution.

The student titrated the diluted vinegar solution against 25.0 cm^3 portions of the NaOH solution.
The reaction is described by the following balanced chemical equation:

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

The student carried out one approximate titration and two accurate titrations.
In the table below, the student recorded the volume of the diluted vinegar solution that was needed to neutralise the sodium hydroxide.

titration	approximate	accurate 1	accurate 2
volume (cm^3)	11.1	10.9	10.8

$$\text{moles} = \frac{25 \times 0.09}{1000}$$

$$= 0.00225 \text{ mol} / 25 \text{ cm}^3$$

(viii) Calculate the number of moles of ethanoic acid in each cm^3 of the diluted vinegar solution.

1 cm^3 per cm^3

$0.00002073 \text{ mol} / 1 \text{ cm}^3$

(ix) Calculate the concentration of ethanoic acid in the undiluted vinegar in moles per litre.

(x) Calculate the concentration of ethanoic acid in the undiluted vinegar in % w/v.

(xi) The NaOH solution used in this titration should have been standardised before it was used. What is meant by standardisation?

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

2024, Q1

A student carried out an experiment to determine the concentration of ethanoic acid in vinegar. A 10.0 cm^3 portion of the vinegar was first diluted to exactly 50.0 cm^3. The diluted vinegar solution was titrated against a 0.09 M sodium hydroxide solution in a conical flask. On average, 12.4 cm^3 of the *diluted* vinegar were required to neutralise 25.0 cm^3 of the **NaOH** solution.

The titration reaction is described by the following balanced chemical equation:

$$\mathbf{CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O}$$

(*a*) (*i*) Name the piece of apparatus used to measure accurately the 10.0 cm^3 portion of the original vinegar.

(*ii*) Describe how this piece of apparatus was rinsed before use.

(*iii*) Describe a suitable method to dilute the 10.0 cm^3 portion of the original vinegar to exactly 50.0 cm^3. (15)

(*b*) (*i*) Calculate the volume of *undiluted* vinegar which would be required to neutralise 25.0 cm^3 of the 0.09 M **NaOH** solution.

(*ii*) Outline an advantage of diluting the vinegar before carrying out the titration. (9)

(*c*) (*i*) Name a suitable indicator for this titration.

(*ii*) State the colour change observed at the end point. (9)

(*d*) (*i*) Calculate the number of moles of sodium hydroxide in each 25.0 cm^3 portion.

(*ii*) Calculate the number of moles of ethanoic acid in each cm^3 of the *diluted* vinegar.

(*iii*) Calculate the concentration of ethanoic acid in the original vinegar in moles per litre.

(*iv*) Calculate the concentration of ethanoic acid in the original vinegar in % (w/v). (17)

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

2016, Q1

1. To determine the concentration of ethanoic acid in a sample of vinegar, 25.0 cm^3 of the vinegar were diluted to 250 cm^3 and then the diluted vinegar was titrated with a previously standardised solution which contained 1.20 g of sodium hydroxide in 500 cm^3 of solution. On average, 18.75 cm^3 of the diluted vinegar were required to neutralise 25.0 cm^3 of this sodium hydroxide solution.

The equation for the titration reaction is:

$$\mathbf{CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O}$$

(*a*) Describe in detail the procedures involved in measuring a 25.0 cm^3 sample of the vinegar and diluting it using deionised water to exactly 250 cm^3. (15)

(*b*) Name a suitable indicator for this titration.

What colour change is observed at the end point? (9)

(*c*) Calculate

(*i*) the number of moles of sodium hydroxide in each 25.0 cm^3 portion,

(*ii*) the number of moles of ethanoic acid per cm^3 of *diluted* vinegar. (12)

(*d*) Find the concentration of ethanoic acid in the *original* vinegar

(*i*) in terms of moles per litre,

(*ii*) as a percentage (w/v). (9)

(*e*) Starting with a primary standard solution made from anhydrous sodium carbonate, what two titrations are required to standardise a sodium hydroxide solution? (5)

Volumetric analysis (Redox)

- In previous titrations, we used acid and base reactions to determine the concentration of a solution. In redox titrations, we use oxidation and reduction reactions instead.
- These reactions involve the transfer of electrons between an oxidising agent and a reducing agent.

- Oxidation is the loss of electrons.
- Reduction is the gain of electrons.
- An oxidising agent accepts electrons and is reduced.
- A reducing agent donates electrons and is oxidised.

- The purpose of a redox titration is to determine the concentration of an oxidising agent or a reducing agent by reacting it with a solution of accurately known concentration.
- Lets begin with potassium permanganate, $KMnO_4$, also called potassium manganate(VII). **It is a strong oxidising agent with a purple colour.**

Potassium Permanganate

- Potassium permanganate, also called potassium manganate(VII), has the formula $KMnO_4$. It is a purple solid that dissolves in water to form a purple solution.

- The permanganate ion, MnO_4^-, acts as the **oxidising agent.**

Why must it be standardised?

- Potassium permanganate is **not a primary standard** because it **cannot readily be obtained in a pure state.**

- Its solutions also **decompose over time**, particularly when exposed to sunlight and heat, causing their concentration to change.

- Therefore, a potassium permanganate solution must be standardised by titration against a suitable standard solution to determine its exact concentration.

- **Ammonium Iron(II) sulfate** is used as the primary standard for redox titrations.

Indicator and Burette Readings:

- Potassium permanganate acts as its own indicator, it is self indicating, so no separate indicator is required.

- During the titration, the added purple permanganate ions are reduced to effectively colourless Mn^{2+} ions. The purple colour disappears as long as there is reducing agent left in the conical flask.

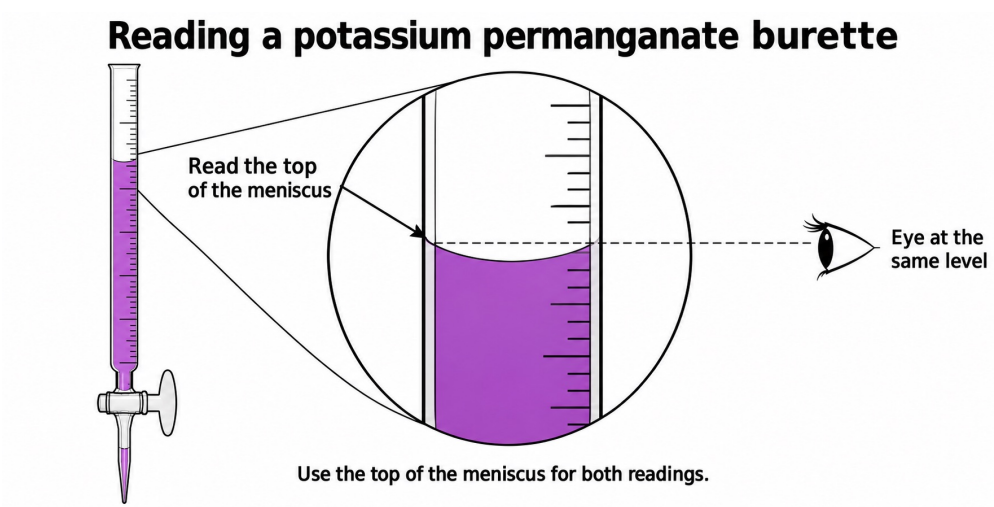
- Once all the reducing agent has reacted, the next drop of potassium permanganate produces a permanent pale pink colour that remains after swirling.

- End point: the **first permanent pale pink colour.**
- The pale pink colour is caused by a slight excess of unreacted permanganate ions.

Reading the burette:

- The intense purple colour of potassium permanganate makes the bottom of the meniscus difficult to see.

- Read the **top of the meniscus at eye level**, using the same method for both the initial and final readings.



Reaction Between Permanganate and Iron(II) Ions:

- Potassium permanganate oxidises Fe^{2+} ions to Fe^{3+} ions in acidic solution.

Half equations:

- Reduction: Permanganate ions accept electrons.

- Oxidation: Iron(II) ions lose electrons.

- Each permanganate ion accepts five electrons, while each Fe^{2+} ion loses one electron. Therefore, multiply the iron half equation by five:

- Overall balanced equation: Adding the half equations and cancelling the electrons gives:

- This **1 : 5** mole ratio is used in the titration calculations.

Ammonium Iron(II) Sulfate

Formula and purpose

- Ammonium iron(II) sulfate has the formula: $\mathbf{(NH_4)_2SO_4{\cdot}FeSO_4{\cdot}6H_2O}$
- It provides the Fe^{2+} ions needed to standardise the potassium permanganate solution.

Why is it suitable as a primary standard?

- It can be obtained in a high degree of purity.
- Its crystals are stable in air.
- It does not readily lose its water of crystallisation.
- Its high molar mass, 392.1 g/mol (4 significant figures), reduces the percentage uncertainty when weighing a sample.

Why is ammonium iron(II) sulfate used instead of hydrated or anhydrous iron(II) sulfate?

- Ammonium iron(II) sulfate is used because it can be obtained in high purity and its crystals are stable in air, making it suitable as a primary standard.
- Hydrated iron(II) sulfate, $FeSO_4 \cdot 7H_2O$, can be oxidised by oxygen in the air and lose water of crystallisation.
- Anhydrous iron(II) sulfate, $FeSO_4$, absorbs moisture from the air.

Why is dilute sulfuric acid added during preparation?

Ammonium iron(II) sulfate is dissolved in water containing dilute sulfuric acid because:

- The acidic conditions prevents the oxidation of Fe^{2+} ions to Fe^{3+} ions by oxygen in the air.
- supplies the H^+ ions needed to reduce MnO_4^- to Mn^{2+}.
- Hydrochloric acid cannot be used because its chloride ions can be oxidised to chlorine by potassium permanganate, consuming extra titrant and giving an inaccurate result.
- Nitric acid cannot be used because it is itself an oxidising agent and can oxidise Fe^{2+} ions to Fe^{3+} ions before they react with potassium permanganate.

The Effect of Air and Moisture on Primary Standards

- To prepare a standard solution, the mass weighed must represent an accurately known amount of the substance. Some substances lose water of crystallisation or absorb moisture from the air, changing their composition or mass. These behaviours are described as efflorescence, hygroscopic behaviour and deliquescence.

- These terms are commonly asked in past examination papers, so you should know their definitions and examples and explain why they make a substance unsuitable as a primary standard.

Examples:

Efflorescence:

- Efflorescence is the spontaneous loss of water of crystallisation from a hydrated crystalline salt.

Deliquescence:

- Deliquescence is the spontaneous process by which a substance absorbs moisture from the atmosphere to the extent that it dissolves into a liquid solution.
- Remember Sodium Hydroxide in the strong acid strong base titration.

Hygroscopic substances:

- A substance is said to be hygroscopic if it has the ability to spontaneously absorb moisture from air without dissolving into a liquid solution.

Exam note:

- Know the difference between deliquescence and hygroscopic behaviour. Both involve absorbing moisture from the air, but a deliquescent substance absorbs enough moisture to dissolve into a liquid solution, while a hygroscopic substance does not dissolve.

TO PREPARE A STANDARD SOLUTION OF AMMONIUM IRON (II) SULFATE AND TO USE THIS SOLUTION TO STANDARDISE A SOLUTION OF POTASSIUM PERMNAGANATE

Intro:

- In this experiment, a standard solution of ammonium iron(II) sulfate is prepared by dissolving an accurately weighed mass in water containing dilute sulfuric acid and making it up to a known volume.
- This solution is then used to determine the exact concentration of potassium permanganate by titration.

Key Info:

- Primary standard: Ammonium iron(II) sulfate, $(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$. (392.1 g/mol)
- Burette: Potassium permanganate solution of unknown concentration.
- Conical flask: A measured volume of standard ammonium iron(II) sulfate solution with excess dilute sulfuric acid.
- Indicator: No separate indicator is needed, potassium permanganate acts as its own indicator
- End point: The first permanent pale pink colour that remains after swirling.
- Burette reading: Read the top of the meniscus at eye level.

Balanced equation:

Procedure:

- **Some ammonium iron(II) sulfate is accurately weighed out on a clock glass using a laboratory balance.**
  -This gives an accurately known mass of ammonium iron(II) sulfate.
- **Using a graduated cylinder, dilute sulfuric acid is added to a beaker containing some deionised water.**
  -The dilute sulfuric acid helps to prevent Fe^{2+} ions from being oxidised to Fe^{3+} ions by oxygen in the air.
- **The ammonium iron(II) sulfate is transferred to the beaker.**
  -The solid is added to the acidified water so that it can dissolve.
- **The mixture is stirred until all the ammonium iron(II) sulfate crystals dissolve.**
  -This ensures that all of the solid has gone into solution.
- **The solution is transferred into a clean volumetric flask using a funnel.**
  -The volumetric flask is used to make the solution up to an accurate final volume.
- **All washings are transferred into the volumetric flask.**
  -This ensures that no ammonium iron(II) sulfate is left behind in the beaker, glass rod, or funnel.
- **The bottom of the meniscus is brought up to the mark with deionised water.**
  -This gives the solution its exact final volume.
- **The stoppered flask is inverted about 20 times.**
  -This makes sure the solution is completely mixed and has the same concentration throughout.

- **The titration is carried out in the usual manner.**
- **The potassium permanganate solution is slowly added from the burette into the conical flask while the flask is continuously swirled.**
- **The level of potassium permanganate in the burette is read from the top of the meniscus.**
  -Potassium permanganate is a very dark purple solution, so the bottom of the meniscus cannot be seen clearly.
- **Using a graduated cylinder, some dilute sulfuric acid is added to the conical flask.**

Why Excess Sulfuric Acid Is Needed.

- Excess sulfuric acid is needed to provide enough H^+ ions.
- The H^+ ions allow the MnO_4^- ions to be reduced to Mn^{2+} ions.
- $MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$
- If there is no acid, or not enough acid, a dark brown precipitate of MnO_2 forms during the titration. (In the conical flask)

- **The titration is continued until a faint permanent pink colour remains in the conical flask.**
  -This is the endpoint.

Observations:

- Potassium permanganate is purple.
- The solution in the conical flask is decolourised as potassium permanganate is added.
- Near the endpoint, the pink colour disappears more slowly.
- At the endpoint, a faint permanent pink colour remains.
- If insufficient sulfuric acid is added, a dark brown precipitate of MnO_2 may form.

Autocatalysis:

- The first few drops of potassium permanganate are decolourised slowly.
- The more potassium permanganate added, it decolourised more quickly.
- This is because the reaction is catalysed by the Mn^{2+} ions produced in the reaction.
- As more Mn^{2+} ions are formed, the reaction speeds up.
- Autocatalysis: a reaction where one of the products acts as a catalyst for the reaction.

Practice questions:

- A student dissolved 9.803 g of ammonium iron(II) sulfate, $(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$, in water containing dilute sulfuric acid and made the solution up to 250 cm^3 in a volumetric flask. A 25.0 cm^3 portion of this solution was titrated with potassium permanganate in the presence of excess dilute sulfuric acid. The average titre was 24.8 cm^3.
- Calculate the concentration of the potassium permanganate solution in moles per litre.

2023, Q1

1. (*a*) The reagent **$(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$** (hydrated ammonium iron(II) sulfate) was used to prepare a standard solution.

State two properties of this substance that make it suitable for use as a primary standard. (8)

(*b*) (*i*) Describe how you would prepare exactly 250 cm^3 of a standard solution containing 9.31 g of **$(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$** (hydrated ammonium iron(II) sulfate) that was supplied on a clock glass.

(*ii*) Why was dilute sulfuric acid added during the preparation of this standard solution? (15)

This standard solution of **Fe^{2+}** ions was used to determine by titration the concentration of a potassium manganate(VII) solution. The balanced equation for the titration reaction is:

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$$

In each titration a little dilute sulfuric acid was added to a 25.0 cm^3 portion of the **Fe^{2+}** ion solution in a conical flask before adding the potassium manganate(VII) solution from a burette. On average, 22.6 cm^3 of the **$KMnO_4$** solution were required for complete reaction with 25.0 cm^3 of the solution of the **Fe^{2+}** ions in excess acid.

(*c*) (*i*) How was the end point of each titration detected?

(*ii*) If insufficient acid was present in the conical flask, what would have been observed as the **$KMnO_4$** solution was added from the burette to the **Fe^{2+}** ion solution? Explain. (9)

(*d*) Calculate

(*i*) the concentration of **$(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$** in the standard solution in moles per litre,

(*ii*) the number of moles of **Fe^{2+}** ion in a 25.0 cm^3 portion of the standard solution,

(*iii*) the number of moles of potassium manganate(VII) reduced to **Mn^{2+}** by this quantity of **Fe^{2+}** ion,

(*iv*) the concentration of the potassium manganate(VII) solution in moles per litre. (18)

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		

1. A solution of **$(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$** (hydrated ammonium iron(II) sulfate), used as a primary standard, was prepared by dissolving 8.82 g of the crystals in dilute sulfuric acid and making the solution up to exactly 250 cm^3 with deionised water. This standard solution was titrated in 25.0 cm^3 portions, to which some more dilute sulfuric acid had been added, with a solution of potassium manganate(VII) (**$KMnO_4$**). On average, 20.45 cm^3 of the potassium manganate(VII) solution were required for complete oxidation of the **Fe^{2+}** ions.

The equation for the titration reaction is:

$$MnO_4^- + 5Fe^{2+} + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$$

(*a*) (*i*) Explain the underlined term.

(*ii*) Give two essential properties of a primary standard for volumetric analysis. (11)

(*b*) Explain why it was necessary

(*i*) to use dilute sulfuric acid when preparing the standard solution from hydrated ammonium iron(II) sulfate,

(*ii*) to add more dilute sulfuric acid to each 25.0 cm^3 portion of the standard solution used in the titrations. (9)

(*c*) Describe fully the procedure used *during the titrations* to ensure the accuracy of the end point.

What observation indicates that the correct end point has been reached? (15)

(*d*) Calculate

(*i*) the number of moles of **Fe^{2+}** ion in a 25.0 cm^3 portion of the standard solution,

(*ii*) the number of moles of potassium manganate(VII) required to oxidise this quantity of **Fe^{2+}** ion,

(*iii*) the molarity of the potassium manganate(VII) solution. (15)

	Reagent x	Reagent y
Molarity		
Volume		
No. of moles		
Ratio		