

A Level Chemistry OCR

1. Development of Practical Skills in Chemistry

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1.1 Physical Chemistry Practical

1.1.1 Moles Determination

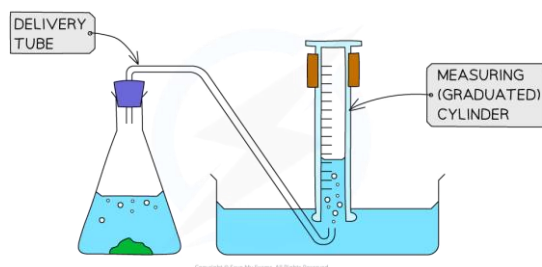
PAG 1 Moles Determination

- There are a series of suggested practicals for PAG 1 - Moles Determination PAG 1.1 -
 - Determination of the composition of copper(II) carbonate basic
 - PAG 1.2 - Determination of the relative atomic mass of magnesium
 - PAG 1.3 - Determination of the formula of magnesium oxide
- All three suggested practicals require the number of moles to be calculated from experimental data to determine the final answer This can be done by:
 - Measuring gas volumes
 - In these experiments, a reaction occurs which produces a gas
 - The number of moles of gas produced is calculated
 - The stoichiometric ratio of the chemical reaction is then used to determine the final answer

For this style of experiment, the final answer could be the composition of copper(II) carbonate or the relative atomic mass of magnesium Other practicals can be used in place of the suggested practicals so you wont be required to apply your knowledge of these specific reactions to other examples in questions
 - Measuring changes in mass
 - In these experiments, a reaction occurs which produces an increase or decrease in mass
 - The suggested practical that goes with this is determining the formula of magnesium oxide
 - oxide
 - A known mass of magnesium is combusted and the mass of the magnesium oxide product is measured
 - The number of moles of magnesium and oxygen are calculated to determine the ratio of each element in the formula of the magnesium oxide
 - A similar practical can be performed on hydrated salts, e.g. $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$, by heating them to a constant mass and using the decreases in mass to calculate the number of water of crystallisations - but this is not a suggested practical

PAG 1.1 - Determination of the composition of copper(II) carbonate basic

1. Set up the apparatus using a measuring cylinder sitting in water bath connected to a conical flask
2. Measure a known mass of $\text{CuCO}_3 \cdot \text{Cu(OH)}_2$ and add it to the conical flask
3. Add an excess of sulfuric acid into the conical flask and immediately insert the stopper. The gas produced will collect in the measuring cylinder
4. Record the final volume of carbon dioxide in the measuring cylinder



Equipment set up for gas collection by displacement



Exam Tip

As an alternative a gas syringe could be set up to measure the volume of gas produced

Calculations

- We can find the number of moles, n , of CO_2 produced using the measured volume, v , with the following equation:

$$n = \frac{V(\text{dm}^3)}{24.0} \quad \text{or} \quad n = \frac{V(\text{cm}^3)}{24000}$$

- Remember:** 24.0 dm^3 is the volume of 1 mole of gas
- This calculation is only correct if the reaction is completed at room temperature (20°C) and pressure (101 kPa or 1 atmosphere)
- If the calculation is completed under conditions that are not RTP, then a rearranged form of the ideal gas equation should be used:

$$n = \frac{PV}{RT}$$

- We can use the overall equation for the reaction to figure out the number of moles of copper carbonate that reacted



- Molar ratio for $\text{CuCO}_3 : \text{CO}_2$ is 1:1
- The mass, m , of CuCO_3 can then be determined using:
 - $m = n \times M_r$
- As our original sample contained $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$ we need to determine the percentage by mass of CuCO_3 present in the original sample

Source of errors

- Some carbon dioxide may escape from the conical flask before inserting the bung This can be
 - avoided by inserting the bung as soon as the acid is poured into the conical flask
- Some of the copper carbonate may not react
 - Swirl the conical flask to ensure the contents are sufficiently mixed and the reaction goes to completion

Carbon dioxide may dissolve in the water

- A gas syringe can be used to measure the volume of CO₂ instead
- Some copper carbonate may remain on the weighing apparatus and not be transferred to the conical flask
 - Weigh the weighing apparatus with and without and copper carbonate on it
 - The difference between these gives the exact amount added to the conical flask

PAG 1.2 - Determination of the relative atomic mass of magnesium

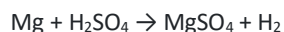
- This practical is completed in a similar way to PAG 1.1 - Determination of the composition of copper(II) carbonate basic in terms of equipment
- Set up the apparatus using a measuring cylinder sitting in water bath connected to a conical flask
- Measure a known mass of magnesium and add it to the conical flask
- Add an excess of sulfuric acid into the conical flask and immediately insert the stopper. The gas produced will collect in the measuring cylinder
- Record the final volume of hydrogen gas in the measuring cylinder

Calculations

- We can find the number of moles, n , of H₂ produced using the measured volume, v , with the following equation:

$$n = \frac{v}{24}$$

- **Remember:** 24 dm³ is the volume of 1 mole of gas
- We can use the overall equation for the reaction to figure out the number of moles of copper carbonate that reacted



- Molar ratio for Mg : H₂ is 1:1
- The mass, m , of Mg can then be determined using:
 - $m = n \times M_r$

Source of errors

- Some hydrogen may escape from the conical flask before inserting the bung This can be avoided by inserting the bung as soon as the acid is poured into the conical flask
- Some of the magnesium may not react
- Use magnesium powder instead of ribbon
 - Ensure an excess of acid is added
 - Swirl the conical flask to ensure the contents are sufficiently mixed and the reaction goes to completion
- Some magnesium may remain on the weighing apparatus and not be transferred to the conical flask
- Weigh the weighing apparatus with and without and magnesium on it The difference between these gives the exact amount added to the conical flask
-
-

? Exam Tip

- The relative atomic mass of magnesium cannot be completed by measuring the mass lost during the reaction
- This is because the mass lost will be relatively insignificant as it is hydrogen that will be being released

PAG 1.3 - Determination of the formula of magnesium oxide

- A known mass of magnesium is measured and placed in a crucible with a lid
- The magnesium is then heated to react with oxygen in the air
 - The lid of the crucible needs to be open enough to allow sufficient air in but closed enough to not lose the product The mass of the product is then measured

Calculations

- With the mass recordings, the number of moles of magnesium and the number of mass moles of oxygen can be calculated using moles = _____

$$\frac{M}{r}$$

- The molar ratio of magnesium : oxygen is then used to determine the formula of magnesium, For example, if the molar ratio was 2 magnesium : 3 oxygen then the formula would be Mg_2O_3

Sources of error

- Some magnesium oxide may escape from the crucible
 - This cannot be avoided but can be reduced by careful heating and careful placement of the crucible lid
- Some of the magnesium may not react
 - Using magnesium ribbon can be better than powder as once ignited the ribbon should continue to burn itself, while use of powder can lead to a loss a product due to the speed of the reaction as well as the potential for some of the magnesium to not react
 - If magnesium ribbon is used, it can be cleaned with sandpaper / glasspaper to remove any oxide coating already formed
- Some magnesium may remain on the weighing apparatus and not be transferred to the crucible
 - Weigh the weighing apparatus with and without magnesium on it
 - The difference between these gives the exact amount added to the crucible

1.1.2 Acid-Base Titration

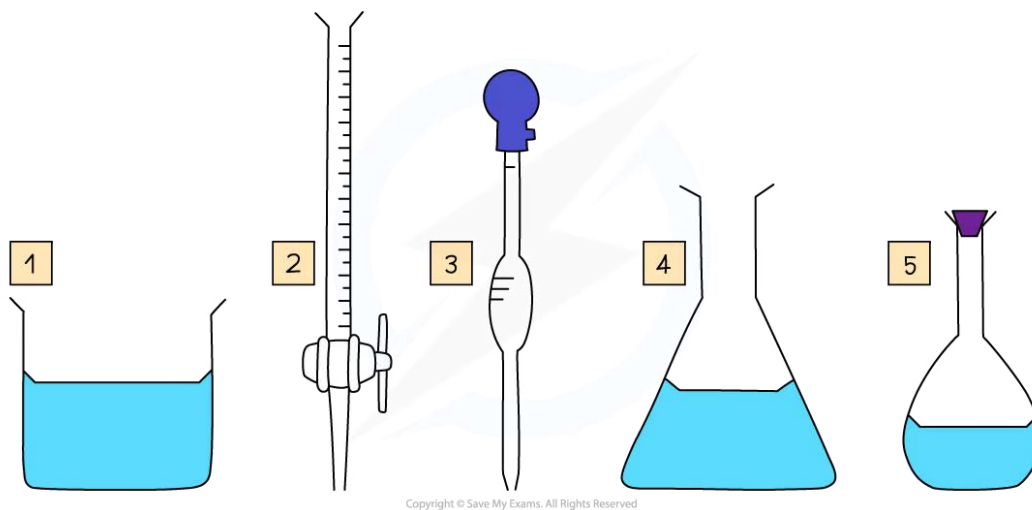
PAG 2 Acid-base titration

- There are a series of suggested practicals for PAG 2 - Acid-base titration
 - **PAG 2.1: Determination of the concentration of hydrochloric acid** This involves making a
 - standard solution of sodium hydrogencarbonate and titrating this against a solution of hydrochloric acid in order to work out the concentration of the acid.

- **PAG 2.2: Determination of the molar mass of an acid**
 - This involves making a solution of an unknown hydrated acid (e.g. citric acid monohydrate), then titrating this against a sodium hydroxide solution of known concentration. This allows the molar mass of the acid to be determined, and with additional information, the formula of the acid.
- **PAG 2.3: Identification of an unknown carbonate**
 - This involves making a solution of an unknown Group 1 carbonate and then titrating this solution against hydrochloric acid of a known concentration to determine the molar mass of the carbonate and hence the identity of the metal.
- All three suggested practicals use volumetric analysis, which requires making standard solutions and completing titration experiments.
 - The main difference between the suggested practicals is how the titration results are processed

Volumetric Analysis

- **Volumetric analysis** is a process that uses the volume and concentration of one chemical reactant (**a volumetric solution**) to determine the concentration of another unknown solution
- The technique most commonly used is a **titration**
- The volumes are measured using two precise pieces of equipment, a **volumetric** or **graduated pipette** and a **burette**
- Before the titration can be done, the standard solution must be prepared Specific apparatus
- must be used both when preparing the standard solution and when completing the titration, to ensure that volumes are measured precisely



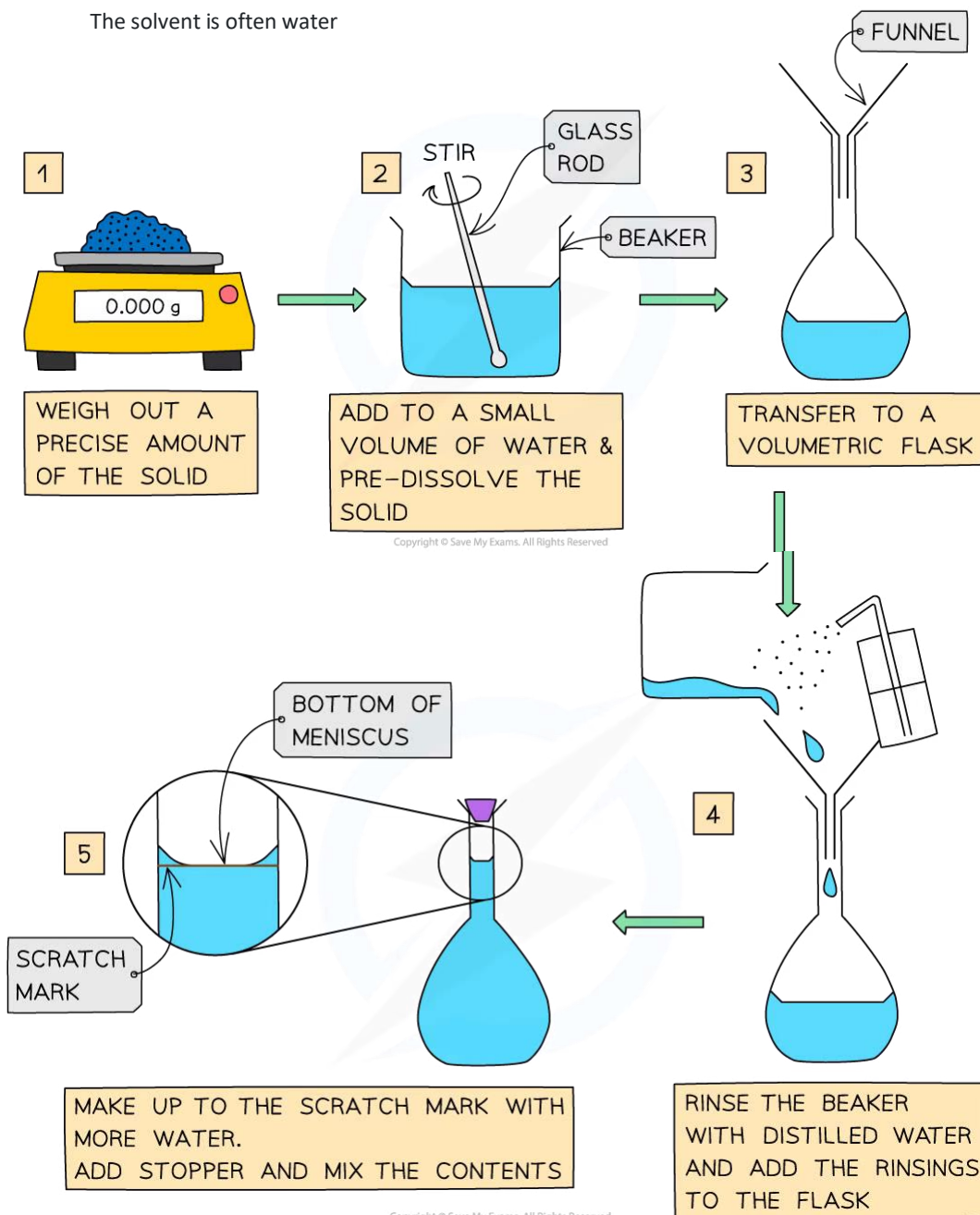
Some key pieces of apparatus used to prepare a volumetric solution and perform a simple titration

1. Beaker
2. Burette
3. Volumetric Pipette
4. Conical Flask
5. Volumetric Flask

Making a Volumetric Solution

- Chemists routinely prepare solutions needed for analysis, whose concentrations are known precisely
- These solutions are termed **volumetric solutions** or **standard solutions** They are made as accurately
- and precisely as possible using three decimal place balances and volumetric flasks to reduce the impact of measurement uncertainties The steps are:
-

The solvent is often water



Volumes & concentrations of solutions

- The **concentration** of a solution is the amount of **solute** dissolved in a **solvent** to make 1 dm³ of **solution**
 - The solute is the substance that dissolves in a solvent to form a solution
 -
 -
 -
 -
 -

- mass per unit volume
- parts per million



Worked Example

Calculate the mass of sodium hydrogencarbonate, NaHCO_3 , required to prepare 250 cm^3 of a $0.200 \text{ mol dm}^{-3}$ solution

A **concentrated** solution is a solution that has a **high** concentration of solute A **dilute** solution is a solution with a **low** concentration of solute Concentration is usually expressed in one of three ways:

moles per unit volume

Answer:

Step 1: Find the number of moles of NaHCO_3 needed from the concentration and volume:

- number of moles = concentration (mol dm^{-3}) x volume (dm^3)
- $n = 0.200 \text{ mol dm}^{-3} \times 0.250 \text{ dm}^3$ $n =$
- **0.0500 mol**

Step 2: Find the molar mass of NaHCO_3

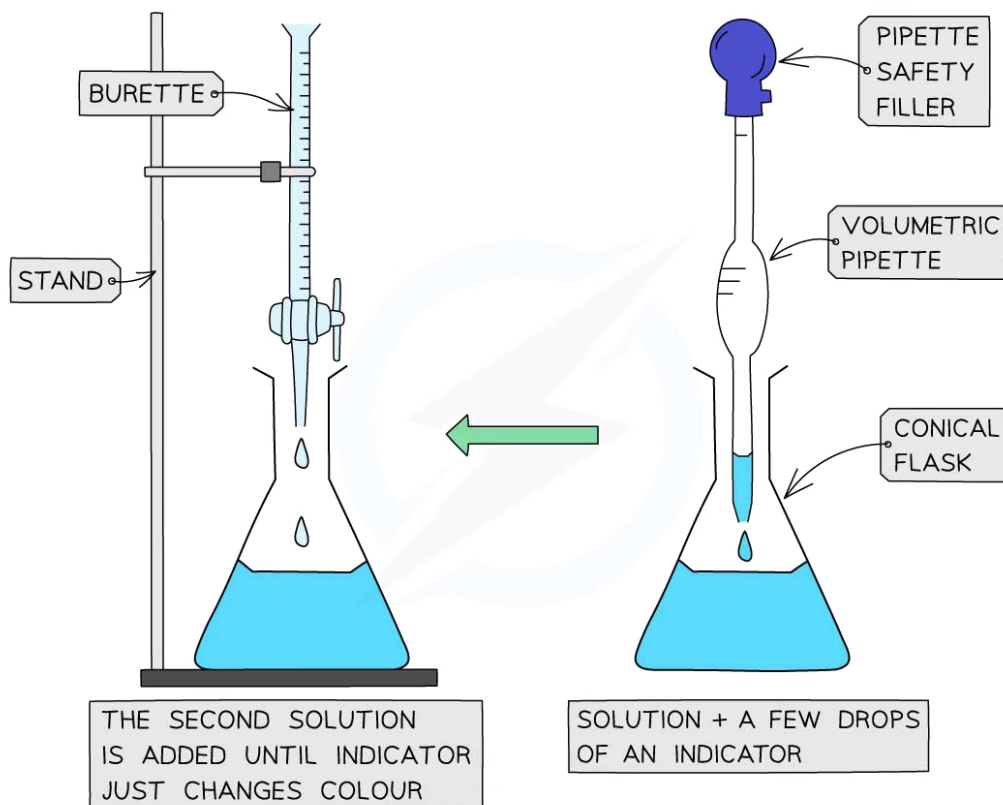
- $M_r = 23.0 + 1.0 + 12.0 + (16.0 \times 3) = 84.0 \text{ g mol}^{-1}$

Step 3: Calculate the mass of NaHCO_3 required

- mass = moles x molar mass mass = $0.0500 \text{ mol} \times$
- $84.0 \text{ g mol}^{-1} = \mathbf{4.2 \text{ g}}$

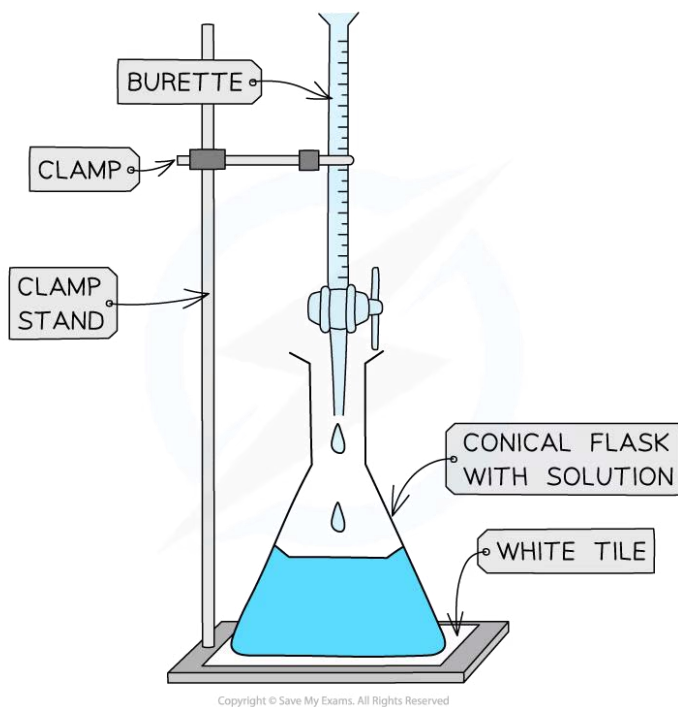
Performing the Titration

- The key piece of equipment used in the titration is the burette
- **Burettes** are usually marked to a precision of 0.10 cm^3
 - Since they are analogue instruments, the uncertainty is recorded to **half** the smallest marking, in other words to $\pm 0.05 \text{ cm}^3$
- The **end point** or **equivalence point** occurs when the two solutions have reacted completely and is shown with the use of an **indicator**



The steps in a titration

- A white tile is placed under the conical flask while the titration is performed, to make it easier to see the colour change



Titration

- The steps in a titration are:
 - Measuring a known volume (usually 20 or 25 cm³) of one of the solutions with a **volumetric pipette** and placing it into a **conical flask**
 - The other solution is placed in the **burette**
 - To start with, the burette will usually be filled to 0.00 cm³
 - A few drops of the **indicator** are added to the solution in the conical flask
 - The tap on the **burette** is carefully opened and the solution added, portion by portion, to the **conical flask** until the **indicator** starts to change colour
 - As you start getting near to the end point, the flow of the burette should be slowed right down so that the solution is added dropwise
 - You should be able to close the tap on the burette after one drop has caused the colour change
 - Multiple runs are carried out until **concordant** results are obtained
 - Concordant results are within 0.1 cm³ of each other

Recording and processing titration results

a **precision** of ± 0.05 cm³, the same as the **uncertainty**

	Rough	Run 1	Run 2	Run 3
Initial burette reading ± 0.05 ml	0.00	23.15	0.20	23.00
Final burette reading ± 0.05 ml	23.75	45.95	23.00	46.10
Volume delivered ± 0.10 ml	23.75	22.80 ✓	22.80 ✓	23.10

ALL RESULTS ARE RECORDED TO 2 DECIMAL PLACES INCLUDING ZERO READINGS

THE FINAL DIGIT IS 0 OR 5

DOUBLE THE UNCERTAINTY

THE ROUGH RESULT IS USUALLY FAR OVER THE END-POINT

THIS RESULT IS DISCARDED AS IT IS TOO HIGH

✓ = CONCORDANT RESULTS

USED TO CALCULATE THE AVERAGE

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Both the initial and final **burette** readings should be recorded and shown to

A typical layout and set of titration results

- The volume delivered (**titre**) is calculated and recorded to an **uncertainty** of ± 0.10 cm³

◦ The **uncertainty** is doubled, because two **burette** readings are made to obtain the **titre** ($V_{\text{final}} - V_{\text{initial}}$), following the rules for **propagation of**

- **Concordant** results are then averaged, and non-concordant results are discarded
- The appropriate calculations are then done

? Worked Example

PAG 2.1: Determination of the concentration of hydrochloric acid

25.0 cm³ of hydrochloric acid was titrated with a 0.200 mol dm⁻³ solution of sodium hydrogencarbonate, NaHCO₃.



Use the following results to calculate the concentration of the acid, to 3 significant figures.

	Rough	Run 1	Run 2	Run 3
Initial burette reading / cm ³ (±0.05 cm ³)	0.00	23.15	0.20	23.00
Final burette reading / cm ³ (±0.05 cm ³)	23.75	45.95	23.00	46.10
Volume delivered / cm ³ (±0.10 cm ³)	23.75	22.80	22.80	23.10

Answer

Step 1: Calculate the average titre from concurrent titrations only (those within 0.10 cm³ of each other)

◦ Average titre = $\frac{22.80 + 22.80}{2} = 22.80 \text{ cm}^3$

Step 2: Calculate the number of moles of sodium hydrogencarbonate

◦ Moles = $\frac{22.80}{1000} \times 0.200 = 4.56 \times 10^{-3} \text{ moles}$

Step 3: Calculate (or deduce) the number of moles of hydrochloric acid

- The stoichiometry of NaHCO₃ : HCl is 1 : 1
- Therefore, the number of moles of sodium hydrogencarbonate is also 4.56 × 10⁻³ moles

uncertainties

Step 4: Calculate the concentration of hydrochloric acid

 moles 4.56×10^{-3}

-3



o Concentration = = $\frac{\text{volume}}{(25.0 / 1000)}$ = **0.182 mol dm**

? Worked Example PAG 2.2: Determination of the molar mass of an acid

0.133 g of an unknown monoprotic acid is added to a 1.0 dm³ volumetric flask and made up to the mark with distilled water. This solution is titrated with 0.070 mol dm⁻³ sodium hydroxide solution. The average titre required for the neutralisation of the unknown monoprotic acid is 23.40 cm³.

- i) Calculate the molar mass of the unknown acid.
- ii) The unknown monoprotic acid is a halogenated acid. Identify the unknown monoprotic acid.

Answer

Part i)

Step 1: Calculate the number of moles of sodium hydroxide

$$\circ \text{ Moles of NaOH} = 0.070 \times \frac{23.40}{1000} = 1.638 \times 10^{-3} \text{ moles}$$

Step 2: State the number of moles of the unknown monoprotic acid

$$\circ \text{ Moles of acid} = 1.638 \times 10^{-3} \text{ moles since the stoichiometric ratio is } 1\text{HX} : 1\text{NaOH}$$

Step 3: Calculate the molar mass of the unknown monoprotic acid

$$\circ M_r = \frac{\text{mass}}{\text{moles}} = \frac{0.133}{1.638 \times 10^{-3}} = 81.20 \text{ g mol}^{-1}$$

Part ii)

Step 1: Calculate the mass of the halogen


$$\circ 81.20 - 1.0 = 80.2$$

Step 2: Use the periodic table to identify the halogen


$$\circ 80.2 \approx \text{Bromine (79.9)}$$

Step 3: State the final answer

$$\circ \text{The unknown monoprotic acid is HBr / hydrobromic acid}$$

 Exam Tip

- You can potentially be asked to calculate the molar mass of any unknown acid including monoprotic, diprotic and even triprotic acids
 - You must account for the stoichiometry of the neutralisation reaction
- To identify the unknown acid, you have to be given more information in order to deduce its identity

 Worked Example

PAG 2.3: Identification of an unknown carbonate

1.19 g of an unidentified Group 1 metal carbonate, X_2CO_3 , was dissolved in water to produce a 250.0 cm^3 standard solution. 25.0 cm^3 aliquots of this solution were titrated with 0.150 mol dm^3 hydrochloric acid. The average titre for this experiment was found to be 14.95 cm^3 .

Identify the Group 1 metal in the unidentified metal carbonate X_2CO_3 .

Answer

Step 1: Calculate the number of moles of hydrochloric acid

$$\circ \text{ Moles of HCl} = 0.150 \times \frac{14.95}{1000} = 2.2425 \times 10^{-3} \text{ moles}$$

Step 2: Calculate the number of moles of X_2CO_3

$$\circ \text{ Moles of } X_2CO_3 = \frac{2.2425 \times 10^{-3}}{2} = 1.12125 \times 10^{-3} \text{ moles (since the stoichiometric ratio is } 1X_2CO_3 : 2HCl)$$

Step 3: Calculate the mass of X_2CO_3 in the 25.0 cm^3 aliquot

$$\circ \frac{1.19}{10} = 0.119 \text{ g}$$

Step 4: Calculate the molar mass of X_2CO_3


$$\circ M_r = \frac{\text{mass}}{\text{moles}} = \frac{0.119}{1.12125 \times 10^{-3}} = 106.132 \text{ g mol}^{-1}$$

Step 5: Calculate the mass of X in X_2CO_3

$$\circ \frac{106.132 - 12.0 - (3 \times 16.0)}{2} = 23.066$$

Step 6: Use the periodic table to identify the Group 1 element in X_2CO_3

o $23.066 \approx$ Sodium/ Na (relative mass of 23.0)

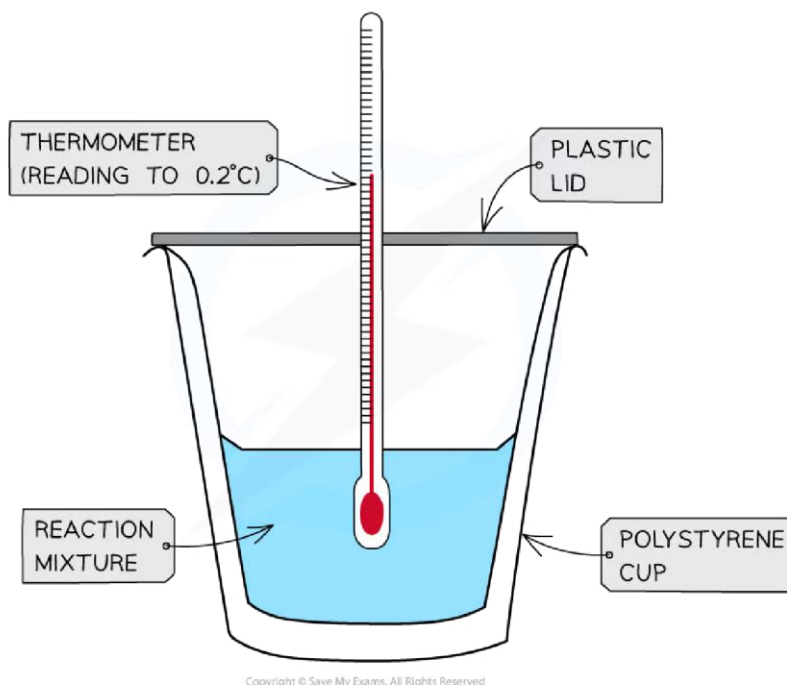
 Exam Tip

- **Careful:** Examiners have been known to construct this question to get the mass of X as around 86.0 - 86.5
 - o This then gets a mixture of answers of X = Rubidium or Francium
 - o This happens because students use the wrong number to identify the element
 - Rubidium has a mass number of 85.5
 - Francium has an atomic number of 87

1.1.3 Determination of Enthalpy Changes

PAG 3 Determination of Enthalpy Changes

- reactions
- A **calorimeter** can be made up of a **polystyrene drinking cup**, a **vacuum flask** or **metal can**



A polystyrene cup can act as a calorimeter to find enthalpy changes in a chemical reaction

- The energy needed to raise the temperature of 1 g of a substance by 1 K is called the **specific heat capacity** (c) of the liquid
- The **specific heat capacity** of water is $4.18 \text{ J g}^{-1} \text{ K}^{-1}$
- The energy transferred as heat can be calculated by:

$$q = m \times c \times \Delta T$$

q = THE HEAT TRANSFERRED, J
 m = THE MASS OF WATER, g
 c = THE SPECIFIC HEAT CAPACITY, $\text{J g}^{-1} \text{K}^{-1}$
 ΔT = THE TEMPERATURE CHANGE, K

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Calorimetry is a technique used to measure changes in enthalpy of chemical



Equation for calculating energy transferred in a calorimeter

- There are two types of calorimetry experiments for you to know:

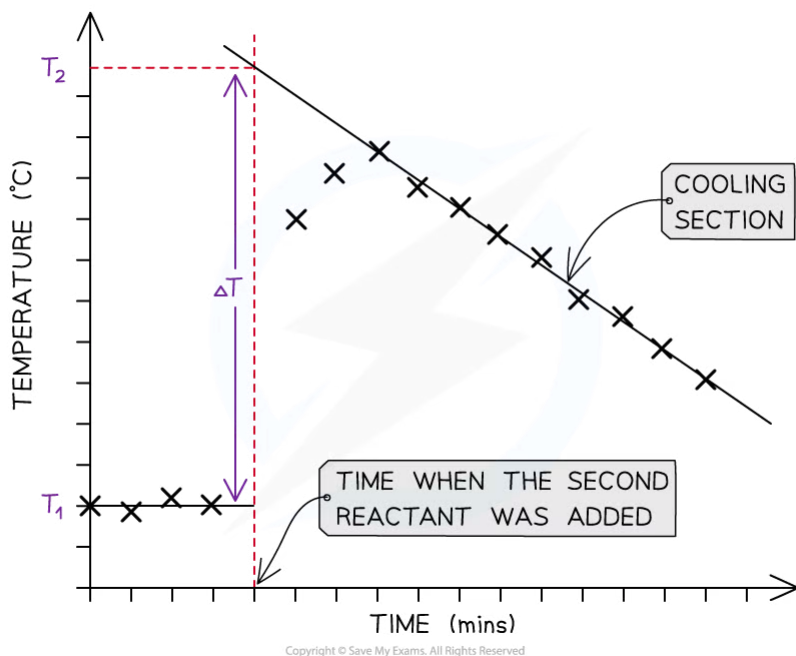
- **Enthalpy changes of reactions in solution** Enthalpy changes of
- **combustion**
- In both cases you should be able to give an outline of the experiment and be able to process experimental data using calculations or graphical methods

PAG 3.1 Determination of the Enthalpy Change of Neutralisation

- The principle of these calorimetry experiments is to mix stoichiometric quantities of acid and alkali then measure the temperature change over the course of a few minutes
- The apparatus needed to carry out an enthalpy of reaction in solution calorimetry experiment is shown above

Sample method for a neutralisation reaction

1. Using a measuring cylinder, place 25.0 cm³ of 1.0 mol dm⁻³ hydrochloric acid into the polystyrene cup
 2. Using a measuring cylinder, place 25.0 cm³ of 1.0 mol dm⁻³ sodium hydroxide solution into a separate polystyrene cup
 3. Draw a table to record the initial temperature of the HCl (aq) and NaOH (aq) and then the temperature of the combined reaction mixture every half minute up to 9.5 minutes
 4. Put a thermometer or temperature probe in each cup, stir, and record the temperature every half minute for 2½ minutes
 5. At precisely 3 minutes, add the contents of the two cups together (DO NOT RECORD THE TEMPERATURE AT 3 MINUTES)
 6. Continue stirring and record the temperature for an additional 6 minutes
- For the purposes of the calculations, some assumptions are made about the experiment:
 - That the specific heat capacity of the solution is the same as pure water, i.e. **4.18 J g⁻¹ K⁻¹**
 - That the density of the solution is the same as pure water, i.e. **1 g cm⁻³**
 - The specific heat capacity of the container is ignored
 - The reaction is complete There are
 - negligible heat losses
 -
 - **Temperature correction graphs**
 - For reactions which are not instantaneous there may be a delay before the maximum temperature is reached
 - During that delay the substances themselves may be losing heat to the surroundings, so that the true maximum temperature is never actually reached To overcome this problem we can use
 - graphical analysis to determine the maximum enthalpy change



A temperature correction graph for a metal displacement reaction between zinc and copper sulfate solution. The zinc is added after 4 minutes

The steps to make a temperature correction graph are:

1. Take a temperature reading before adding the reactants for a few minutes to get a steady value
2. Add the second reactant and continue recording the temperature and time
3. Plot the graph and **extrapolate** the cooling part of the graph until you intersect the time at which the second reactant was added

• For the neutralisation experiment, there can be two main possibilities:

1. The initial temperatures of the HCl (aq) and NaOH (aq) are the same ■ In this case, they can both be plotted as the same line, the graph can be extrapolated to determine the change in temperature at 3 minutes and the subsequent $q=mc\Delta T$ calculation completed
2. The initial temperatures of the HCl (aq) and NaOH (aq) are the different ■ In this case, the initial temperature of the HCl (aq) is plotted as one line and the initial temperature of the NaOH (aq) are plotted as a separate line on the same graph.
 - The temperature of the reaction mixture can be plotted and then you will do two extrapolations for the HCl (aq) and the NaOH (aq)
 - After completing the subsequent $q=mc\Delta T$ calculations for the HCl (aq) and NaOH (aq), you calculate the average of those two values to get the overall enthalpy change of neutralisation

PAG 3.2 Determination of an enthalpy change of reaction by Hess' law

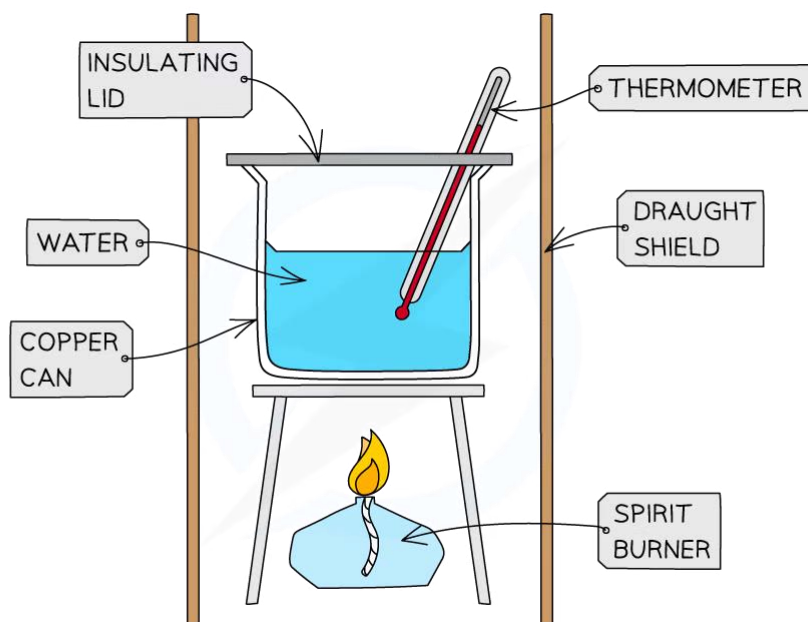
- This practical is an extended variation of PAG 3.1

- 9. Use the equation $q = m \times c \times \Delta T$ to calculate:
 - ΔH_1 (enthalpy change of reaction for KHCO_3 with HCl)
 - ΔH_2 (enthalpy change of reaction for K_2CO_3 with HCl)
 - 10. Apply Hess' law to calculate ΔH_r (enthalpy change for the decomposition of KHCO_3 to K_2CO_3)
- Using $\Delta H_r = \Delta H_1 - \Delta H_2$

PAG 3.3 Determination of Enthalpy Changes of Combustion

The principle here is to use the heat released by a combustion reaction to increase

- the heat content of water
- A typical simple calorimeter is used to measure the temperature changes to the water

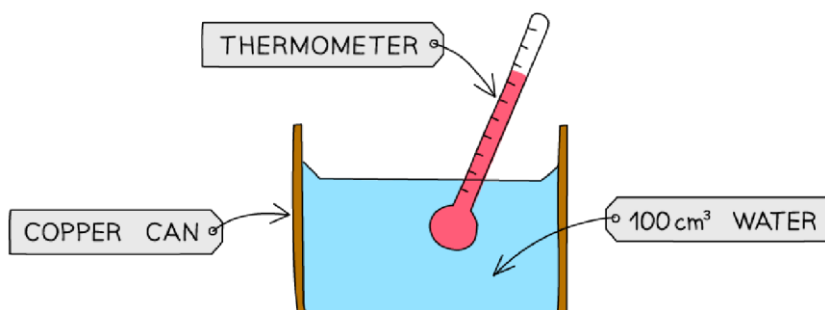


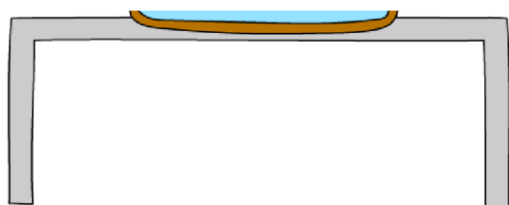
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A simple combustion calorimeter

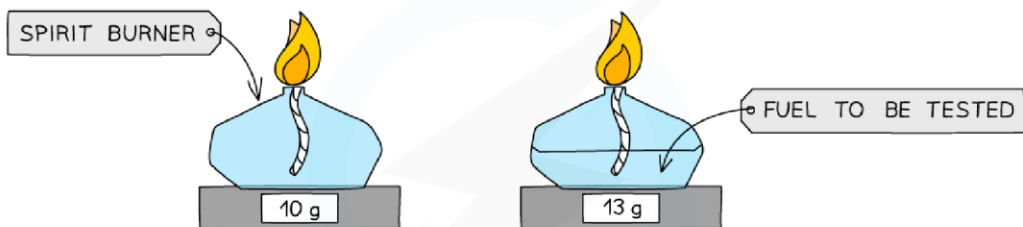
- To complete this experiment, the following steps will need to be completed:

1. FILL COPPER CAN WITH 100cm^3 OF WATER. RECORD INITIAL TEMPERATURE WITH THERMOMETER.

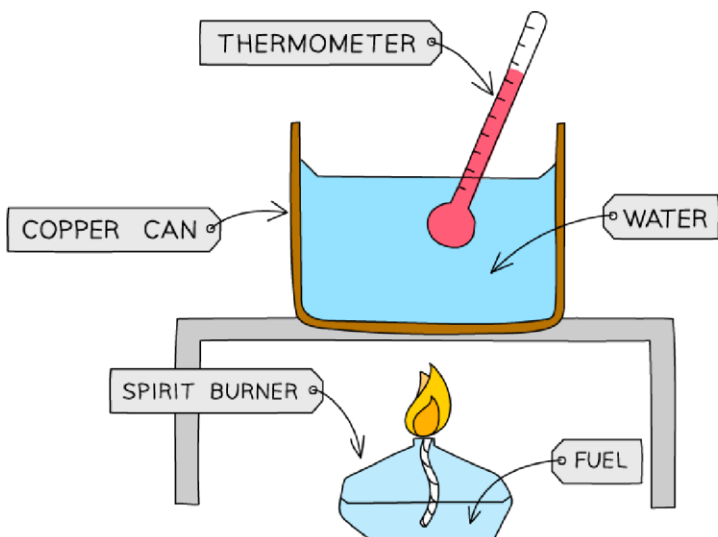




2 MEASURE AND RECORD THE MASS OF AN EMPTY SPIRIT BURNER. ADD FUEL AND RECORD MASS



3 LIGHT THE WICK. HEAT WATER (STIRRING CONSTANTLY) FOR SET TIME/UNTIL FUEL IS COMPLETELY BURNT. RECORD TEMPERATURE OF WATER. MEASURE MASS OF BURNER + ANY REMAINING FUEL. SUBTRACT TO FIND MASS OF FUEL BURNT.



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- It is important that you record the starting temperature, and the final temperature in order to
 - complete the calculations
- You must also record the starting mass of the spirit burner and the final mass of the spirit burner, so that you can work out the mass of the fuel burned during the reaction
- This will then be used to calculate the moles, which will be used to convert Q to an enthalpy change in your calculations

Key points to consider

- Not all the heat produced by the combustion reaction is transferred to the water
 - Some heat is lost to the surroundings
 - Some heat is absorbed by the calorimeter
- To minimise the heat losses the copper calorimeter should not be placed too far above the flame and a lid placed over the calorimeter
- Shielding can be used to reduce draughts
- In this experiment the main sources of error are
 - **Heat losses**
 - **Incomplete combustion**

? Worked Example

1.023 g of propan-1-ol ($M = 60.11 \text{ g mol}^{-1}$) was burned in a spirit burner and used to heat 200 g of water in a copper calorimeter. The temperature of the water rose by $30 \text{ }^\circ\text{C}$. Calculate the enthalpy of combustion of propan-1-ol using this data.

Answer:

Step 1: Calculate q

- $q = m \times c \times \Delta T$
- $q = 200 \text{ g} \times 4.18 \text{ J g}^{-1} \text{ K}^{-1} \times 30 \text{ K} = -25\,080 \text{ J}$

Step 2: Calculate the amount of propan-1-ol burned

$$\text{moles} = \frac{\text{mass}}{\text{molar mass}} = \frac{1.023 \text{ g}}{60.11 \text{ g mol}^{-1}} = 0.01702 \text{ mol}$$

Step 3: Calculate ΔH

$$\Delta H = \frac{q}{n} = \frac{-25080 \text{ J}}{0.01702 \text{ mol}} = -1\,473\,560 \text{ J} = -1\,474 \text{ kJ} = -1.5 \times 10^3 \text{ kJ mol}^{-1}$$

1.2 Organic & Inorganic Practicals

1.2.1 Qualitative Analysis of Ions

PAG 4: Qualitative Analysis of Ions

- Simple test tube reactions can be done to identify the following positive ions:
 - Group 2 ions (M^{2+})
 - Ammonium ions (NH_4^+)
- If the sample to be tested is a solid, then it must be dissolved in deionised water and made into an aqueous solution

Testing for Group 2 Metal ions

- Four test tubes should be placed in a test tube rack
 - Around 10 drops of 0.1 mol dm^{-3} barium chloride solution should be added to the first test tube
Around 10 drops of dilute sodium hydroxide solution ($NaOH$) should be added to the same test tube
 - Swirl the test tube carefully to mix well
Continue to add sodium hydroxide dropwise to the test tube, until it is in excess This should then be
 - repeated in the other test tubes, for calcium chloride solution,
 - magnesium chloride solution and strontium chloride solution
 - Any observations should be noted down in a suitable results table
- The same test as above can also be done using ammonia solution and sulfuric acid solution

The positive results testing for the presence of Group 2 ions

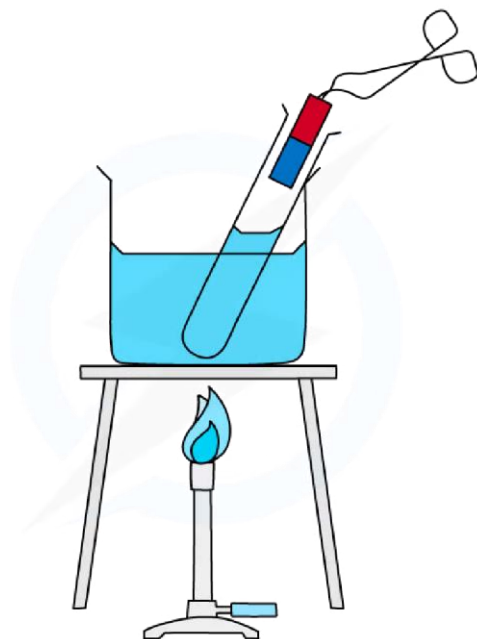
	Mg^{2+}	Ca^{2+}	Sr^{2+}	Ba^{2+}
Ammonium solution	White precipitate – $Mg(OH)_2$	No change seen	No change seen	No change seen
Excess sodium hydroxide	White precipitate – $Mg(OH)_2$	White precipitate – $Ca(OH)_2$	Slight white precipitate – $Sr(OH)_2$	No change seen
Excess sulfuric acid	Colourless solution	Slight white precipitate – $CaSO_4$	White precipitate – $SrSO_4$	White precipitate – $BaSO_4$

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Testing for Ammonium Ions

- About 10 drops of a solution containing ammonium ions, such as ammonium chloride, should be added to a clean test tube
- About 10 drops of sodium hydroxide should be added using a pipette
- The test tube should be swirled carefully to ensure that it is mixed well
- The test tube of the solution should then be placed in a beaker of water, and the beaker of water should be placed above a Bunsen burner, so that it can become a water bath

- As the solution is heated gently, fumes will be produced
- A pair of tongs should be used to hold a damp piece of red litmus paper near mouth of the test tube, to test the fumes
- The red litmus paper will change colour and become blue in the presence of ammonia gas

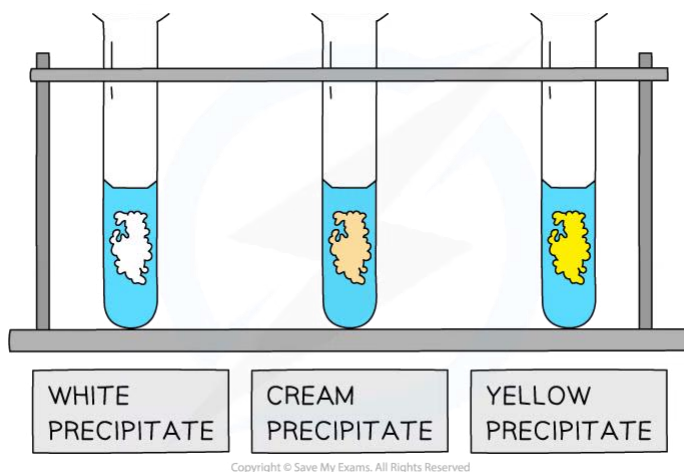


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Damp red litmus paper turning blue in the presence of ammonia gas

Testing for negative ions

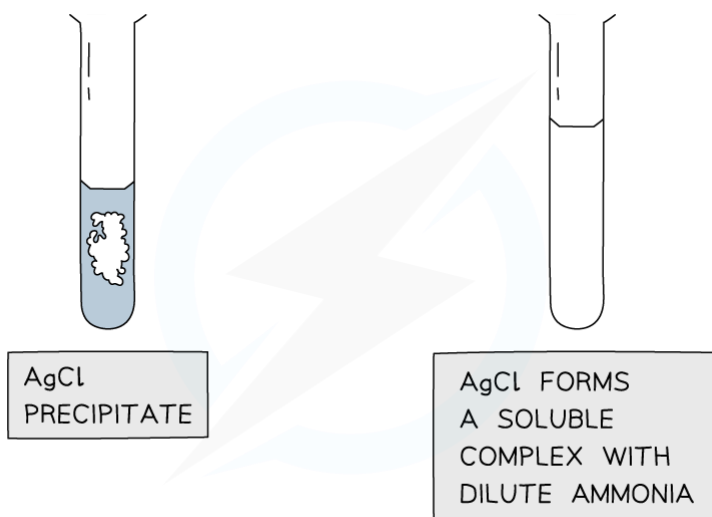
- Simple test tube reactions can be done to identify the following negative ions:
 - Halide ions (X^-)
 - Hydroxide ions (OH^-)
 - Carbonate ions (CO_3^{2-})
 - Sulfate ions (SO_4^{2-})
- If the sample to be tested is a solid, then it must be dissolved in deionised water and made into an aqueous solution
- The sample being tested should be added using a pipette to a test tube
- The test tube should be placed into a test tube rack
- A small amount of nitric acid should be added to the sample using a pipette, followed by a small amount of silver nitrate solution
- A precipitate will form, either white, cream or yellow, if a halide ion is present in the sample



The white, cream and yellow precipitates formed when halide ions react with silver nitrate solution

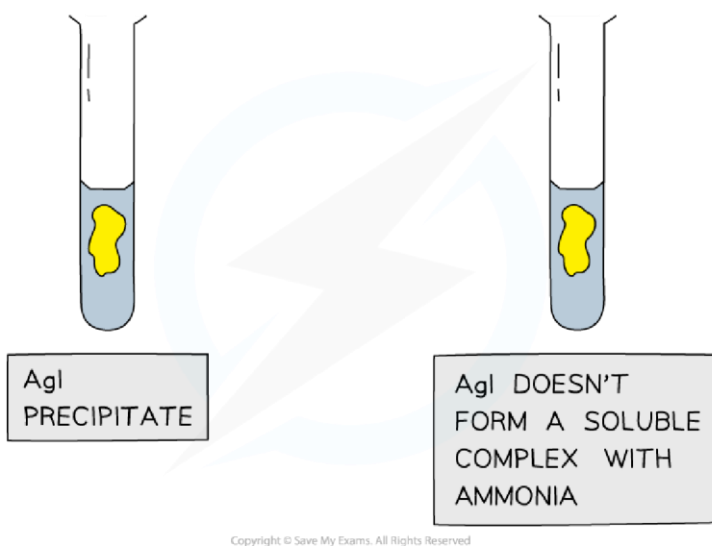
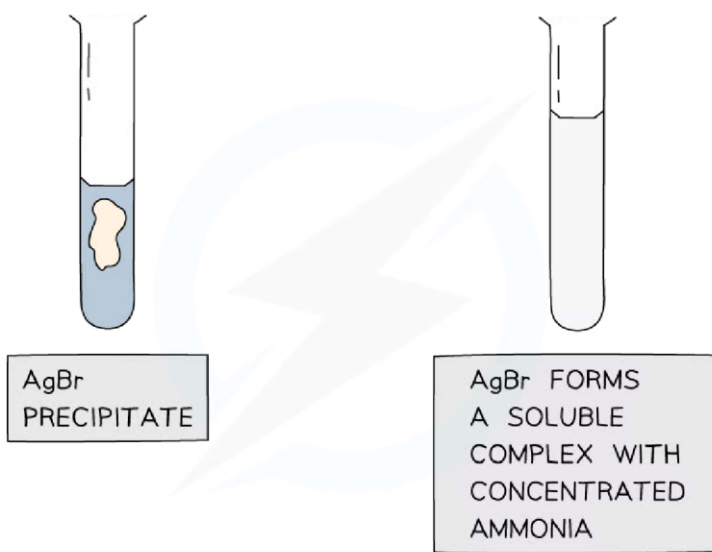
- -
- - The cream precipitate is AgBr
- The yellow precipitate will form if iodide ions are present in the sample
 - The yellow precipitate is AgI

Further test for halides using ammonia solution



The white precipitate will form if chloride ions are present in the sample
The white precipitate is AgCl

The cream precipitate will form if bromide ions are present in the sample



Results of the test with ammonia to further distinguish between silver halide precipitates

Testing for Hydroxide Ions

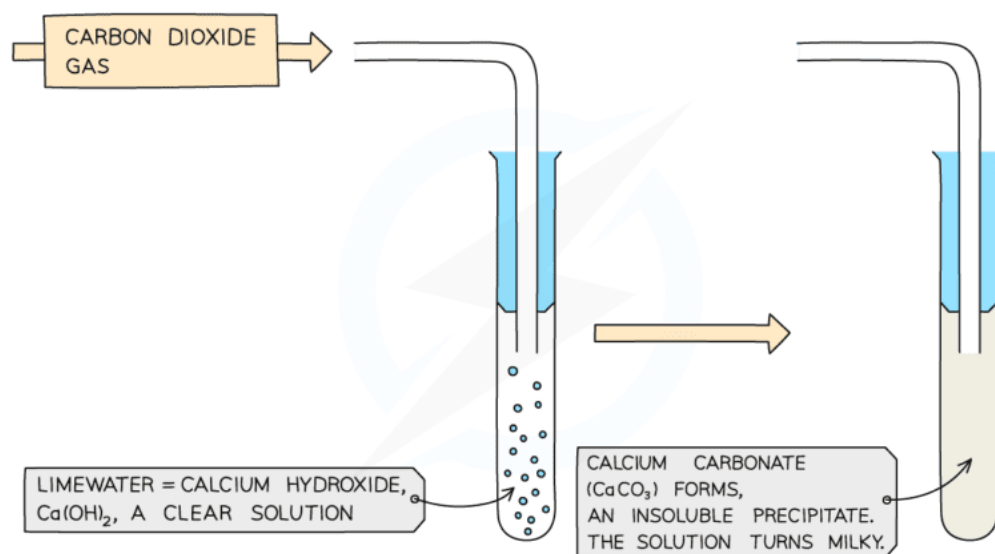
- A small amount (around 1 cm³) of the solution should be added to a test tube using a pipette
- Test the pH of the solution using red litmus paper or universal indicator paper
 - The presence of hydroxide ions will turn the red litmus paper blue and the pH will be clearly alkaline on the universal indicator paper if hydroxide ions are



Testing for Carbonate Ions

- A small amount (around 1 cm^3) of dilute hydrochloric acid should be added to a test tube using a pipette

- An equal amount of sodium carbonate solution should then be added to the test tube using a clean pipette
- As soon as the sodium carbonate solution is added, a bung with a delivery tube should be attached to the test tube
 - The delivery tube should transfer the gas which is formed into a different test tube which contains a small amount of limewater (calcium hydroxide solution)
- Carbonate ions will react with hydrogen ions from the acid to produce carbon dioxide gas
- Carbon dioxide gas will turn the limewater milky



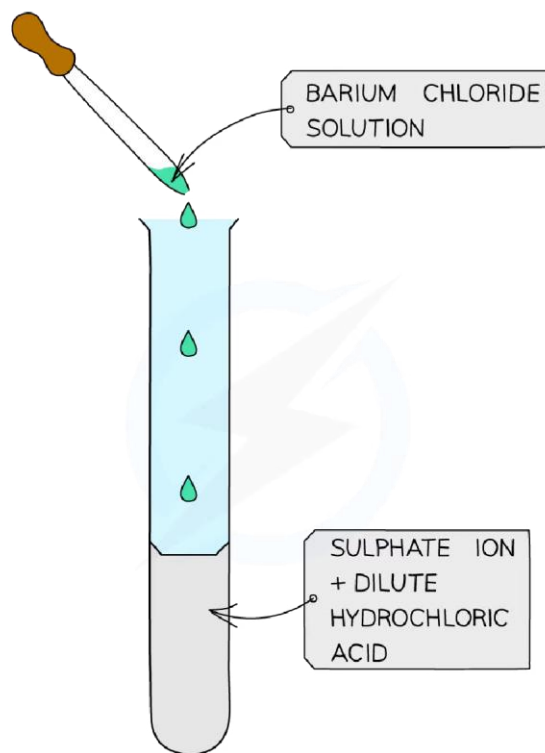
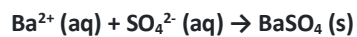
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When carbon dioxide gas is bubbled into limewater it will turn cloudy as calcium carbonate is produced

Testing for Sulfate Ions

- Acidify the sample with dilute hydrochloric acid and then add a few drops of aqueous barium chloride
- If a sulfate is present then a **white** precipitate of barium sulfate is formed:



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A white precipitate of barium sulfate is a positive result for the presence of sulfate ions



Exam Tip

HCl is added first to remove any carbonates which may be present and would also produce a precipitate and interfere with the results.

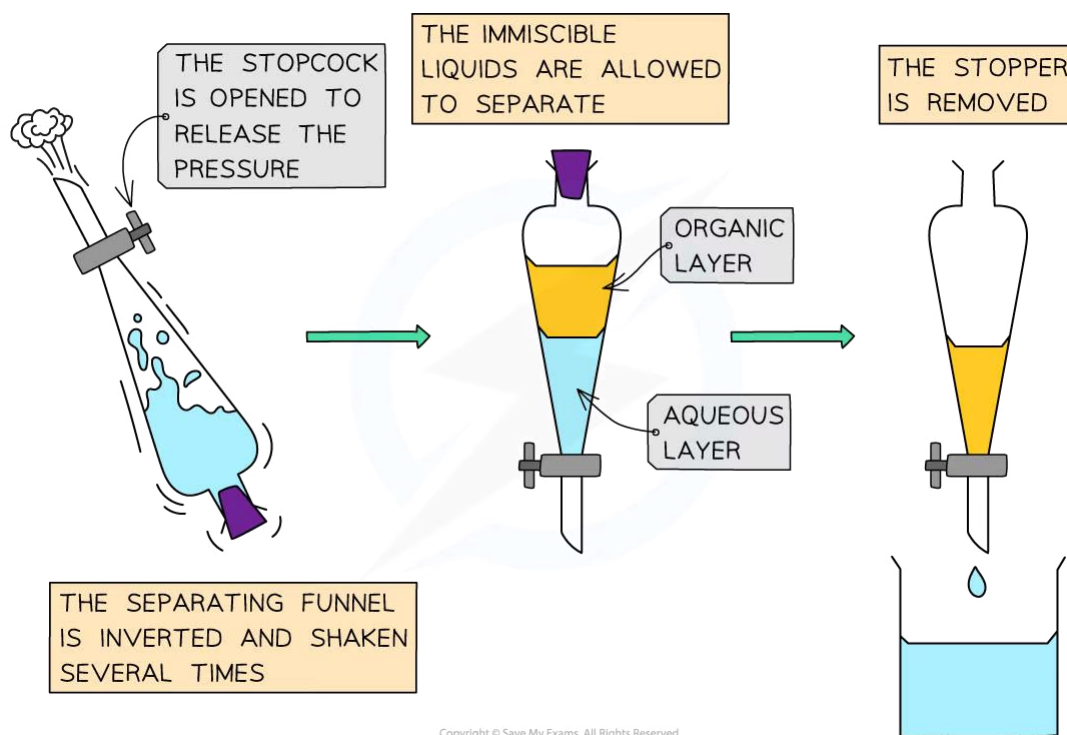
1.2.2 Synthesis of a Haloalkane

PAG 5 1 Synthesis of a Haloalkane

Synthesis of a haloalkane from an alcohol

- Measure 8 cm³ of 2-methylpropan-2-ol in a measuring cylinder
- Pour the 2-methylpropan-2-ol into a separating funnel
- Using a fume hood, add 20 cm³ of concentrated hydrochloric acid to the separating funnel in small portions of 2 - 3 cm³ making sure to release the pressure by opening the stopper after each addition

- Leave the separating funnel to stand in the fume hood for 20 minutes and gently shake the separating funnel at 2 minute intervals
- Allow the layers to separate and dispose of the lower aqueous layer by opening the tap
- Add sodium hydrogen carbonate solution in small portions to the funnel and gently shake the funnel, again, release the pressure at regular intervals
 - This removes acidic impurities
- Once these layers have separated, open the tap once more and dispose of the aqueous layer
- Pour the organic layer into a clean dry conical flask and add two spatulas of anhydrous magnesium sulfate
 - This will remove water
- Once clear, decant the liquid into distillation apparatus
- Distill the liquid and collect the distillate in the range of 47 - 53°C



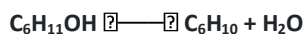
A separating funnel allows the product to be cleaned and isolated

1.2.3 Preparation of Cyclohexene

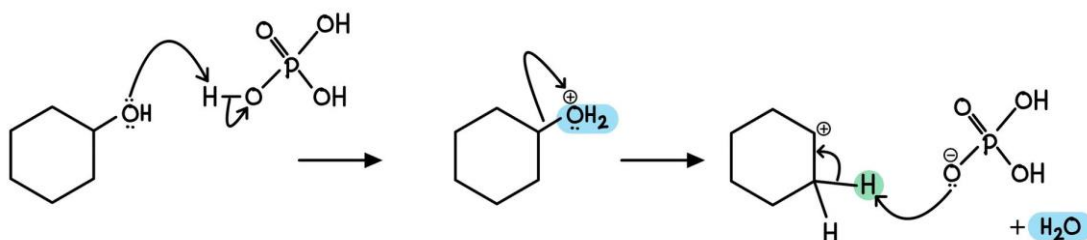
PAG 5 2 Preparation of Cyclohexene

Preparation and Purification of Cyclohexene

- In this experiment an alkene, cyclohexene, will be prepared by dehydration of an alcohol, cyclohexanol, using an acid catalyst such as phosphoric acid



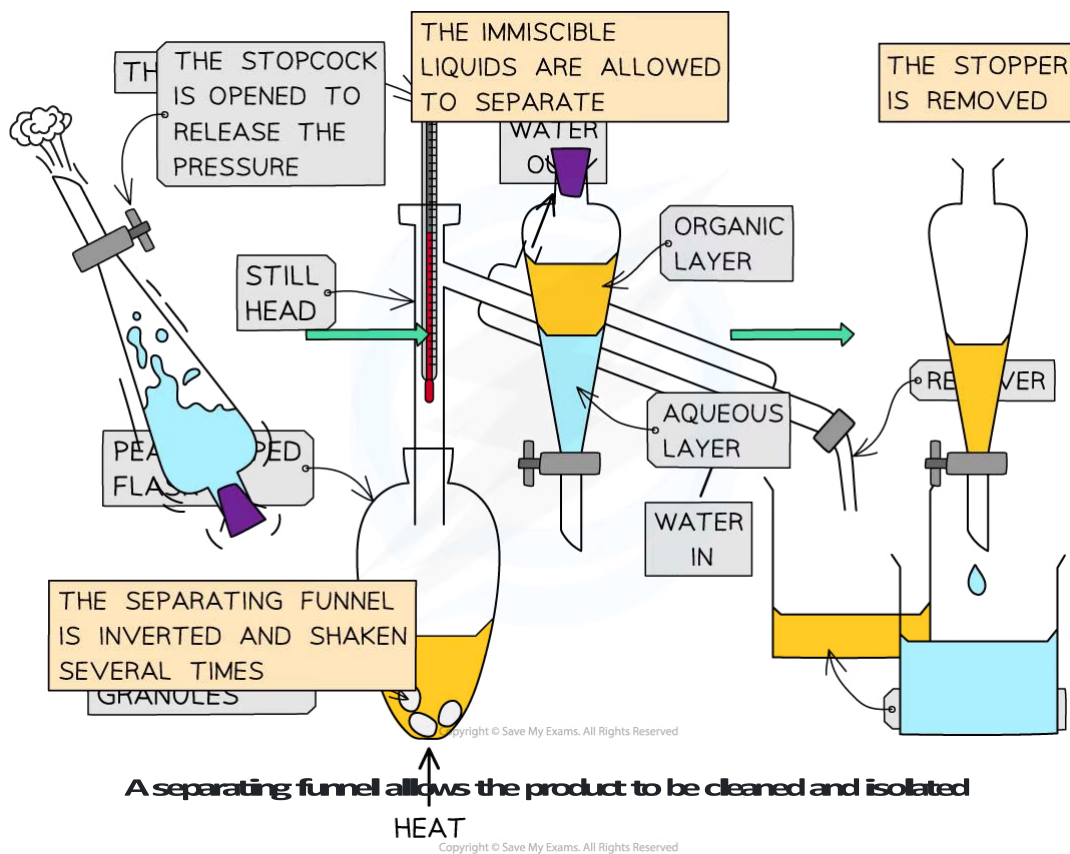
- This is one of the most common methods of preparing alkenes
- The crude product is contaminated with water, unreacted alcohol, phosphoric acid and some side products
- Treatment with a saturated solution of sodium hydrogen carbonate removes traces of acid and a final wash with water removes any remaining carbonate
- Addition of solid calcium chloride will remove remaining water
- The mechanism for the reaction is shown below - you do not need to know this, but you could be asked in a questions to draw in the relevant charges or lone pairs



Elimination mechanism for cyclohexanol

Preparation

- Pour approximately 20 cm³ of cyclohexanol into a weighed 50 cm³ pear shaped flask
- Slowly add 8 cm³ of concentrated phosphoric acid into the flask using a pipette
- Add a few **anti-bumping** granules to the flask and assemble the distillation apparatus, so that the contents of the flask may be distilled
- Heat the flask gently using an electric heater or water bath
- Collect the distillate



Purification

- Pour the distillate into a separating funnel and add 10 cm³ of sodium hydrogen carbonate solution, this will remove unreacted acid and acidic impurities in the organic liquid
- Shake the funnel and allow the mixture to separate, release the pressure built up in the funnel
- Carefully run off the lower aqueous layer and then transfer the upper layer which contains the crude cyclohexene into a conical flask
- Add solid anhydrous calcium chloride to the crude cyclohexene to remove water
- Stopper the flask, shake the contents and allow to stand
- Once clear, transfer the liquid to a clean, dry beaker
- A small portion of the distillate can be tested with bromine water to confirm if the product is an alkene
 - The colour change will be from orange brown to colourless

1.2.4 Oxidation of Ethanol

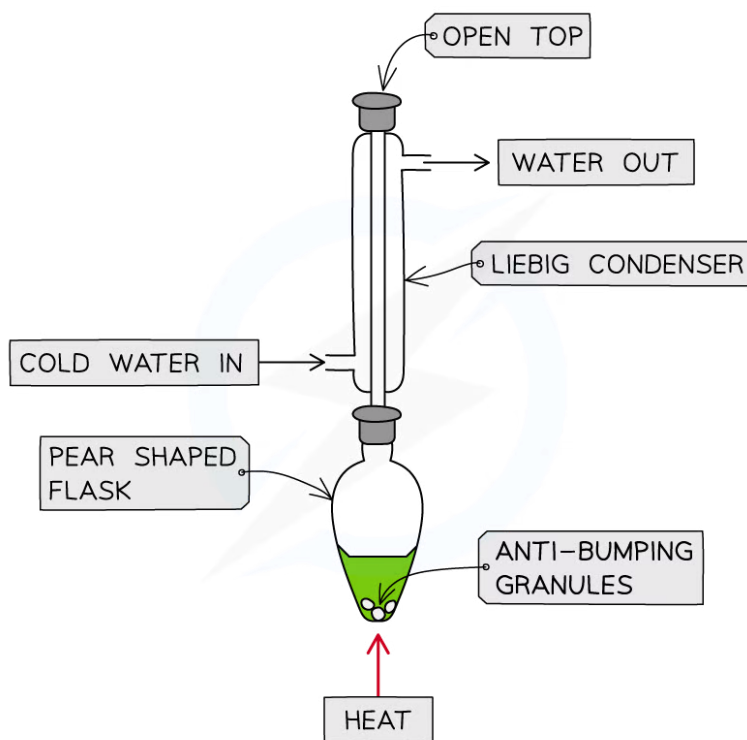
PAG 5 3 Oxidation of Ethanol

Oxidation of ethanol

- Primary alcohols can be oxidised to form **aldehydes** which can undergo further oxidation to form **carboxylic acids**
 - When ethanol is oxidised, ethanal is produced and when oxidised further ethanoic acid will be formed

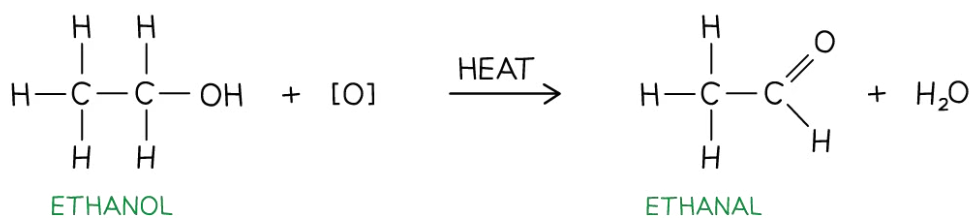
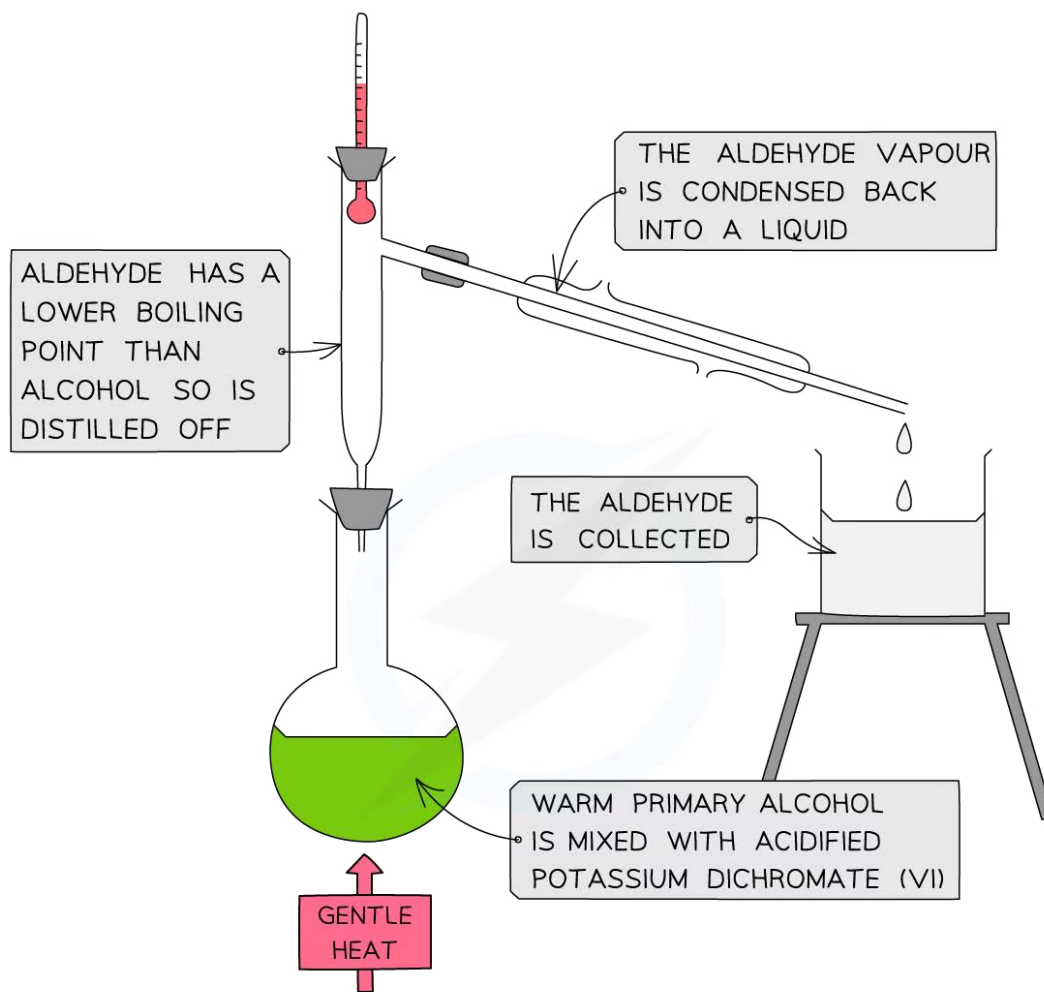
Synthesis and purification of ethanal and ethanoic acid

- Carefully add 20 cm³ of acidified potassium dichromate(VI) solution, K₂Cr₂O₇ (aq), to a 50 cm³ pear-shaped flask and cool the flask in an iced water bath
- Set up the reflux apparatus keeping pear shaped flask cool
- Place **anti-bumping granules** into the pear shaped flask
- Measure out 1 cm³ of ethanol
- Using a pipette, slowly add the ethanol drop wise into the reflux condenser When the ethanol has been added remove the ice bath and allow to warm up to room temperature
- Position the flask over an electric heater or in a water bath and heat for 20 minutes
- Ethanol is flammable, therefore, naked flames should not be used when heating which is why the use of an electric heater or water bath is an
 - important safety precaution
- Purify the product using distillation apparatus



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Reflux apparatus for the oxidation of ethanol to ethanoic acid



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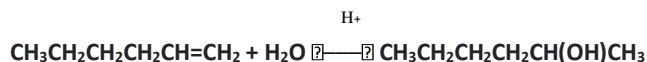
Oxidation of ethanol by acidified $\text{K}_2\text{Cr}_2\text{O}_7$ to form an aldehyde by distillation

1.2.5 Hydration of Hex-1-ene

PAG 5.4 Hydration of Hex 1-ene

Hydration of an alkene

- Hex-1-ene, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}=\text{CH}_2$, can be reacted with concentrated sulfuric acid, followed by water, to make hexan-2-ol, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$. The overall equation is:



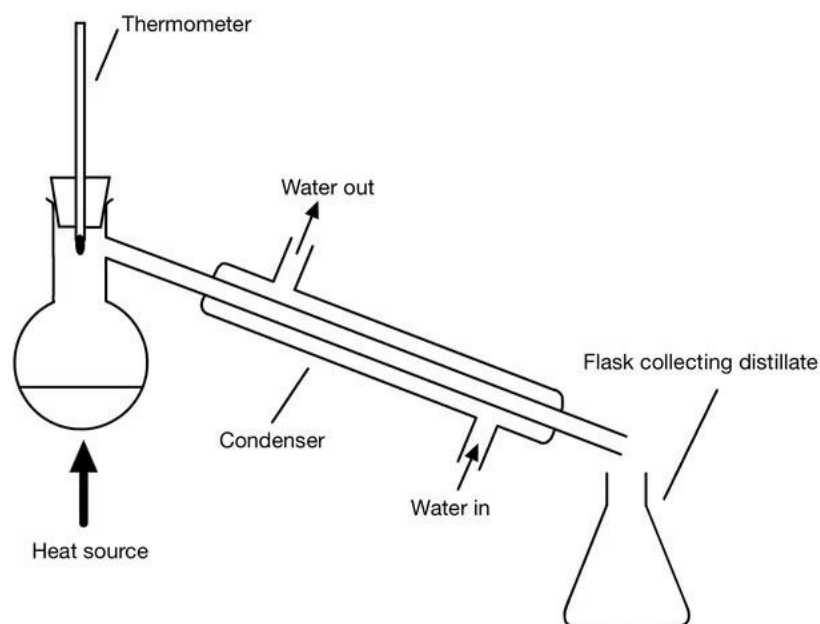
- The hexan-2-ol is then separated, distilled and the product is tested

Preparation of hexan-2-ol

- Prepare an ice bath in a 250 cm³ beaker
 - Using a measuring cylinder, measure out 5 cm³ of hex-1-ene and pour this into a boiling tube
- Plug the boiling tube with mineral wool and cool in the ice bath
- Using a measuring cylinder measure out 5 cm³ of concentrated sulfuric acid. Once the hex-1-ene has been cooling for about 5 minutes, remove the mineral wool bung and slowly add the acid to the hex-1-ene
 - Make sure the boiling tube is kept in the ice bath during this addition.
- Stir the mixture until it becomes one layer (homogenous)
- Keep the boiling tube in the ice bath and add 10 cm³ of distilled water which will separate the reaction mixture into two layers
 - The **top** layer will contain hexan-2-ol. The **bottom** layer will contain unreacted acid

Separation and distillation of hexan-2-ol

- Pour the contents of the boiling tube into a separating funnel. Shake several times
 - and allow the contents to settle
- Remove the stopper and allow the lower layer to run off
- Add 10 cm³ of distilled water to the funnel and shake, remove the lower layer once again
- Add 25 g of **anhydrous sodium carbonate** in a 250 cm³ conical flask and run the hexan-2-ol layer into this flask
 - This will dry the hexan-2-ol
 - This could also neutralise any unreacted acid, however, this should have already been removed when the aqueous layers were removed
- Filter the contents of the flask
- Set up distillation apparatus and distil, collecting the fraction between 130 °C to 160 °C
 - Boiling point of hexan-2-ol is 136 °C
 - Boiling point of hex-1-ene 63 °C



Distillation apparatus

- Hexan-2-ol has hydrogen bonding between its molecules giving it a much higher boiling point than hex-1-ene, which just has temporary induced dipole-dipole forces

Testing hex-1-ene and hexan-2-ol

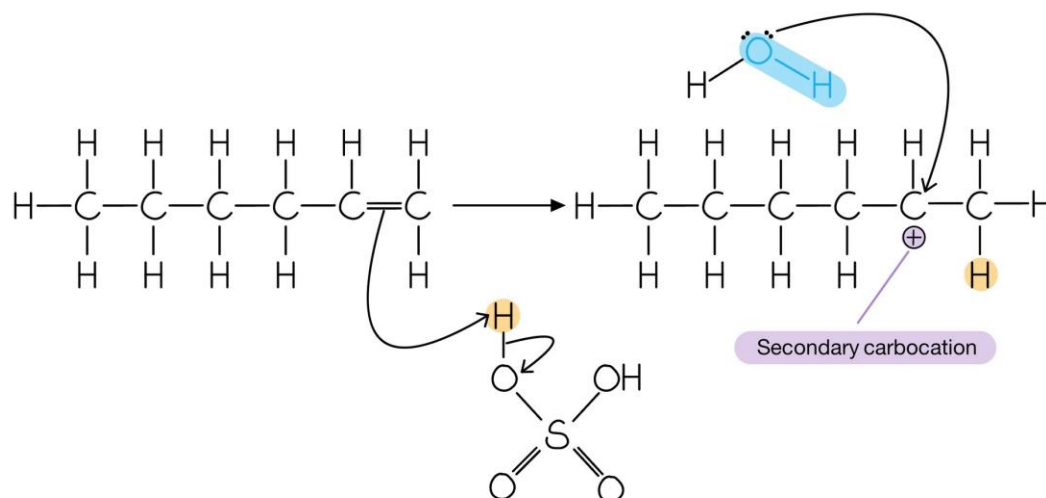
Table of tests and results for hex-1-ene and hexan-2-ol

Test	Result for hex-1-ene	Result for hexan-2-ol
Shake with bromine water	Orange to colourless	No change
Shake with acidified KMnO_4	Purple to colourless	No change
Addition of sodium metal	No reaction	Fizzing seen
Combustion	Smokey flame	Less smokey flame

Why is hexan-2-ol formed?

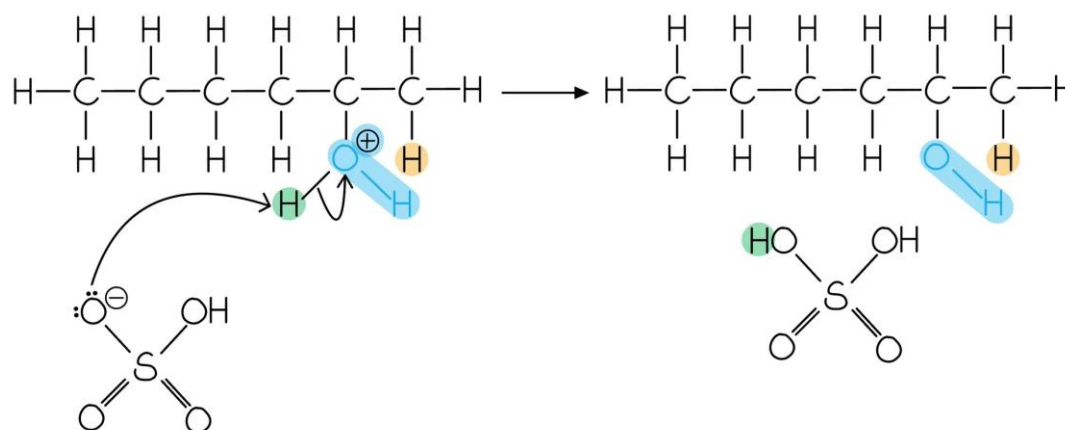
- Hexan-2-ol is formed as the **secondary carbocation** that is formed in the mechanism is more stable than the **primary carbocation**. The mechanism
- is as follows:

Stage 1:



A secondary carbocation is formed when the double bond in hex-1-ene attacks the H atom in sulfuric acid

Stage 2:



The bond between the green hydrogen and the blue oxygen undergoes heterolytic cleavage, and both the electrons from the bond move onto the blue oxygen. The now negatively-charged strong acid picks up the green electrophilic hydrogen reforming the original sulfuric acid catalyst.

1.3 Further Organic & Inorganic Chemistry Practicals

1.3.1 Synthesis of Aspirin

PAG 6.1 Synthesis of Aspirin

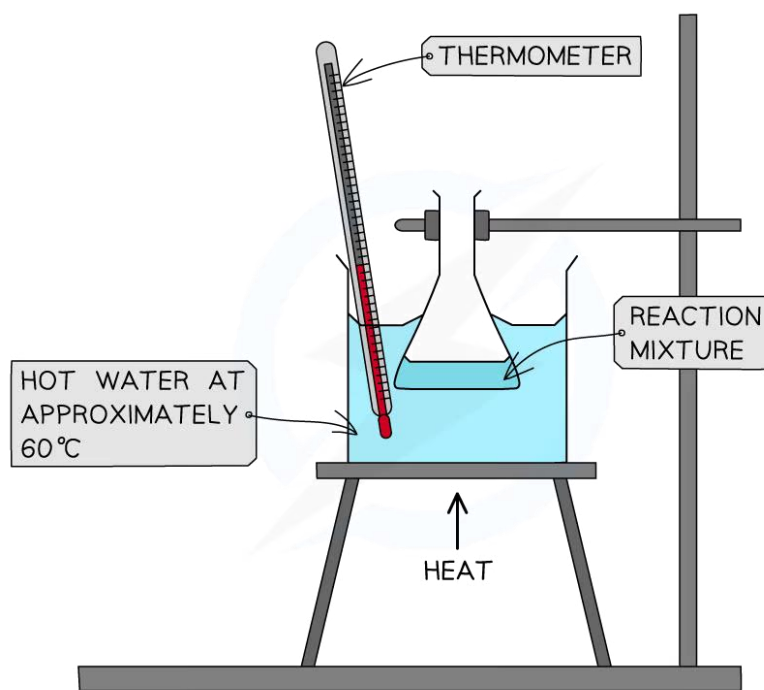
Synthesis of an Organic Solid

- This practical covers a number of key laboratory skills use appropriate
 - apparatus to record a range of measurements use water bath or electric heater
 - or sand bath for heating use laboratory apparatus for a variety of experimental techniques purify a solid product by recrystallisation
 - use melting point apparatus safely and carefully
 - handle solids and liquids
 - There are a few choices of experiments that are commonly done in schools which cover these laboratory skills and three which can be done are
- The preparation of aspirin
 - The purification of benzoic acid
 - The nitration of methyl benzoate
 -

Synthesis of Aspirin

Key steps in the procedure

- 6.0 g of salicylic acid is added to a conical flask along with 10 cm³ of ethanoic anhydride and 5 drops of concentrated sulfuric acid
- The mixture is swirled and held in a warm water bath around 60 °C for about 20 minutes
The flask is then allowed to cool and the contents are added to 75 cm³ cold water
- in a beaker at which point the aspirin crystallises out
The aspirin is recovered using Buchner filtration and left to dry
-



The preparation of aspirin using a hot water bath

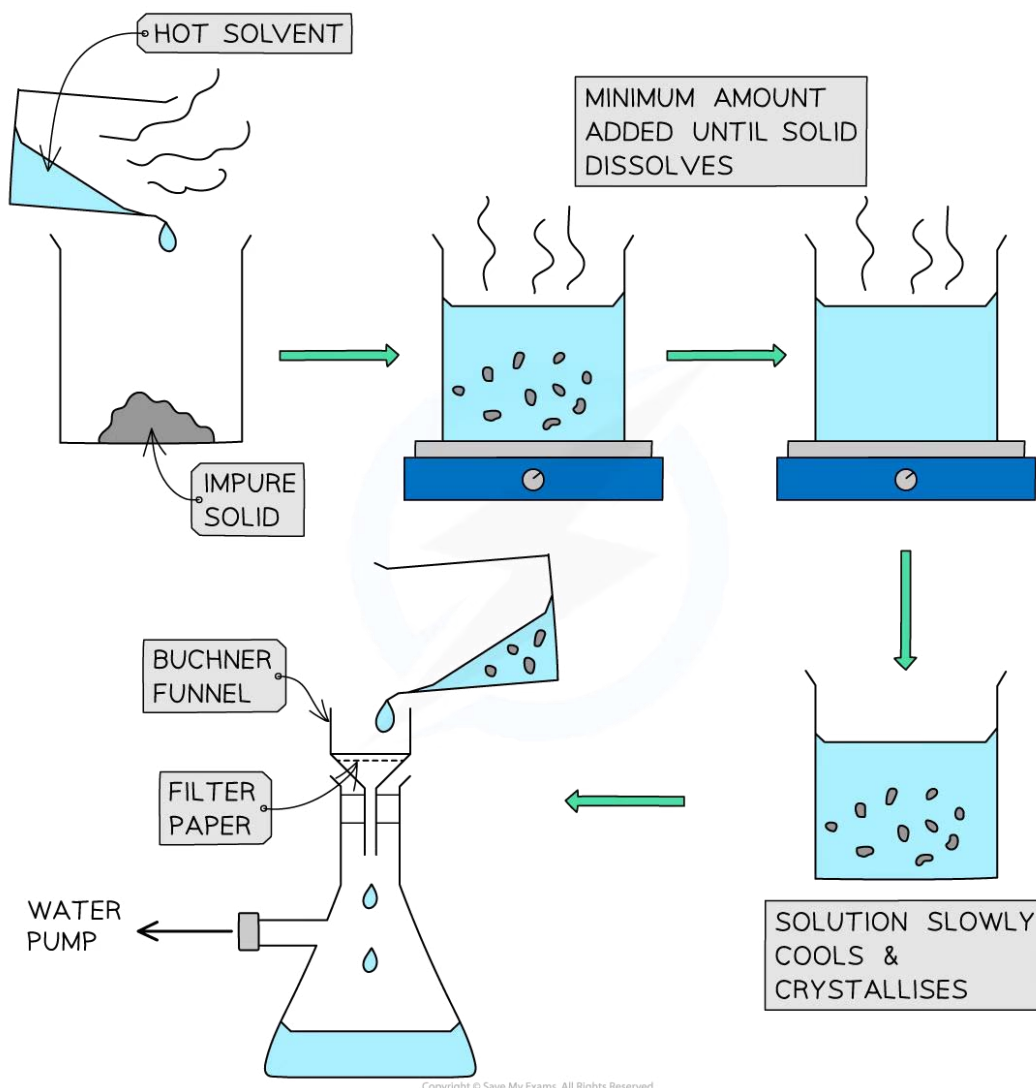
Recrystallisation and melting point test

- The impure aspirin is recrystallised using ethanol, and when the solid has dissolved the solution is poured over 40 cm³ cold water and recovered using Buchner filtration as before
- The melting point of pure aspirin is 135°C, so the purity of the product can be assessed

Recrystallisation

- Recrystallisation is used to purify impure solids
- The principle is that a hot solvent is used to dissolve both the organic solid and the impurities and then as the solution cools the solid crystallises out and leaves behind the impurities in the solution
- The key is using the minimum amount of solvent to dissolve the solid and avoid loss of the product
- If any solid impurities remain in the solution, a hot filtration can be carried out
- Once the solution has cooled down to room temperature and crystallised then the product crystals can be recovered by filtration
- This is faster using Buchner apparatus in which filtration occurs under reduced pressure





Recrystallisation and Buchner filtration

- After filtration the product is washed with fresh cold solvent and then allowed to dry on filter paper

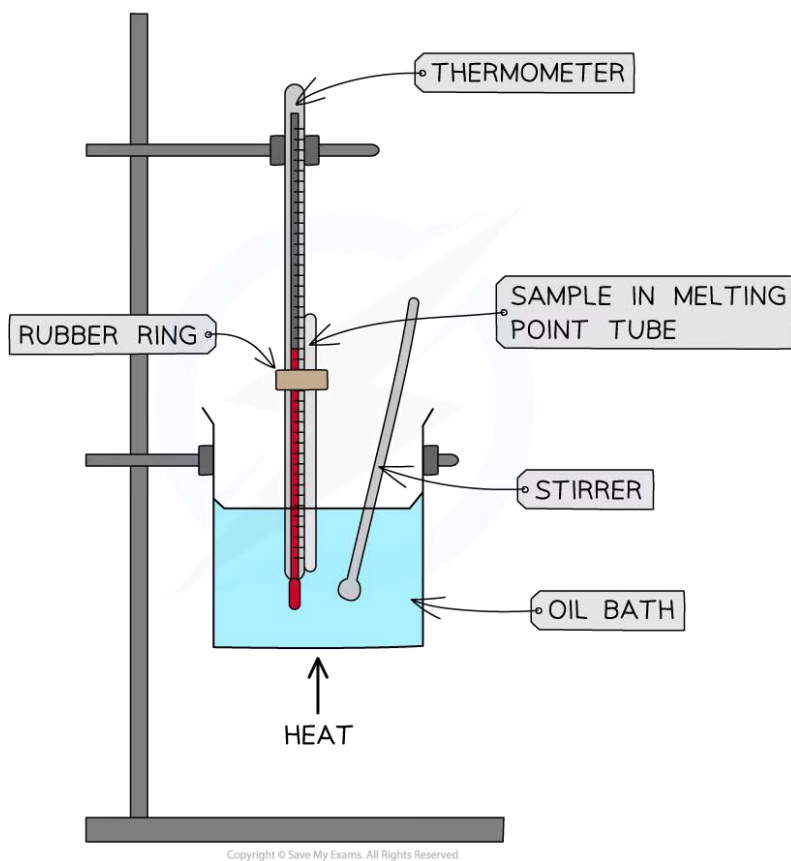


Exam Tip

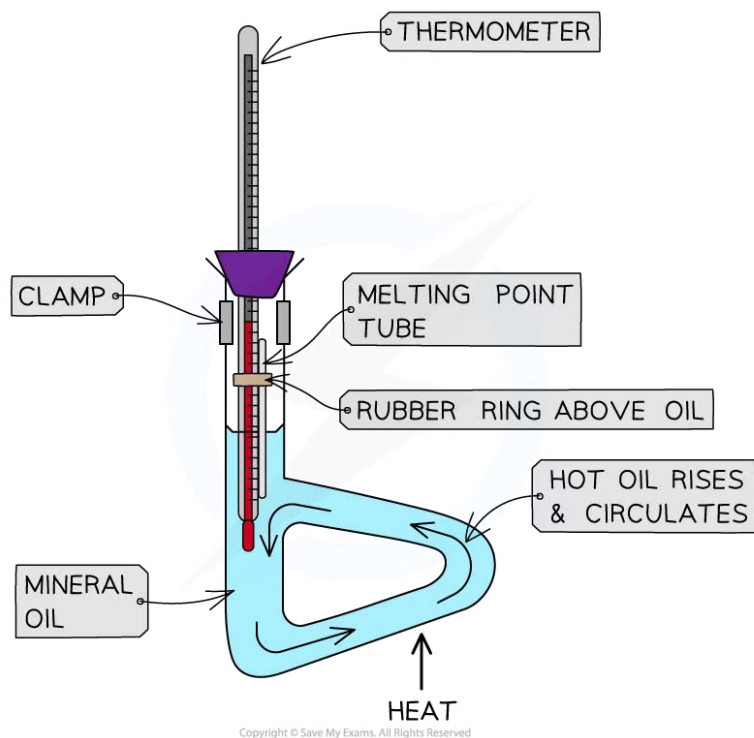
Recrystallisation can be repeated more than once to ensure a very pure product, but each time the yield of product will decrease. Slow cooling results in bigger well defined crystals which are easier to filter and dry.

Melting point analysis

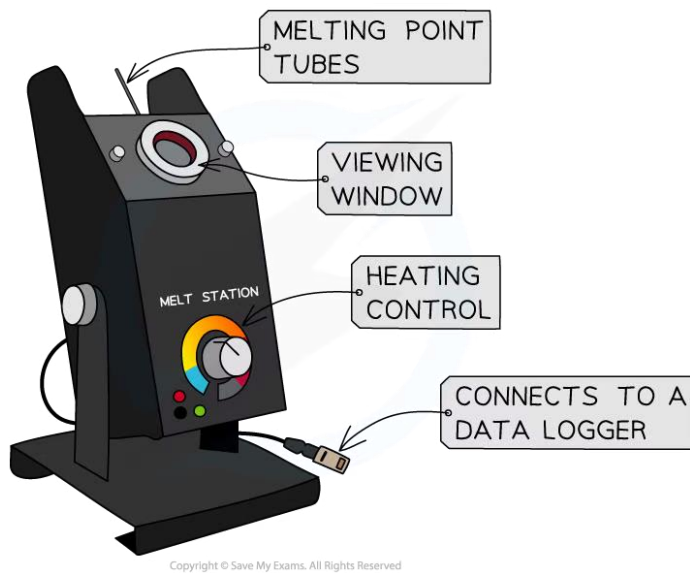
- The melting point of a solid is indicative of its purity and identity
- A melting point can be matched to a known substance as a means of identification or confirmation of a desired product
- The proximity of a melting point to the actual data book value can express purity
 - Impurities tend to lower the melting point of a solid
- The melting point range also reveals the degree of purity
 - Pure substances have sharp well defined melting points
 - Impure substances have a broad melting point range, i.e. a large difference between when the substance first melts until it completely melts
- The skills needed in performing a melting point test are largely dependent on the specific melting point apparatus you are using However, there are
- some common key skills:
 - Correctly preparing the melting point tubes
 - Heating the tubes very slowly
 - Repeating to get a range of measurements (three would be normal)
- The sample solid must be totally dry and finely powdered - this can be achieved by crushing it with the back of a spatula onto some filter paper or the back of a white tile (this absorbs any moisture)
- Use the first tube to find the approximate melting point range and then repeat using a much slower heating rate



Melting point test using an oil bath



Melting point test using a Thiele tube



Melting point test using a melt station



Exam Tip

Always quote a melting point as a range + or - and reference a data book value if you have one.



1.3.2 Preparation of Benzoic Acid

PAG 6.2 Preparation of Benzoic Acid

- Benzoic acid is a suitable substance for purification by recrystallisation as it
 - Readily available in most school labs
 - Dissolves in hot water, but not in cold water
 - Is relatively safe to handle in the laboratory

Key steps in the procedure

-
-
-
-
-
-
- and blocking the funnel
- The filtrate is allowed to cool and crystallise
 - Sometimes crystallisation can be difficult to achieve, so agitating the flask or scratch the inner walls with a glass rod can help initiate the process
- The solid product is then recovered by Buchner filtration, washed and dried



Exam Tip

Benzoic acid lends itself particularly well to melting point testing as it

- does not decompose on heating
- has a melting point in the range 100-200°C
- has a sharp and precise melting point

1.0 g of impure benzoic acid is added to a small conical flask and the flask is placed on a hot plate. Using a supply of hot water, the minimum amount of hot water is added to the flask to dissolve the benzoic acid.

It should be around 30-35 cm³ of water



When is it dissolved a hot filtration is carried out to remove any insoluble impurities

To do this a pre-heated glass funnel is rested on a hot conical flask and a piece of fluted filter paper is added

The funnel should have a short stem to avoid the product solidifying prematurely

1.3.3 Preparation of Methyl 3-Nitrobenzoate

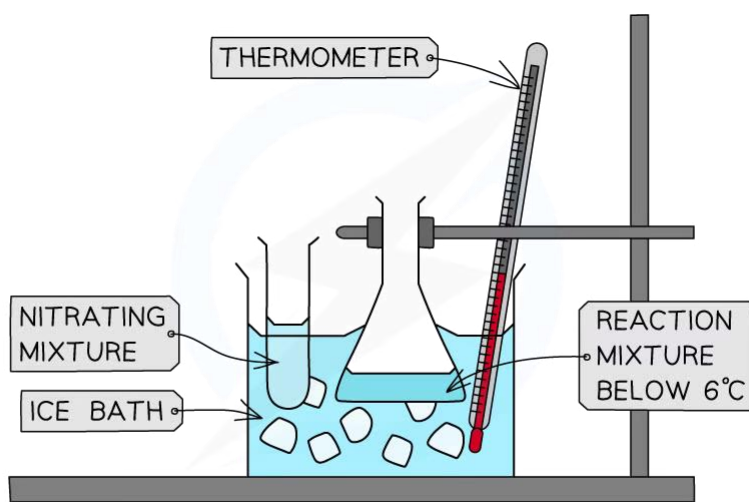
PAG 6.3 Preparation of Methyl 3 Nitrobenzoate

- The equation for the reaction is:



Key steps in the procedure

- 2.0 g of methyl benzoate is added to a small conical flask
- The flask is immersed into an ice-water bath and 4.0 cm³ of concentrated sulfuric acid is slowly added
- A mixture of 1.5 cm³ of concentrated nitric acid and 1.5 cm³ concentrated sulfuric acid is made in a separate test tube and immersed in the ice bath - this is the nitrating mixture
- A thermometer is placed in the conical flask: the temperature must be kept below 6 °C
- The nitrating mixture is very slowly added to the flask over about 15 minutes ensuring the temperature remains low
- After the addition the mixture is allowed to remain at room temperature for around 15mins
- The contents of the flask are then poured over crushed ice where the product solidifies
- The ice is allowed to melt and the product is recovered by Buchner filtration



The nitration of methyl benzoate using an ice bath

Recrystallisation & melting point

- Recrystallisation is carried out in a water-ethanol mixture
-



The crystals are recovered and dried

- The melting point of 3-nitromethylbenzoate is 78 °C

Key hazards

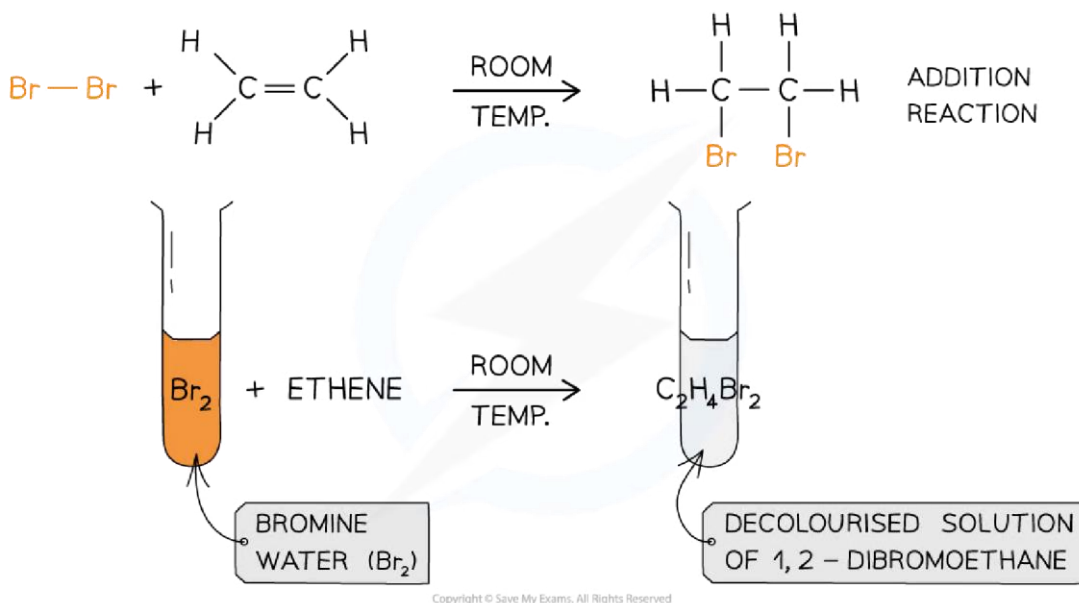
- The concentrated sulfuric and nitric acid are **very corrosive** and **oxidising** so safety glasses and gloves should be worn
- Methyl benzoate is **moderately harmful**
- Ethanol is **flammable** so recrystallisation should be done with a hot plate not a naked flame

1.3.4 Qualitative Analysis of Organic Functional Groups

PAG 7 Qualitative Analysis of Organic Functional Groups

Testing for alkenes / saturation

- Halogens can be used to test if a molecule is **unsaturated** (i.e. contain a double bond)
- Br₂ is an orange or yellow solution, called **bromine water**
- The unknown compound is **shaken** with the bromine water
- If the compound is unsaturated, an addition reaction will take place and the coloured solution will decolourise



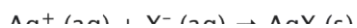
The bromine water test is the standard test for unsaturation in alkenes

Testing for a haloalkane

- The haloalkane must first be warmed with ethanol sodium hydroxide in a mixture of ethanol and water
- The haloalkane will undergo nucleophilic substitution releasing the halide ion into solution, which can then be tested using acidified silver nitrate

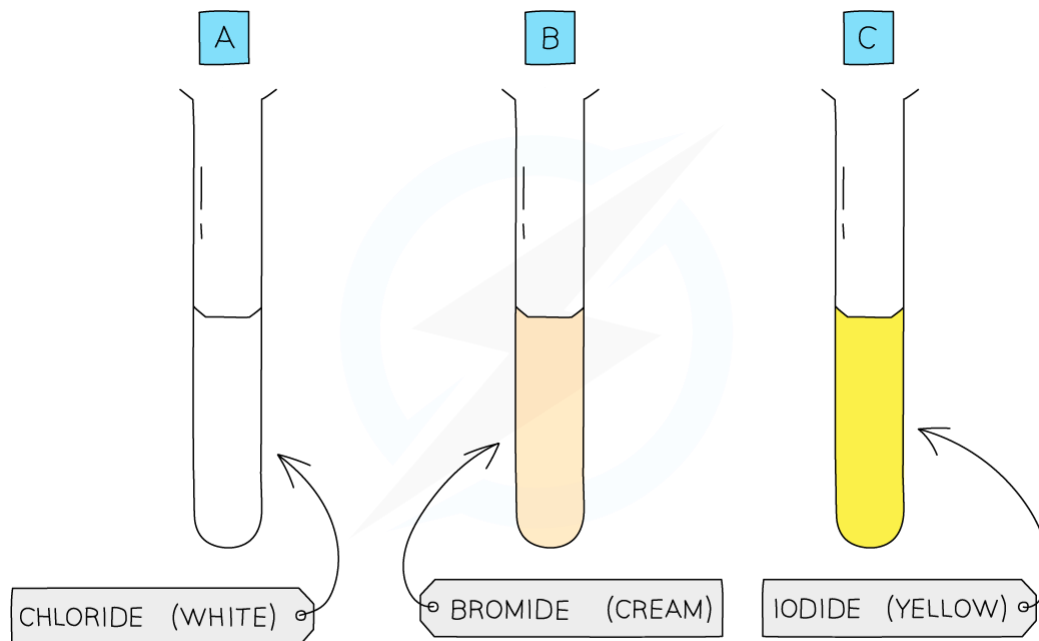
Positive test result:

- If a halide is present it forms a silver halide precipitate:



- Depending on the halide present, a different coloured precipitate is formed, allowing for identification of the halide ion
 - Silver chloride forms a white precipitate
 - Silver bromide forms a cream precipitate
 -

Silver iodide forms a yellow precipitate



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Each silver halide produces a precipitate of a different colour

- It can sometimes be hard to distinguish between the colours of the precipitates
- In this case, ammonia can be added
 - The white silver chloride precipitate will dissolve in dilute ammonia
 - The cream silver bromide precipitate will dissolve in concentrated ammonia
 - The yellow silver iodide precipitate is insoluble in both dilute and concentrated ammonia

Testing for carbonyls (general)

- The carbonyl group undergoes a **condensation** reaction with 2,4-dinitrophenylhydrazine

Positive Test Result:

- The **product** formed when 2,4-DNPH is added to a solution that contains an aldehyde or ketone is a **deep-orange precipitate** which can be purified by recrystallisation



NEGATIVE TEST WITH 2,4-DNPH
(UNKNOWN SUBSTANCE IS NOT
AN ALDEHYDE/KETONE)

POSITIVE TEST WITH 2,4-DNPH
(UNKNOWN SUBSTANCE IS
AN ALDEHYDE/KETONE)

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The test tube on the left shows a negative 2,4-DNPH test and the tube on the right shows a positive test

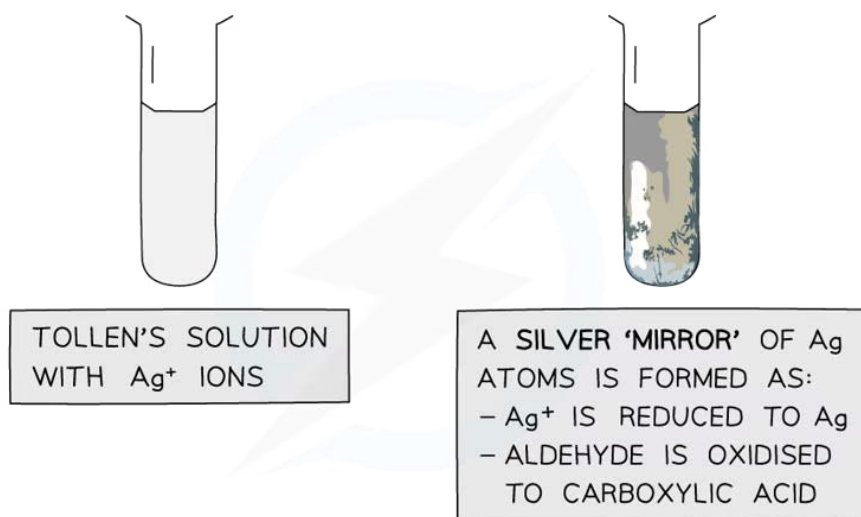
- The **melting point** of the formed precipitate can then be measured and compared to literature values to find out which specific aldehyde or ketone had reacted with 2,4-DNPH

Testing for Carbonyls (aldehyde / ketone specific)

- Tollens' reagent, also known as ammoniacal silver nitrate, is a more specific test tube reaction that can distinguish between aldehydes and ketones

Positive Test Result:

- When Tollens' reagent is gently warmed with an aldehyde, the silver mirror is formed
 - This is the positive test result
- When Tollens' reagent is gently warmed with a ketone, no silver mirror will be seen, as the ketone cannot be oxidised by Tollens' reagent, so no reaction takes place
- This is a negative test result



The Ag^+ ions in Tollens' reagent are oxidising agents, oxidising the aldehyde to a carboxylic acid and getting reduced themselves to silver atoms

Testing for carboxylic acids

- Solid sodium carbonate, Na_2CO_3 (s), or aqueous sodium hydrogen carbonate, NaHCO_3 (aq), can be used to test for the presence of a carboxylic acid

Positive Test Result:

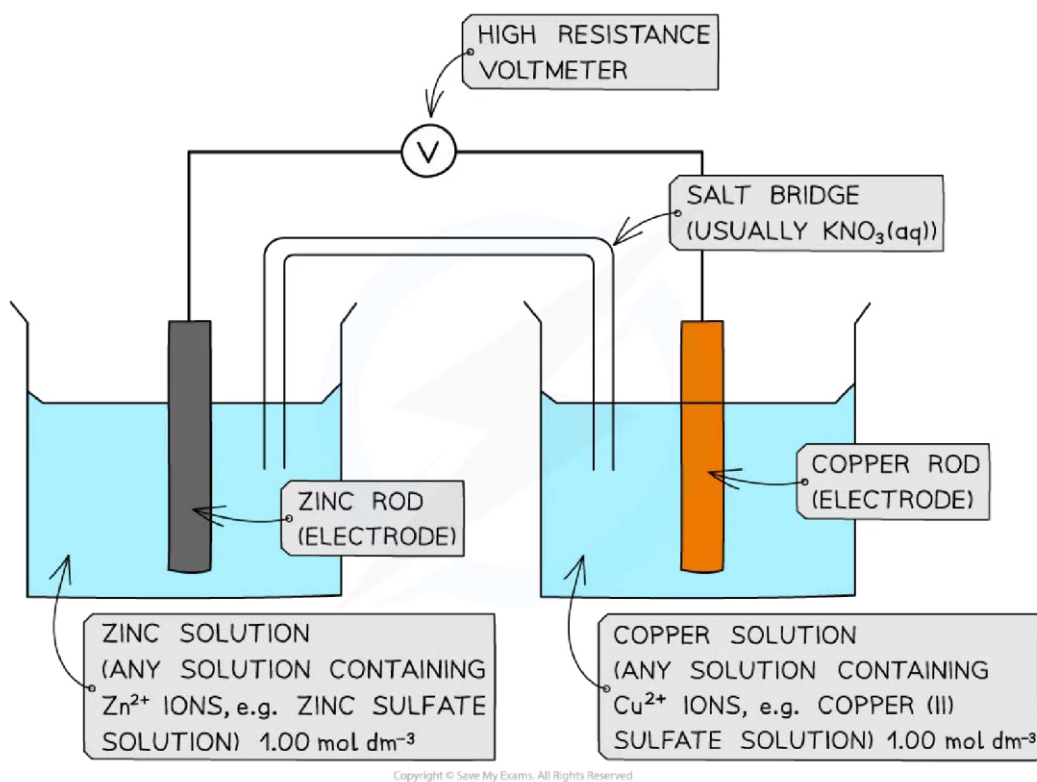
- Effervescence / bubbles of gas are seen as carbon dioxide is evolved

1.3.5 Electrochemical Cells



PAG 8: Electrochemical Cells

- To measure a cell EMF you will need
 - Two small beakers, around 75 cm³ capacity
 - Strips of suitable metals such as copper, zinc, iron and silver
 - 1.0 mol dm⁻³ solutions of the metal ions (nitrates, chlorides or sulfates depending on their solubility)
 - A high resistance voltmeter (usually a digital multimeter has this)
 - Two sets of wires with crocodile clips
 - A salt bridge consisting of a strip of filter paper soaked in saturated potassium nitrate



The experimental set up for measuring the EMF of a cell made of two metal / metal ion half cells

Steps in the procedure

- The strips of metals need to be freshly cleaned to remove any oxide coatings
 - This can be done with a piece of sandpaper
-
- To support the metals, it is easiest to have long strips that can be folded over the side of the beaker and held in place with the crocodile clips

Fill up the beakers to about two thirds of the way with the metal ion solutions • Using tongs, dip a strip of filter paper into a beaker of saturated potassium nitrate solution and then place it between the two beakers making sure the ends of the strip are well immersed in the solutions

- Connect the crocodile clips to the voltmeter, wait for a steady reading and record the measurement

Practical tips

- If you don't get a positive reading on the voltmeter swap the terminals around Voltmeters will have marked positive and negative terminals (usually in red and black, respectively), so when you get a positive reading this tells you the relative polarity of the metals in the cell
- Change the salt bridge each time, to prevent cross contamination of ions between half cells

Specimen Results

- Here is a set of typical results for this experiment

Specimen EMF Results (theoretical values) Table

Negative electrode	Positive electrode	EMF / V
Zn(s) / Zn ²⁺ (aq)	Cu ²⁺ (aq) / Cu(s)	1.10
Zn(s) / Zn ²⁺ (aq)	Fe ²⁺ (aq) / Fe(s)	0.32
Fe(s) / Fe ²⁺ (aq)	Cu ²⁺ (aq) / Cu(s)	0.78
Zn(s) / Zn ²⁺ (aq)	Ag ⁺ (aq) / Ag(s)	1.56
Cu(s) / Cu ²⁺ (aq)	Ag ⁺ (aq) / Ag(s)	0.46

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Analysis

- It is unlikely you will get very close to the theoretical results as these would be obtained under standard conditions which are hard to achieve in a school laboratory
- However, the **relative** EMF of cells you construct should match the theoretical values
- The higher the EMF, the larger the difference in reactivity ('electron pushing power') between the metals

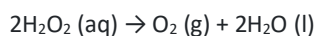
1.4 Further Physical Chemistry Practicals

1.4.1 Rate of Decomposition of Hydrogen Peroxide

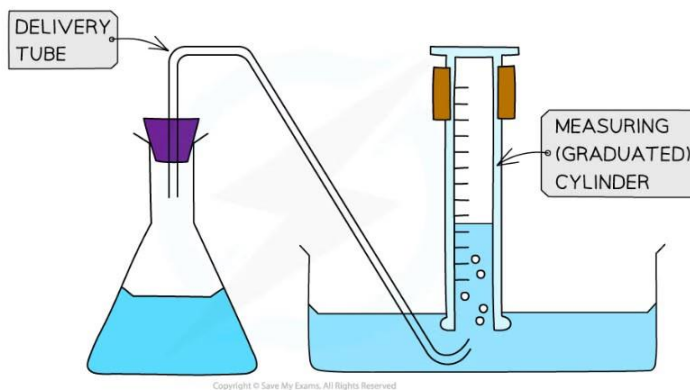
PAG 9.1 Rate of Reaction Decomposition of Hydrogen Peroxide

The Decomposition of Hydrogen Peroxide

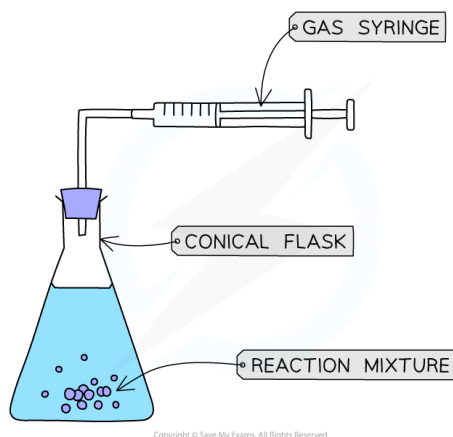
- At room temperature, hydrogen peroxide solution decomposes slowly to form water and oxygen



- The rate of this decomposition reaction can be monitored by measuring the volume of oxygen gas released over time
 - This experiment can be performed using:
 - Downward displacement of water - bubbling the oxygen into an upturned measuring cylinder / burette that is placed in a trough of water



- A gas syringe



- In both cases, the volume of gas produced may need to be considered so that it doesn't exceed the volume of the measuring cylinder / burette / gas syringe
- Since this reaction is slow, a catalyst is often added to speed up the reaction

- Common catalysts for this reaction include iron, liver and the enzyme catalase However, the
- most common and OCR exam board specified catalyst for this reaction is powdered manganese(IV) oxide, MnO_2 (s)

Method:

- Add 50 cm^3 of hydrogen peroxide solution into a conical flask
- Fill a fresh measuring cylinder with water, seal the top with your finger, and invert it into the water trough
- Loosely connect the bung into the conical flask and make sure the delivery tube connects to the upturned measuring cylinder
- Measure 0.5 g of manganese(IV) oxide
- Add 0.5 g of manganese(IV) oxide into the conical flask, replace the bung and start the stopwatch
Record the volume of gas released every 10 seconds. Continue timing until no more oxygen appears
- to be given off
 - It may be necessary to perform a rough initial experiment to determine the potential maximum volume of oxygen released and adjust the size of the upturned measuring cylinder accordingly

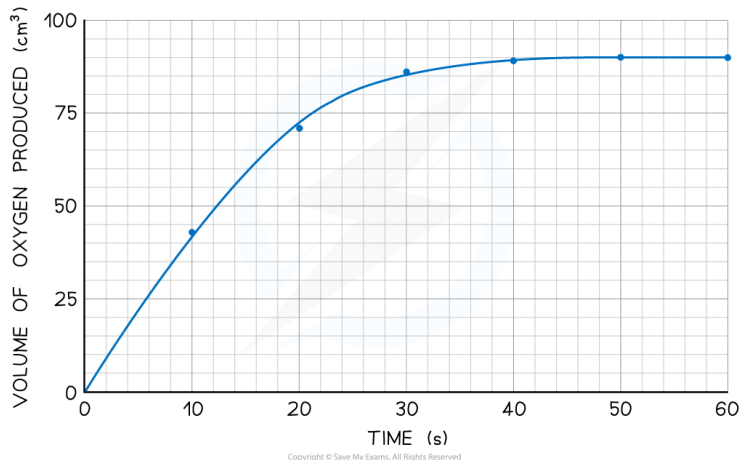
Specimen results

- Here is a set of typical results for this experiment

Rate of oxygen production in the MnO_2 catalysed decomposition of hydrogen peroxide table

Time (s)	10	20	30	40	50	60
Volume of oxygen produced (cm^3)	43	71	86	89	90	90

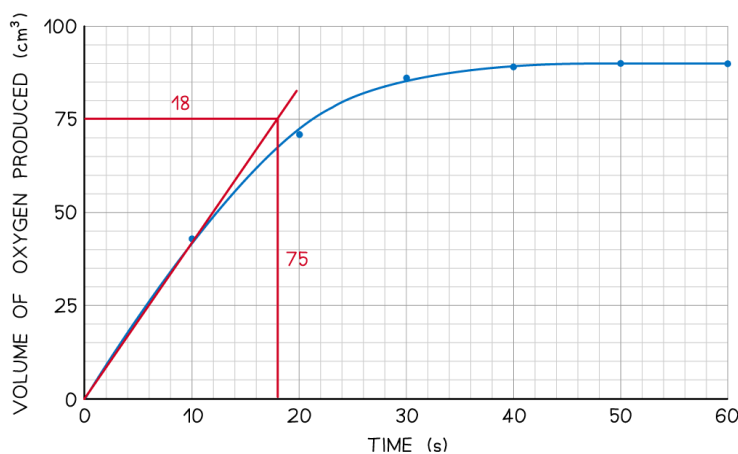
Graphing the results



Analysis

- The curve of best fit is drawn on the graph
- A tangent can then drawn starting from (0,0) to find the **initial rate** of reaction

- The gradient of the tangent is calculated
 - This is the rate of reaction



- In the example above, the rate of reaction is:

- $$\text{Gradient} = \frac{\Delta y}{\Delta x} = \frac{75}{18} = 4.17 \text{ cm}^3 \text{ s}^{-1}$$

? Exam Tip

Extend your tangent to be as long as possible

- This is because shorter tangent lines have the greatest potential for error
If your tangent line is longer then you are less likely to be outside the examiner's accepted range of answers

- The example above is the OCR specified practical for PAG 9.1
One simple variation of this practical could be using a variety of catalysts and comparing rates
- Another variation, although it has **never yet** been seen in exams and most teaching classrooms, is that this practical could potentially be performed (or examined) by measuring the mass lost from the conical flask
 - This could potentially bring in other aspects of practicals:
 - Would the rate be too high / low to get meaningful results? Would powdered
 - catalysts be lost up the sides / out of the top of the conical flask? Therefore, affecting the results
 - Would the volume of oxygen produced cause a high enough mass loss? Does the
 - concentration of hydrogen peroxide need to be considered / calculated to ensure that enough mass is lost for meaningful results?

1.4.2 Rate of Reaction - Calcium Carbonate & Hydrochloric Acid

PAG 9 2 Rate of Reaction Calcium Carbonate & Hydrochloric Acid

The Reaction of Calcium Carbonate and Hydrochloric Acid

- At GCSE level, this reaction is a standard experiment to see the effect of changing the surface area of a reactant on the rate of reaction
 - Three sizes of calcium carbonate (marble) are commonly used:
 - Large chips
 - Small chips
 - Powder
- The reaction itself cannot change



- However, the application and analysis of the results shifts more towards the mathematical skills of accurately plotting graphs, drawing tangents and calculating gradients
- This reaction can be monitored by measuring:
 1. The volume of carbon dioxide gas produced
 2. The mass loss from the reaction vessel

Method 1 - Volume of CO₂ produced

1. Support a gas syringe with a stand, boss and clamp.
 2. Add 50 cm³ of dilute hydrochloric acid to a conical flask
 3. Loosely connect the gas syringe
 4. Measure 0.40 g of calcium carbonate
 5. Add the 0.40 g of calcium carbonate into the conical flask, replace the gas syringe and start the stopwatch
 6. Record the volume of gas produced every 10 seconds. Continue timing until no more carbon dioxide appears to be given off
- Another method to achieve these results would be the downward displacement of water method, where an upturned measuring cylinder of water is placed in a water trough
 - This method still records the volume of carbon dioxide produced

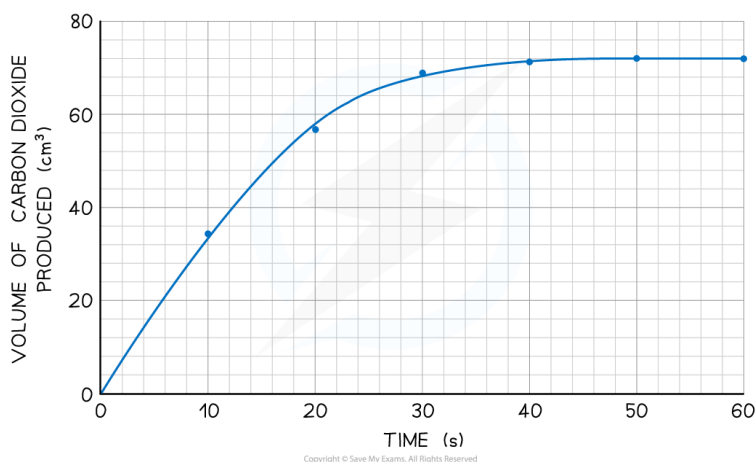
Specimen results 1 - Volume of CO₂ produced

- Here is a set of typical results for this experiment

Rate of carbon dioxide production in the reaction of calcium carbonate and hydrochloric acid table

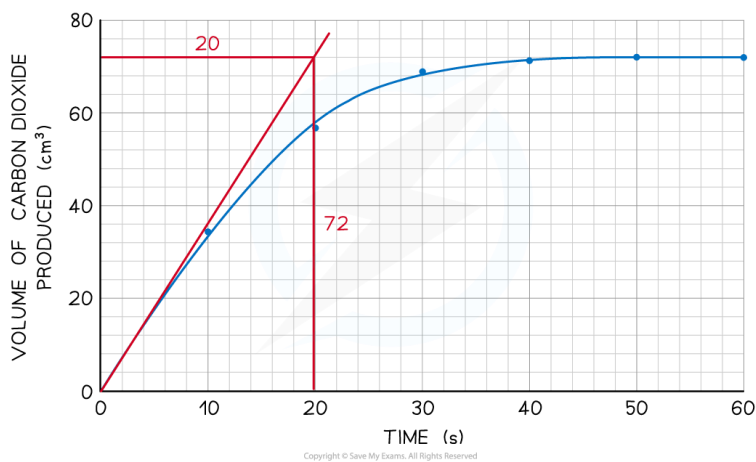
Time (s)	10	20	30	40	50	60
Volume of CO ₂ produced (cm ³)	34	57	69	71	72	72

Graphing the results 1 - Volume of CO₂ produced



Analysis 1 - Volume of CO₂ produced

- The curve of best fit is drawn on the graph
- A tangent can then be drawn starting from (0,0) to find the **initial rate** of reaction
- The gradient of the tangent is calculated
 - This is the rate of reaction



- In the example above, the rate of reaction is:

$$\text{Gradient} = \frac{\Delta y}{\Delta x} = \frac{72}{20} = 3.6 \text{ cm s}^{-1}$$

$$\frac{72}{20}$$

Method 2 - Mass of the reaction vessel

1. Measure 0.40 g of calcium carbonate into a weighing boat
2. Add 50 cm³ of dilute hydrochloric acid to a conical flask
3. Place the conical flask of hydrochloric acid **AND** the weighing boat of calcium carbonate onto the balance
4. Measure the combined mass and record this as the t = 0 result

5. Add the 0.40 g of calcium carbonate into the conical flask, replace the weighing boat onto the balance and start the stopwatch
6. Record the mass every 10 seconds. Continue timing until no further mass is lost
 - It may be necessary to use more dilute concentrations of hydrochloric acid because it may not be realistically possible to record the mass every second
 - A data logger could be connected to the balance (if available) and this can track the mass over time

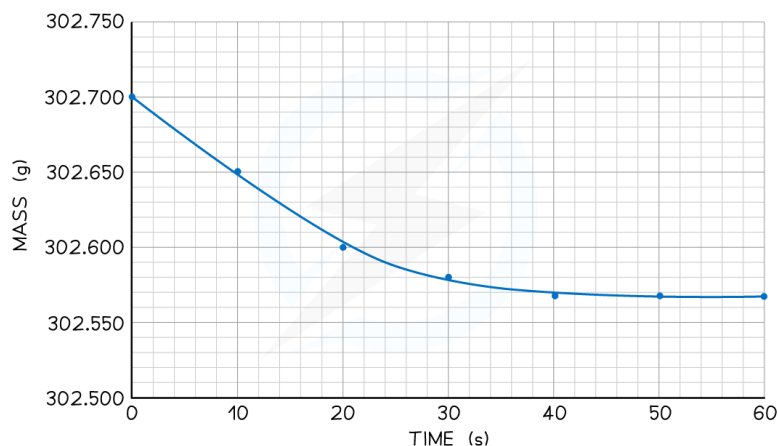
Specimen results 2 - Mass of the reaction vessel

- Here is a set of typical results for this experiment

Rate of change in mass in the reaction of calcium carbonate and hydrochloric acid table

Time (s)	0	10	20	30	40	50	60
Mass (g)	302.700	302.650	302.600	302.580	302.568	302.568	302.568

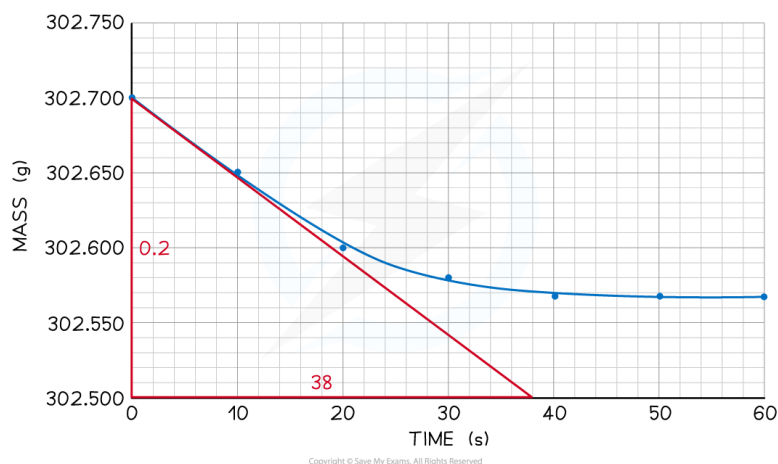
Graphing the results 2 - Mass of the reaction vessel



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Analysis 2 - Mass of the reaction vessel

- The curve of best fit is drawn on the graph
- A tangent can then be drawn starting from (0,0) to find the **initial rate** of reaction
- The gradient of the tangent is calculated
 - This is the rate of reaction



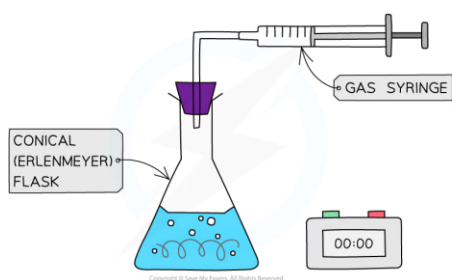
In the example above, the rate of reaction is:

- $$\text{Gradient} = \frac{\Delta y}{\Delta x} = \frac{-0.2}{38} = (\text{mass loss of}) 5.26 \times 10^{-3} \text{ g s}^{-1}$$
- A simpler version of this practical can be done by hitting the TARE / reset button on the balance when the calcium carbonate is added
 - This then records the mass lost and can be used to give a graph with a curve more like the volume of CO₂ produced graph

1.4.3 Reaction - Magnesium & Hydrochloric Acid

PAG 9 3 Rate of Reaction Magnesium & Hydrochloric Acid

- This reaction can be used to investigate the effect of varying the concentration of the acid while keeping the temperature constant
- When a gas is released in a reaction you can either try to measure the volume of gas given off or the mass change in the reaction flask
 - However, in this case the gas hydrogen is too low in density so the mass change will be far too small to register on a laboratory balance
- Volume can be measured either by displacement of water into an inverted measuring cylinder or by using a gas syringe
 - The choice of the size of the gas syringe needs to be considered and the quantities of reagents judged accordingly so that a reasonable volume of gas can be evolved and also recorded



The rate of reaction between magnesium and dilute hydrochloric acid can be measured using a gas syringe and stopwatch Steps in the procedure

- Before you begin you need to check your apparatus is gas tight
 - This can be done by assembling everything without the acid or magnesium and trying to move the plunger
 - If you feel resistance the apparatus is gas tight
- The acid will go in first because it is quicker to drop a piece of magnesium ribbon in than to pour in the acid
- To vary the concentration of the acid you need to dilute it by measuring portions of acid in a measuring cylinder and then portions of distilled water in another measuring cylinder and adding them to the conical flask
- Choose a suitable volume of acid to match the size of the flask, e.g. 40 cm³ You don't want to use
- acid that is stronger than 2 mol dm⁻³ because the reaction will be too fast, so its best to start with 2 mol dm⁻³ and perform a serial dilution, e.g. 40 cm³(acid) + 0 cm³ (water), 35 cm³ (acid) + 5 cm³ (water), etc

Practical tips

- Make sure the plunger is fully inserted before you start the experiment otherwise you will have a volume error

- If the magnesium does not look new and shiny, you may need to clean the surface with a bit of sandpaper
- Make sure the plunger is secure and does not fall out of the barrel if the volume exceeds 100 cm³

Specimen Results

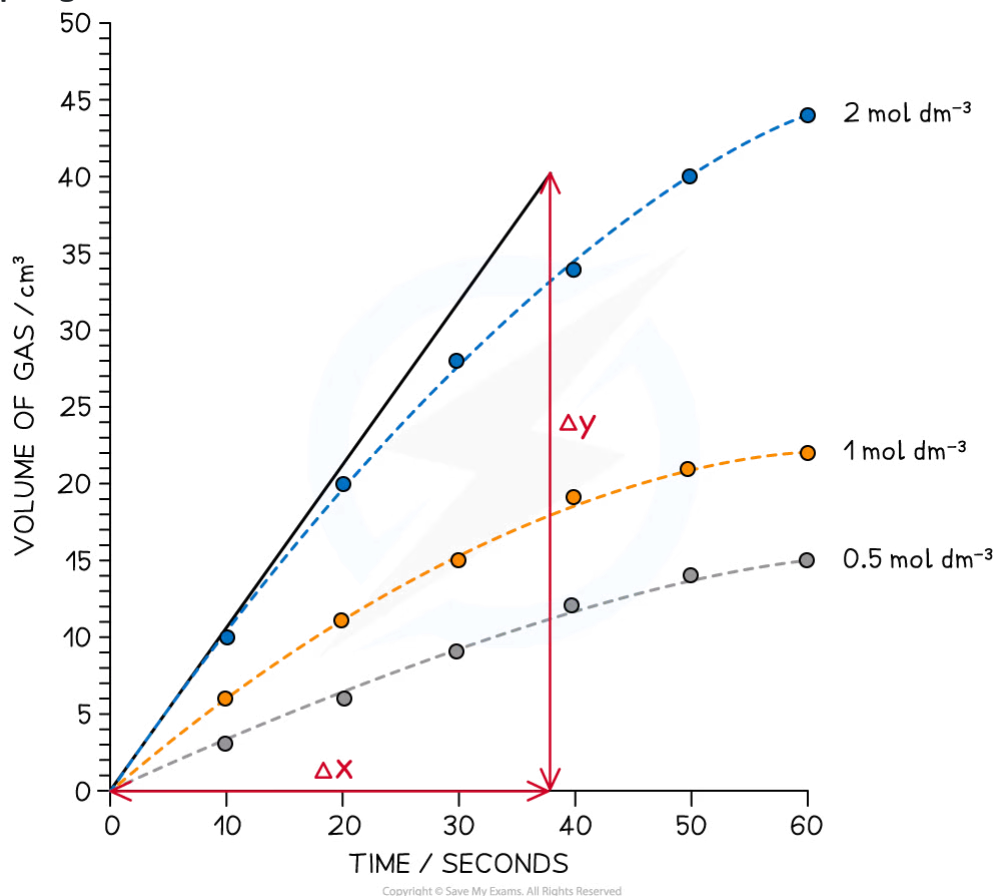
- Here is a set of typical results for this experiment

Rate of reaction between magnesium and acid results table

	Volume of gas / cm ³		
Time /seconds	2.0 mol dm ⁻³	1.0 mol dm ⁻³	0.5 mol dm ⁻³
0	0	0	0
10	10	6	3
20	20	11	6
30	28	15	9
40	34	19	12
50	40	21	14
60	44	22	15

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Graphing the results



Graph showing typical results for the rate of reaction between hydrochloric acid and magnesium at different concentrations

Analysis

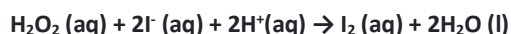
- The lines of best fit are drawn for each concentration on the same graph
- A tangent is then drawn starting from (0,0) since this method is to find the **initial rate** of reaction
- The gradient of the tangent is determined which gives the rate of reaction
- In the example above, the rate of reaction for 2.0 mol dm⁻³ acid is
- Gradient = $\frac{\Delta y}{\Delta x} = \frac{40}{38} = 1.05 \text{ mol dm}^{-3} \text{ s}^{-1}$

1.4.4 Rates – Iodine Clock

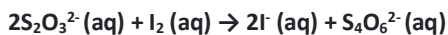
PAG 10 1 Rates Iodine Clock

The Iodine Clock Reaction

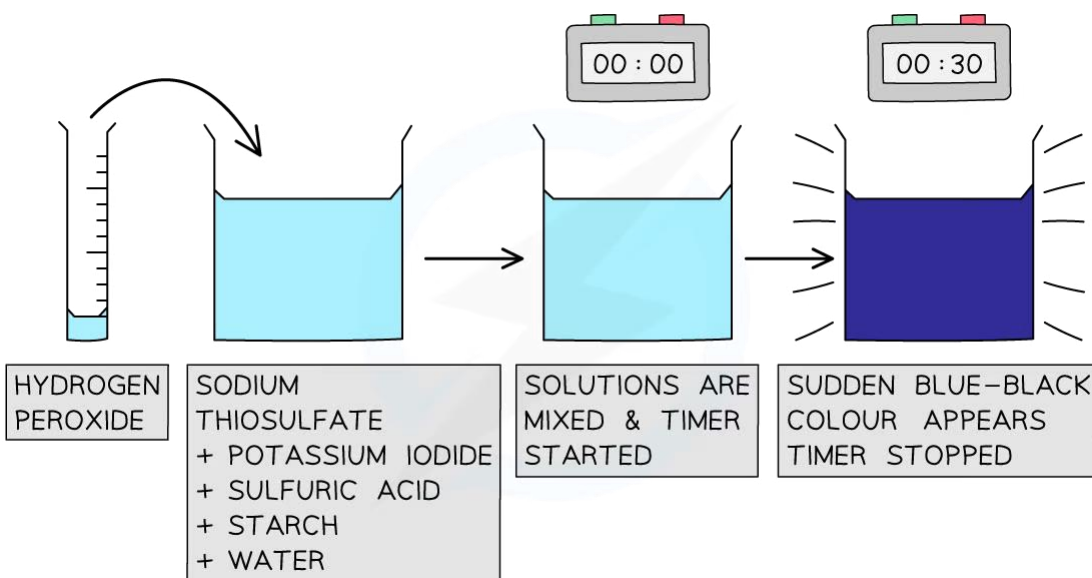
- Clock reactions are so called because they show a sharp dramatic colour change after a period of time has elapsed
- They make ideal reactions for studying kinetics
- Iodine clock reactions come in a number of variations, but they generally all use starch to show a sudden purple-black colour at the end of the reaction
- A common iodine clock reaction uses the reaction between hydrogen peroxide and iodine



- Adding sodium thiosulfate to the reaction mixture uses up the iodine and acts as the reaction timer



- The amounts chosen are such that the iodine produced is in **excess** compared to the other reagents
 - Therefore, as soon as the iodine is in excess the blue-black colour of iodine in starch is seen



The iodine clock reaction provides a good way to study reaction kinetics

Steps in the procedure

- The solutions are measured in burettes and placed in a small beaker
- The sulfuric acid is in excess so can be measured in a measuring cylinder rather than burette

- The reaction is started by adding 1cm³ of 0.25 mol dm⁻³ hydrogen peroxide and starting a timer
- The timer is stopped when the blue black colour appears Suitable volume
- compositions to use could be as follows:

Iodine clock volume compositions table

Run	0.01 mol dm ⁻³ Na ₂ S ₂ O ₃ (aq)/cm ³	H ₂ O / cm ³	0.5 mol dm ⁻³ KI(aq) / cm ³	0.05 mol dm ⁻³ H ₂ SO ₄ / cm ³	Starch solution / cm ³
1	1	4	1	25	1
2	1	3	2	25	1
3	1	2	3	25	1
4	1	1	4	25	1
5	1	0	5	25	1

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Practical tips

- Hydrogen peroxide is typically found in 'volume' concentrations, based on the volume of oxygen given off when it decomposes:



- For example in school laboratories, a suitable concentration of hydrogen peroxide may be listed as 3% or '10 vol'
 - '10 vol' means that when 1cm³ of hydrogen peroxide decomposes it generates 10 cm³ of oxygen
 - '10 vol' or 3% hydrogen peroxide has a concentration of 0.979 mol dm³

Specimen Results

- Here is a set of typical results for the iodine clock reaction

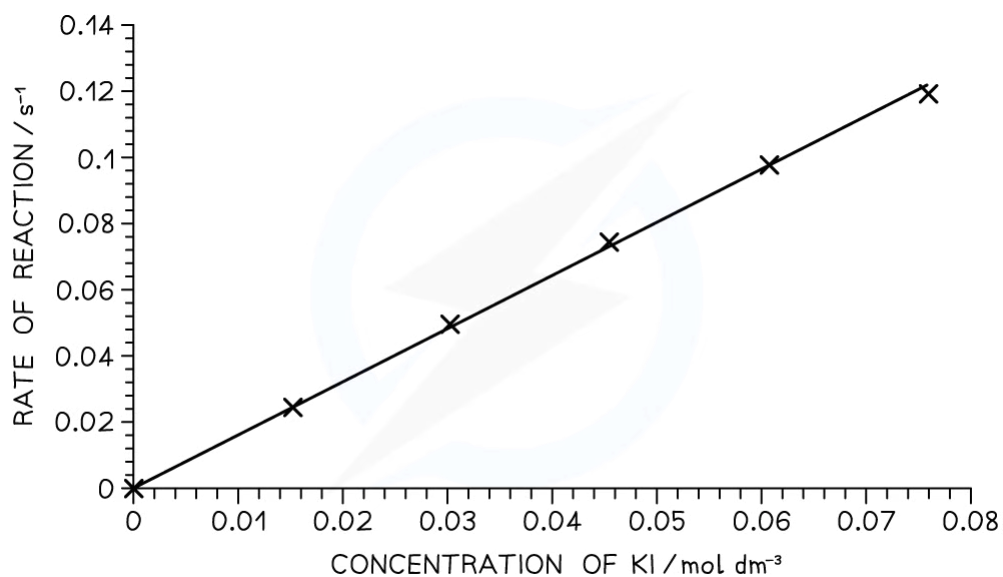
Specimen results for the iodine clock reaction table

Concentration of potassium iodide / $\text{mol dm}^{-3} \times 10^{-2}$	Time for blue colour to appear / s	Rate, $1/t \text{ s}^{-1}$
1.515	40	0.025
3.030	20	0.050
4.545	13	0.075
6.060	10	0.100
7.576	8	0.120

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Analysis

- The time of reaction is converted to rate of reaction by calculating the reciprocal value
- A graph is plotted of rate versus concentration



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A rate-concentration graph for the iodine clock reaction

- From this graph we can see that the rate of reaction is directly proportional to the concentration of potassium iodide:

o

For more help, please visit www.exampaperspractice.co.uk



As concentration doubles; the rate of reaction also doubles

This tells us that the reaction is first order with respect to potassium iodide



1.4.5 Rates - Thiosulfate

PAG 10 2 Rates Thiosulfate & Acid

The Disappearing Cross Experiment

- A simple experiment which can be done to determine how the rate of reaction is affected by concentration is the disappearing cross experiment
- This experiment can be done for a number of different reactions, but the following reaction is commonly used:



In this reaction, sodium thiosulphate reacts with hydrochloric acid

The key product in this experiment is the solid sulfur which causes the solution to become opaque

There are two factors which can be easily investigated using the disappearing cross reaction:

Changing the concentration - this can be for the hydrochloric acid or the sodium thiosulfate solution

Changing the temperature (OCR tend to use this one to calculate the activation energy of a reaction)

Volume of sodium thiosulfate (cm ³)	Volume of water (cm ³)	Relative dilution
---	------------------------------------	-------------------

10	40	0.2
----	----	-----

? Exam Tip

The disappearing cross experiment can be used for any reaction where a solid is produced as one of the products, as this will cause the solution to become cloudy.

- It does not have to be done in a conical flask, it could be done in a test tube with the cross placed underneath / behind the test tube. **Careful:** This is a very simple
- experiment, but exam questions can take it in many different directions from a relatively straight forward rate calculation to the more complex Arrhenius plot and equation to determine activation energy

Method

1. Measure 50.0 cm³ of sodium thiosulfate solution into a conical flask
2. Place the conical flask on the black cross
3. Measure 5.0 cm³ of dilute hydrochloric into a measuring cylinder
4. Add the hydrochloric acid to the conical flask and immediately start the stopwatch
5. Swirl the mixture
6. Stop the stopwatch when the cross disappears and record the time
7. Repeat the experiment for a minimum of four more / different concentrations

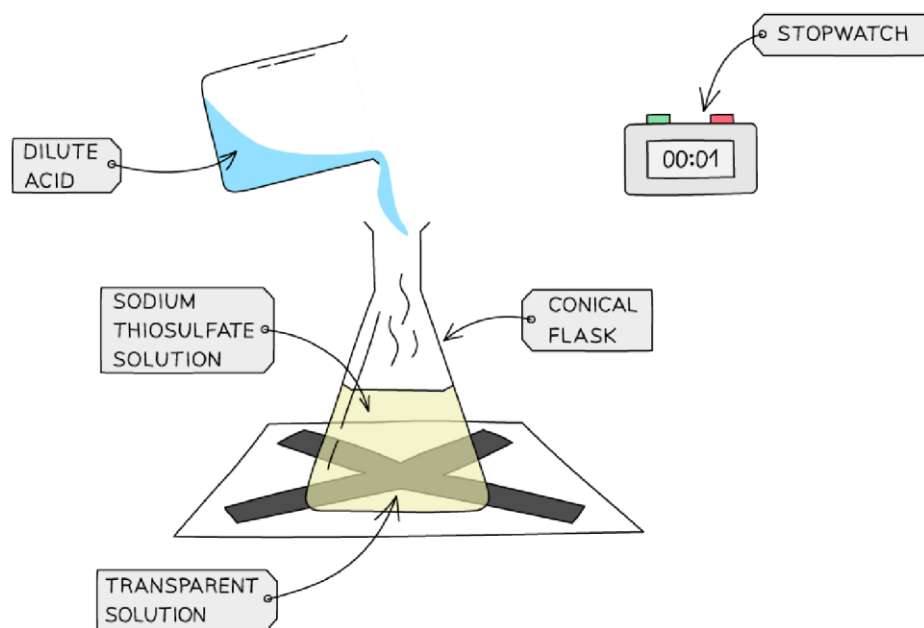
Dilution of sodium thiosulfate solution table

20	30	0.4
30	20	0.6
40	10	0.8
50	0	1.0

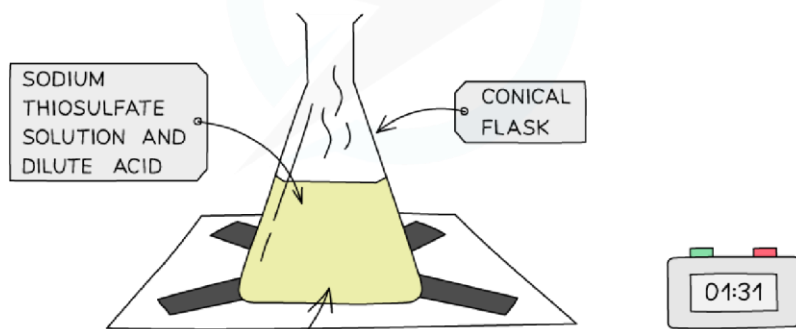
- It is more likely that you would be given a known concentration of sodium thiosulfate solution, typically 0.25 mol dm³, and asked to create different concentrations by diluting that solution

○
volume **must** remain constant when diluting

1 ADD ACID TO SODIUM THIOSULFATE SOLUTION, START TIMER.



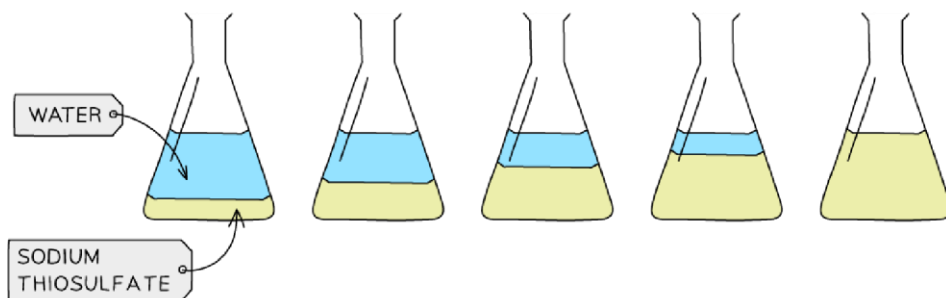
2 STOP TIMER WHEN 'X' IS NO LONGER VISIBLE. RECORD TIME.



Careful: The process is the same as outlined in the table above where the total

OPAQUE SOLUTION

3 REPEAT STEPS 1–2 WITH DIFFERENT CONCENTRATIONS OF SODIUM THIOSULFATE (MADE BY MIXING DIFFERENT VOLUMES OF WATER AND SODIUM THIOSULFATE)



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Diagram showing the apparatus needed to investigate reaction rate in the disappearing cross experiment

Specimen Results

- Results could be expressed in terms of relative dilution of the sodium thiosulfate solution (less common) or in terms of concentration of the sodium thiosulfate solution

Relative dilution of sodium thiosulfate solution	Time for cross to disappear (s)
0.2	115.2
0.4	57.6
0.6	30.0
0.8	15.6
1.0	7.2
Concentration of sodium thiosulfate solution (mol dm ⁻³)	Time for cross to disappear (s)
0.05	115.2
0.10	57.6
0.15	30.0
0.20	15.6
0.25	7.2

Analysis

- These results could be used in a number of different ways:
 1. Plot a graph of the results with the concentration of sodium thiosulfate on the x-axis and time on the y-axis
 2. Comment on the effect of changing concentration (this is more GCSE level) Increasing the
 - concentration of the sodium thiosulfate solution increases the rate of reaction
This means that the time for the cross to disappear decreases
 - 3. Use the graph to determine the order of the reaction with respect to sodium thiosulfate
 - The shape of the plotted graph will indicate if the reaction is 0, 1 or 2 order
- If this reaction was performed at different temperatures instead of with different concentrations, then the results could be manipulated, plotted and used to calculate the activation energy for this reaction

1.4.6 Rates - Activation Energy

PAG 10 3 Rates Activation Energy

The Ethanedioic Acid and Potassium Manganate(VII) Reaction

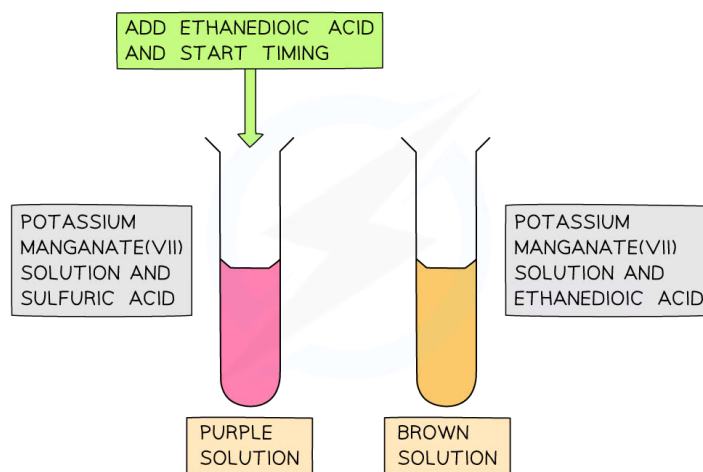
- A less common experiment to determine the activation energy of a reaction is the reaction between potassium manganate(VII) solution and ethanedioic acid



- In this reaction, potassium manganate(VII) with ethanedioic acid in the presence of sulfuric acid
- The key point in this experiment is the colour change of the manganese which changes from purple to brown

Method

- Using a different / clean measuring cylinder for each chemical, transfer 10.0 cm³ of each of the following into a separate boiling tube
 - 0.1 mol dm⁻³ potassium manganate(VII) solution, KMnO₄ (aq)
 - 2.0 mol dm⁻³ sulfuric acid, H₂SO₄ (aq)
 - 0.25 mol dm⁻³ ethanedioic acid, H₂C₂O₄ (aq)
- Warm / heat all three boiling tubes in a water bath
- Place a test tube in a test tube rack
- Measure and record the temperature of all three solutions
- Add 0.5 cm³ of the potassium manganate(VII) solution to the test tube
- Add 0.5 cm³ of the sulfuric acid to the test tube
- Then add 1.0 cm³ of the ethanedioic acid to the test tube
- Immediately start the stopwatch and swirl the mixture
- Stop the stopwatch when the purple colour changes to pale brown and record the time
- Repeat the experiment for a minimum of four more / different temperatures



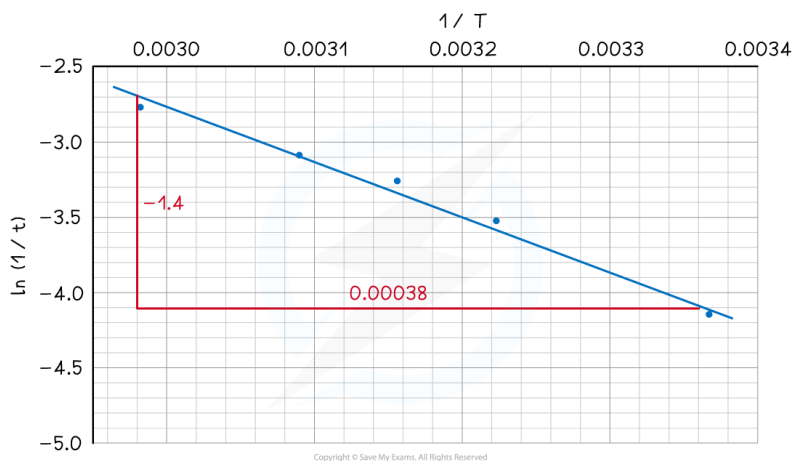
The Ethanedioic Acid and Potassium Manganate(VII) Reaction

Specimen Results

Temperature, T (K)	1 / T	Time, t (s)	1 / t	ln (1 / t)
297.0	0.003367	63	0.015873	-4.1431
310.3	0.003223	34	0.029412	-3.5264
316.9	0.003156	26	0.038462	-3.2581
323.6	0.003090	22	0.045455	-3.0910
335.3	0.002982	16	0.062500	-2.7726

- The temperature of the reaction is converted to $1 / T$ by calculating the reciprocal value
The rate is found by calculating the reciprocal of time, t
- This is then converted into the natural logarithm function to give $\ln(1 / t)$ or $\ln(\text{rate})$
- A graph of $\ln(\text{rate})$ versus $1 / T$ is then plotted:

Analysis



An $\ln(\text{rate})$ versus $1 / T$ graph for the Ethanedioic Acid and Potassium Manganate(VII) Reaction

- From this graph, we can see that the line of best fit is a straight line
 - The equation for a straight line is $y = mx + c$
 - The Arrhenius equation can be rearranged to $\ln k = \ln A - E_a/RT$
 - Therefore, the gradient of the line is $-E_a / R$
- This means that we can use the gradient from the graph to calculate the activation energy for this reaction
 - Gradient = can be rearranged to $E_a = -\text{gradient} \times R$
 - $$\frac{-1.4}{0.00038} \quad E_a = -x 8.31 = 30616 \text{ J mol}^{-1}$$
 - 0.
 - $$E_a = \frac{30616}{1000} = 30.616 \text{ kJ mol}^{-1}$$

1.4.7: pH – Problem Solving

PAG 11 1 pH Problem Solving

Testing the pH of different solutions

- According to the Practical Activities Support Guide published by OCR:
 - In PAG 11.1, students will be given a number of colourless solutions that could be acids and / or alkalis
 - Students are to plan and implement a practical procedure to work out the identity of each solution
- However, the key skill / technique for Practical Activity Group 11 is the measurement of pH using charts, meters and probes attached to data loggers Therefore, the aim of this practical will be to test
- a range of solutions using a combined sequence of tests

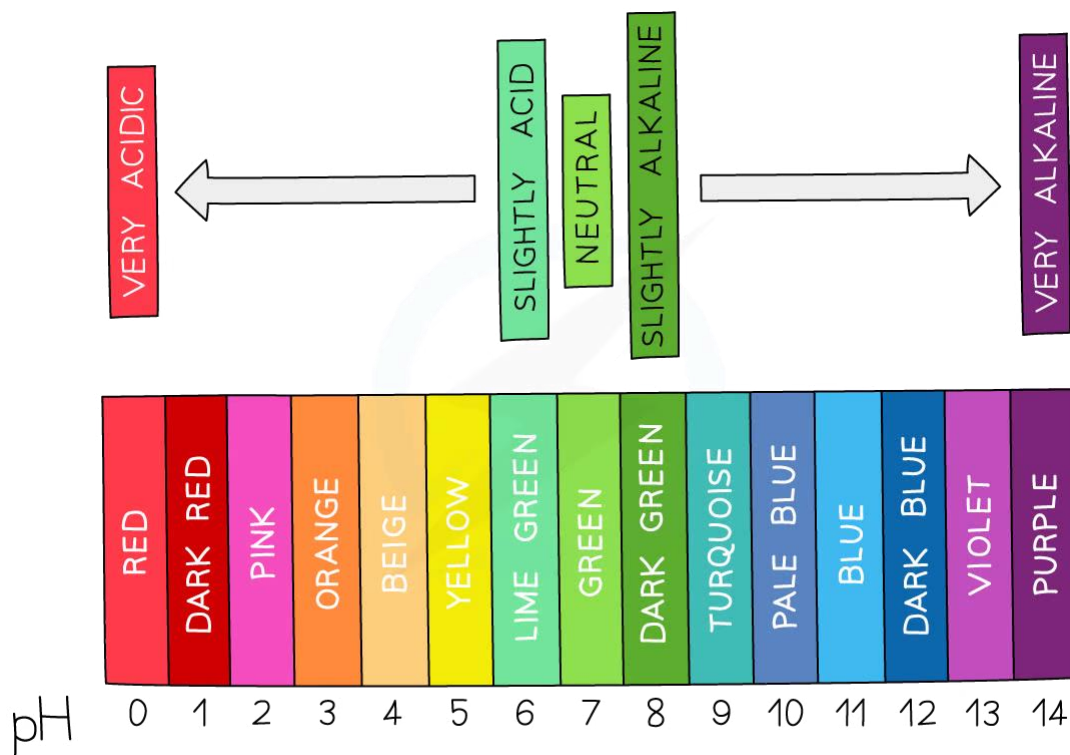
The solutions given could be the following:

- | Solution | Approximate pH |
|---------------------------|----------------|
| Ammonia | 11.00 |
| Ethanoic acid | 2.90 |
| Hydrochloric acid | 1.10 |
| Sulfuric acid | 1.00 |
| Methanoic acid | 2.40 |
| Sodium hydroxide | 13.00 |
| Ethanoate buffer solution | 4.76 |

Method

Test 1 - Addition of universal indicator

- Add 5 cm³ of each solution to separate test tubes
- Add universal indicator solution to each test tube
- Record the pH's of each solution in a results table
 - Use the colour chart to identify the correct pH



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Universal indicator pH chart

Test 2 - Using a pH probe

- Before completing this test, the pH probe will need calibrating
 - After storage, a pH meter does not give accurate readings because the glass electrode in the pH meter does not give a reproducible emf over longer periods of time
- Calibration of a pH probe
 - Rinse the pH probe with distilled water and shaken to remove excess water
 - Place the tip of the pH probe in pH 4 buffer solution, ensuring the bulb is fully immersed
 - Allow to sit until the pH stabilises
 - Adjust reading to the pH of the buffer
 - Repeat this process, including the washing with distilled water with a pH 9 buffer solution
- Once calibrated, the tip of the pH probe is rinsed with distilled water and shaken to remove excess water
- The tip is placed in the unknown solution, again, ensuring the bulb is fully immersed
-
-
- The pH is allowed to stabilise and then the reading recorded in a results table In practice,



recalibration may sometimes be required during testing This process can then be repeated for each unknown solution

Test 3 - Using phenolphthalein indicator

-
-
-
- Record observations in a results table

Specimen results

Solution	Test 1 - Using UI	Test 2 - pH probe	Test 3 - Phenolphthalein
Ammonia	Pale blue = pH 10	10.39	Turns pink
Ethanoic acid	Orange = pH 3	2.89	No change
Hydrochloric acid	Red = pH 1	1.20	No change
Sulfuric acid	Red = pH 1	1.46	No change
Methanoic acid	Pink = pH 2	2.30	No change
Sodium hydroxide	Purple = pH 14	13.60	Turns pink
Ethanoate buffer	Orange = pH 3	2.96	No change



Exam Tip

- Due to the potential variation in results, you would be given more guidance or more information about the unknown solutions in order to be able to identify them

This can be done with a spotting tile

Add a few drops of each solution to a labelled spotting tile (so each solution can be identified)

Add 1 drop of phenolphthalein in each well

1.4.8 pH – Titration Curves

PAG 11 2 pH Titration Curves

- Titration or pH curves are obtained by reacting known concentrations of acid with alkalis and measuring the pH during the neutralisation
- A suitable selection to use would be 1.0 mol dm⁻³ solutions of hydrochloric
 - acid ethanoic acid sodium hydroxide ammonia solution
 -
- A pH probe is calibrated by placing the tip of the probe in pH 4 buffer solution and adjusting until the reading is 4.0
 - The probe is washed in distilled water and then checked against pH 9 buffer
- If it is working correctly it should read 9.0 This is known as a **two-point calibration**
 -
- 25 cm³ of ethanoic or hydrochloric acid is measured using a volumetric pipette and filler and then transferred into a beaker
 - Its pH is measured and recorded
- The alkali (either sodium hydroxide or ammonia solution) is placed in the burette 5 cm³ at a time is added to the beaker and the pH measured after each addition, until a total of 50 cm³ has been added
- The procedure is repeated for all four combinations of acids and alkalis
-
-



Specimen Titration Curve Results Table



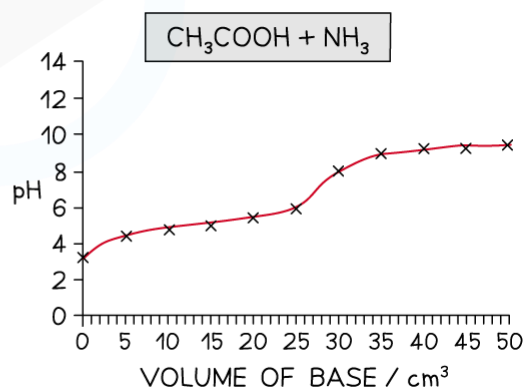
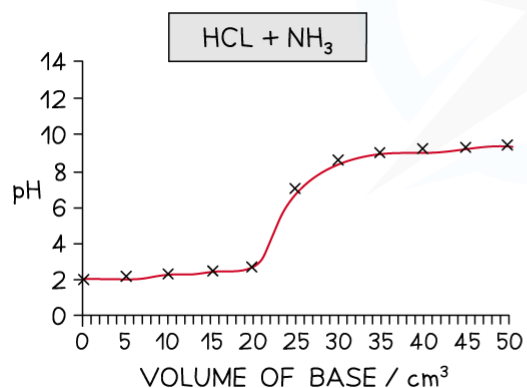
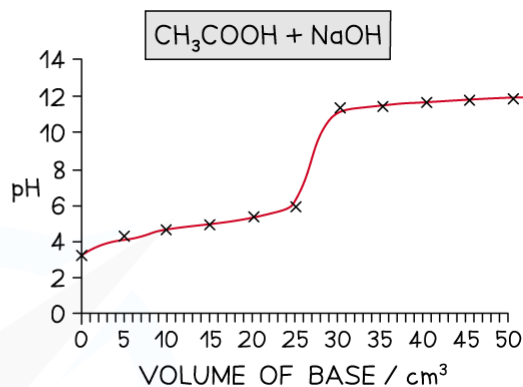
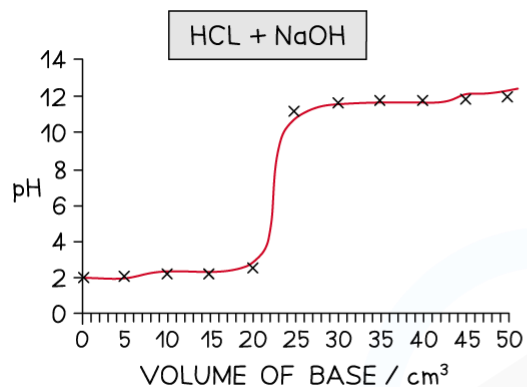
Volume of NaOH / cm ³	pH	
	HCl	CH ₃ COOH
0	2.0	3.3
5	2.1	4.3
10	2.2	4.7
15	2.3	5.0
20	2.6	5.4
25	11.2	6.0
30	11.7	11.5
35	11.8	11.5
40	11.8	11.8
45	11.9	11.9
50	12.0	12.0

Volume of NH ₃ (aq) / cm ³	pH	
	HCl	CH ₃ COOH
0	2.0	3.3
5	2.2	4.4
10	2.3	4.8
15	2.4	5.0
20	2.6	5.4
25	7.0	5.8
30	8.6	7.9
35	9.0	8.9
40	9.2	9.2
45	9.3	9.2
50	9.3	9.3

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Analysis

- The four characteristic titration curves are shown below
- The more data points that are available; the easier it is to draw the shape of the curves.
 - So, it is often better to add smaller portions nearer the equivalence point



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The four characteristic acid-base titration curves



Exam Tip

You need to:

- Be familiar with the shapes of the four titration curves
- Be able to identify the type of acid and base (weak/strong)
- Know where to locate the equivalence point

You also need to know the difference between the 'end point' and the 'equivalence point'

- End point = The point at which the indicator changes colour
- Equivalence point = The point at which stoichiometric (equal) amounts of acid and alkali have been added
 - This is found at the midpoint of the vertical section of a pH curve

1.4.9 pH – Acids & Buffers

PAG 11.3 pH Acids & Buffers

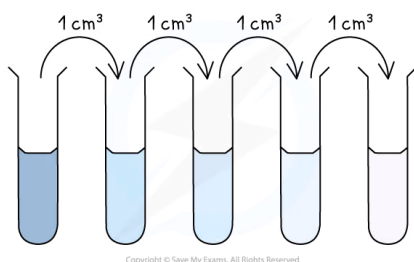
- PAG 11.3 comes in three parts:

For more help, please visit www.exampaperspractice.co.uk

1. Preparing hydrochloric acid solutions of a range of concentrations by dilution and measuring their pH values
2. Preparing a range of buffer solutions and measuring their pH values
3. Using both types of solutions, investigating how the pH changes with the addition of sodium hydroxide solution.

Preparing hydrochloric acid solutions with different concentrations

- This can be done by serial dilution
 - Serial dilution is any dilution in which the concentration decreases by the same factor in each successive step



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Serial dilution method

- Example 1
 - Adding 1.0 cm³ of HCl (aq) to 9.0 cm³ of distilled water is a 1 : 10 dilution
 - This means that the concentration will decrease by a factor of 10
- Example 2
 - Adding 1.0 cm³ of HCl (aq) to 4.0 cm³ of distilled water is a 1 : 5 dilution
 - This means that the concentration will decrease by a factor of 5

Method

1. Label 7 test tubes **1 - 7**
2. Add 10.0 cm³ of 0.1 mol dm⁻³ hydrochloric acid to test tube **1**
 - Test tube **1** should not be diluted with distilled water
 - Test tube **1** will, therefore, only contain 0.1 mol dm⁻³ HCl (aq)
3. Transfer 1.0 cm³ of this hydrochloric acid, from test tube **1**, into a 10.0 cm³ measuring cylinder
4. Add 9.0 cm³ of distilled water to the measuring cylinder
 - The measuring cylinder should now contain 10.0 cm³ of liquid
5. Transfer the resulting diluted solution from the measuring cylinder into test tube **2**
 - This is a 1 : 10 dilution
 - Theoretically, the concentration of test tube **2** will be $\frac{0.1}{10} = 0.01 \text{ mol dm}^{-3}$
6. This process now repeats
7. Transfer 1.0 cm³ of this hydrochloric acid, from test tube **2**, into a 10.0 cm³ measuring cylinder
8. Add 9.0 cm³ of distilled water to the measuring cylinder
9. Transfer the resulting diluted solution from the measuring cylinder into test tube **3**
 - Theoretically, the concentration of test tube **3** will be $\frac{0.01}{10} = 0.001 \text{ mol dm}^{-3}$

10. Transfer 1.0 cm³ of this hydrochloric acid, from test tube **3**, into a 10.0 cm³ measuring cylinder
11. Add 9.0 cm³ of distilled water to the measuring cylinder
12. Transfer the resulting diluted solution from the measuring cylinder into test tube **4**
13. Continue this process until test tubes **1 - 6** are prepared
14. Test tube **7** should only contain distilled water
15. The pH of test tubes **1 - 7** can then be tested in the usual ways
 - Use of a calibrated pH probe
 - Addition of universal indicator solution and comparison to its colour chart

Specimen results

Test Tube	Concentration (mol dm ⁻³)	pH
1	0.1	1.00
2	0.01	2.00
3	0.001	3.00
4	0.0001	4.00
5	0.00001	5.00
6	0.000001	6.00
7	0.0000001	7.00

- The resulting solutions can be reacted with sodium hydroxide solution
 - This will cause a change in pH
 - It can be monitored by titrimetric methods - using a pH probe / meter to track the pH as the titration proceeds
 - This should produce results / graphs similar to those seen in [5.3.5 Neutralisation](#)

Preparing a buffer solution

- A buffer solution is a solution that is resistant to small changes in pH
- Eventually enough acid or base can be added to overcome that capacity
- The amount of acid or base needed to change the pH of a buffer is known as the "buffer capacity"

Method

- The volume and concentration of ethanoic acid used in this preparation is 5 cm³ and 0.30 mol dm⁻³ respectively
- First, calculate the concentration of ethanoic acid that will be present in 100 cm³
 - $$\text{Number of moles of ethanoic acid in } 5 \text{ cm}^3 = \frac{0.3}{1000} \times 5 = 0.0015 \text{ moles}$$
 - Therefore, this is the number of moles in 100 cm³
 - $$\text{Therefore the concentration} = \frac{0.0015}{100} \times 1000 = 0.015 \text{ mol dm}^{-3}$$
- Add 50 cm³ of distilled water to a 100 cm³ beaker
- Use a pipette to add 5 cm³ of 0.30 mol dm⁻³ of ethanoic acid to the beaker Calculate the mass of sodium ethanoate required by determining the concentration of [A⁻] using:

$$\text{pH} = \text{pK}_a + \log_{10} \frac{[\text{A}^-]}{[\text{HA}]}$$

The pK_a of ethanoic acid is 4.76

$$\circ \quad 5.00 = 4.76 + \log_{10} \frac{[\text{A}^-]}{0.015}$$

$$\circ \quad 10^{0.24} = \frac{[\text{A}^-]}{0.015}$$

$$\circ \quad [\text{A}^-] = 0.0261 \text{ mol dm}^{-3}$$

Therefore the number of moles of A^- needed in 100 cm^3 is

- $\frac{0.0261}{10} \times 100 = 0.0261$ moles
- Therefore the mass of sodium ethanoate required is
 - $0.0261 \times 82.04 = 0.214 \text{ g}$
- Measure 0.22 g of sodium ethanoate into a weighing boat
 - Slightly more sodium ethanoate than required should be measured because some will be left behind when transferring in the next steps
- Calibrate a pH meter and add it to the beaker
- Slowly add the sodium ethanoate to the beaker, stirring with a glass rod to dissolve the solid
 - Do not stir using the pH probe
 - Continue until the pH reaches 5.00
- Measure the weighing boat that contained the sodium ethanoate
- Transfer your made buffer solution to a 100 cm^3 volumetric flask
- Add distilled water up to the mark, cap and mix

Preparing a buffer solution with a higher buffer capacity

- A buffer solution with a pH of 5.0 with a higher "buffer capacity" can be prepared using 0.50 mol dm^{-3}
- The method to make this buffer solution will be the same as above but the calculations will change:
 - Number of moles of ethanoic acid in $5 \text{ cm}^3 = \frac{0.5}{1000} \times 5 = 0.0025$ moles Therefore this is the number of moles in 100 cm^3
 - Therefore the concentration $\times \frac{0.0025}{100} \times 1000 = 0.025 \text{ mol dm}^{-3}$

Calculate the mass of sodium ethanoate required by determining the concentration of $[\text{A}^-]$ using:

$$\circ \quad \text{pH} = \text{pK}_a + \log_{10} \frac{[\text{A}^-]}{[\text{HA}]}$$

◦ The pK_a of ethanoic acid is 4.76

$$\circ \quad 5.00 = 4.76 + \log_{10} \frac{[\text{A}^-]}{0.025}$$



$$0.24 = \frac{[A^-] 10}{0.025}$$

- $[A^-] = 0.0434 \text{ mol dm}^{-3}$
- Therefore the number of moles of A^- needed in 100 cm^3 is
 - $\frac{0.0434}{10} \times 100 = 0.00434 \text{ moles}$
- Therefore the mass of sodium ethanoate required is
 - $0.00434 \times 82.04 = 0.356 \text{ g}$

Testing the buffer capacity

Calibrate a pH probe

- Rinse the pH probe with distilled water and shaken to remove excess water Place the tip of the pH probe in pH 4 buffer solution ensuring bulb is fully immersed
- Allow the probe to sit until the pH stabilises
- Adjust reading to the pH of the buffer
- Repeat this process with a pH 9 buffer solution
 - Ensure that the first step, washing with distilled water, is not missed Using a
- volumetric pipette, transfer 25.0 cm^3 of the first buffer solution into a conical flask
- Load a 50 cm^3 burette with a standardised NaOH solution
- Use the pH probe to monitor the titration of the buffer until the pH changes 1 unit
- Repeat with the second buffer solution
-