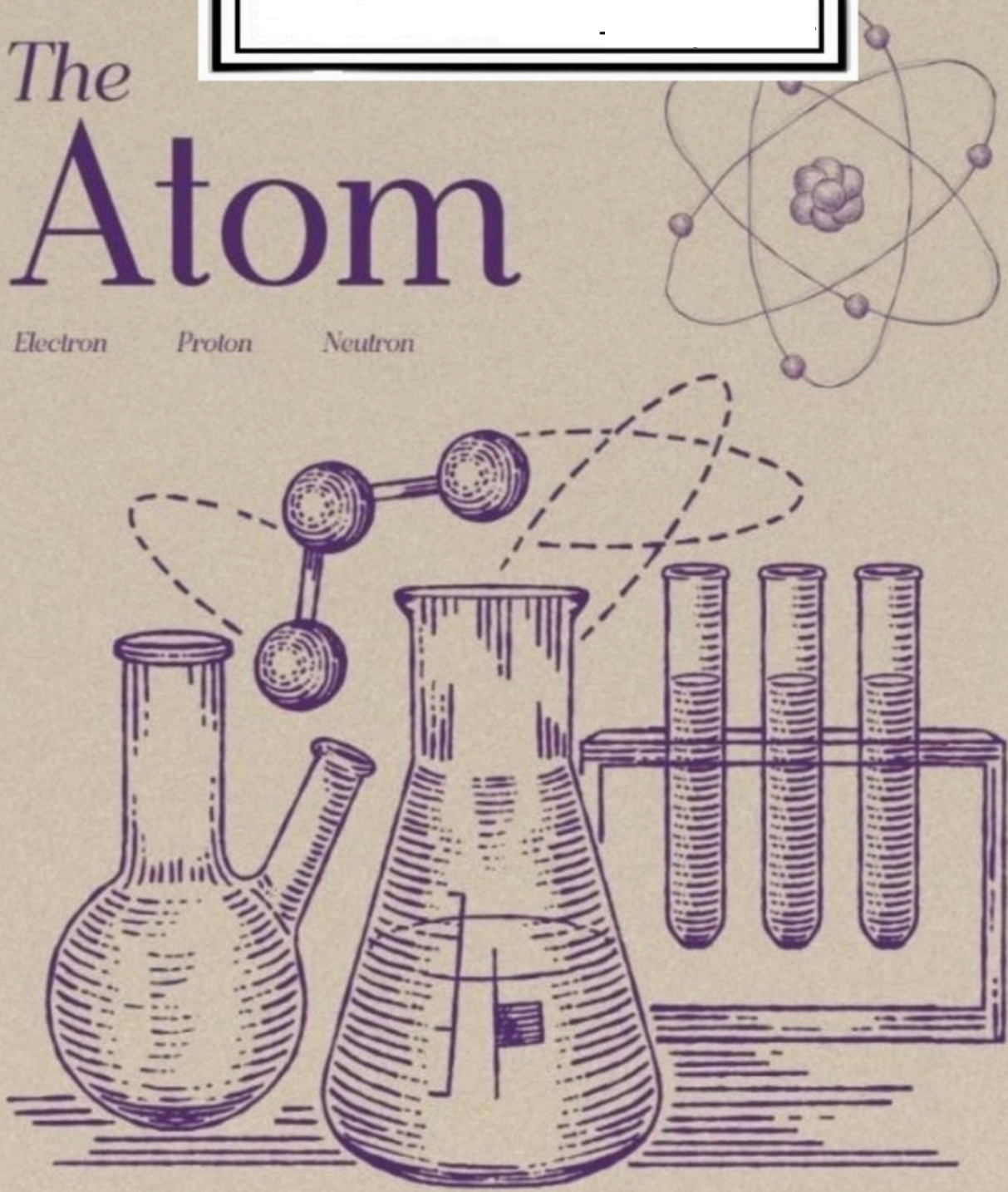


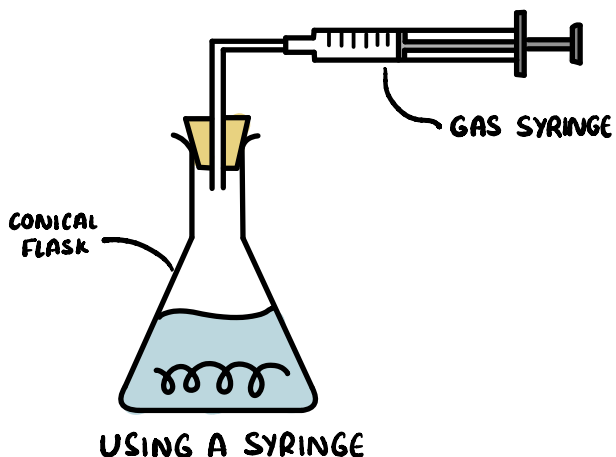
Core practical 01

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CORE PRACTICAL 01: Finding the molar volume of a gas



PROCEDURE

- Measure 30 cm³ of ethanoic acid and transfer it to a conical flask.
- Measure the mass of a test tube with approximately 0.05 g of calcium carbonate.
- Add the calcium carbonate to the conical flask quickly, sealing the bung so that no gas escapes.
- Measure and record the volume of carbon dioxide gas produced using one of two methods:
 - . Using a gas syringe
 - . Collection over water
- Reweigh the test tube
- Repeat the experiment by increasing the mass of calcium carbonate by 0.05 g successively.

How To Use The Gas Syringe

- Ensure the gas syringe reads zero before starting.
- Add calcium carbonate to the acid.
- Immediately fit the bung.
- Carbon dioxide pushes the syringe plunger outwards.
- Record the final gas volume when the reaction stops.

Why use a gas syringe?

- More accurate than collecting gas over water.
- Carbon dioxide dissolves slightly in water.
- Prevents loss of gas through dissolution.



SAFETY PRECAUTIONS

- Wear eye protection
- Remove the bung if the delivery tube gets blocked clear the blockage and repeat the procedure from the start
- Avoid skin contact with ethanoic acid especially if the skin is sensitive

Calculated molar volume TOO LOW

- CO_2 escaped before the bung was fitted.
- Leaky apparatus.
- CO_2 dissolved in water (if collected over water).
- Reaction not complete.

All of these make the measured gas volume smaller.

Calculated molar volume TOO HIGH

- Gas syringe already contained air and was not zeroed.
- Room temperature above RTP.
- Atmospheric pressure below RTP.

Exam questions

Q1. A student investigates the molar volume of CO_2 using the reaction between CaCO_3 and ethanoic acid collected in a gas syringe. Volume = 48.0 cm^3 at 25°C and 100 kPa ; mass of CaCO_3 = 0.200 g .

(a) Write the equation for the reaction.



(b) Calculate the moles of CaCO_3 ($M_r = 100.1$).

$$n(\text{CaCO}_3) = \frac{m}{M_r} = \frac{0.2}{100.1} = 2 \times 10^{-3} \text{ mol}$$

(c) Calculate the molar volume of CO_2 and compare with $24.0 \text{ dm}^3 \text{ mol}^{-1}$ at RTP. Suggest one reason for any difference.

$$n_{\text{gas}} = \frac{V_{\text{gas}}}{\text{molar volume}}$$

$$\text{molar volume} = \frac{48}{2 \times 10^{-3}} = \frac{24\,000 \text{ cm}^3 \text{ mol}^{-1}}{1000} = 24 \text{ dm}^3 \text{ mol}^{-1}$$

A small difference could be there due to CO_2 being slightly soluble in water OR some of the gas escaped before bung was inserted

Q2. 0.150 g of a volatile liquid was injected into a sealed gas syringe placed in an oven at 70°C and 100 kPa . A volume of 80 cm^3 was measured.

(a) Show that the moles of gas present $\approx 2.8 \times 10^{-3}$. ($R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$)

$$PV = nRT$$

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

$$n = \frac{100\,000 \times 8 \times 10^{-5}}{8.31 \times 343}$$

$$n = 2.8066... \times 10^{-3}$$

$$n = \underline{\underline{2.81 \times 10^{-3}}}$$

$$\begin{array}{ccc} 80 \text{ cm}^3 & \xrightarrow{\times 10^{-6}} & 8 \times 10^{-5} \text{ m}^3 \\ 70 & \xrightarrow{+273} & 343 \text{ K} \\ 100 \text{ kPa} & \xrightarrow{\times 10^3} & 100\,000 \text{ Pa} \end{array}$$

(b) Calculate the M_r of the volatile liquid.

$$M_r = \frac{m}{n} = \frac{0.15}{2.8 \times 10^{-3}} = \underline{\underline{53.4 \text{ g mol}^{-1}}}$$

Q3. A student uses the over-water method to collect CO_2 .

(c) State two sources of error specific to this method and the effect of each on the calculated molar volume.

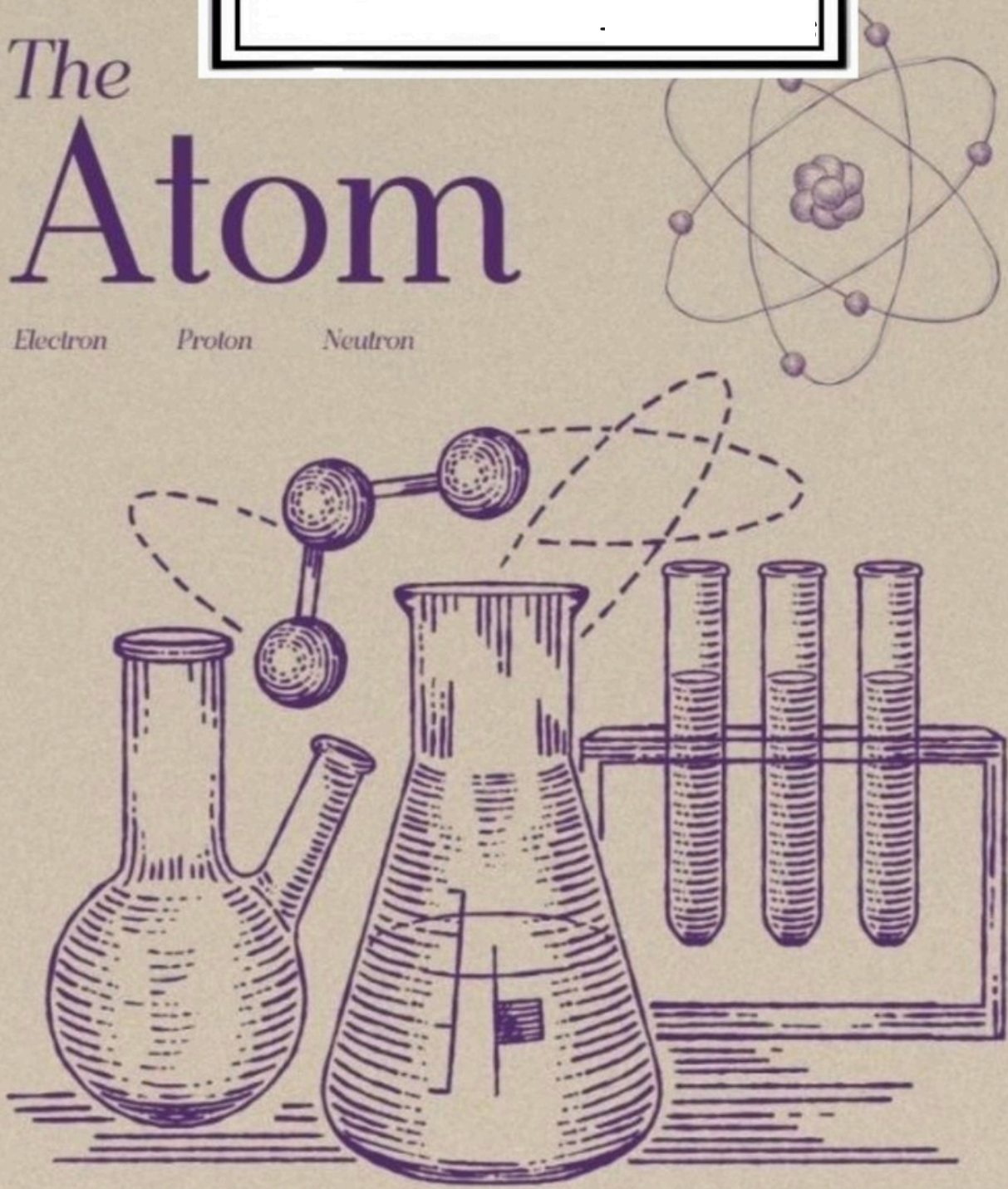
CO_2 dissolves slightly in water causing the volume of the gas to decrease hence molar volume becomes too low

Water level would have been difficult to read accurately due to uneven surface and water vapour is also present in the cylinder which is not recorded causing the volume to be too low hence molar a volume becomes too low

Core practical 02

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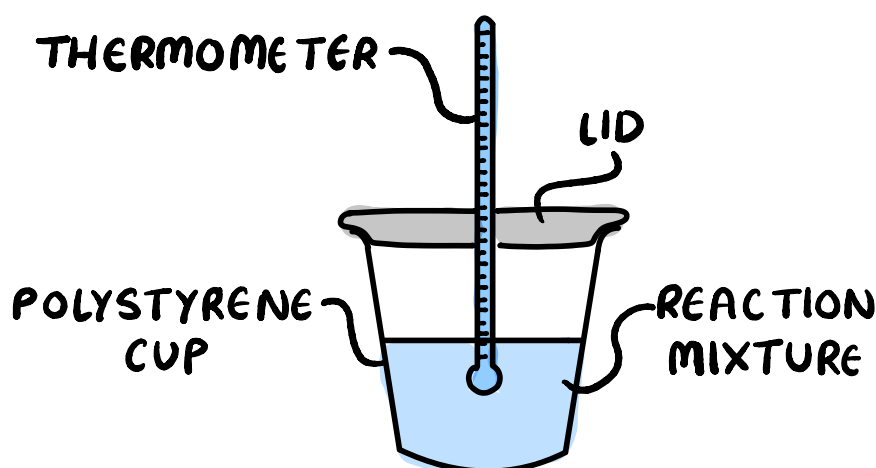
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CORE PRACTICAL 02: Determining Enthalpy change of a reaction

Procedure

- Place a polystyrene cup in a beaker for both insulation and support.
- Using a pipette, transfer desired volume of hydrochloric acid (or other solution) to the cup.
- Place a thermometer in the cup and measure the initial temperature of the solution.
- Potassium carbonate (or other solid reagent) should be added to the cup after the test tube with the solid is weighed using a balance.
- Stir the mixture and record the temperature every minute for several minutes.
- The empty test tube should be reweighed.
- The experiment should then be repeated using potassium hydrogencarbonate and the lowest temperature reached should be recorded.



SAFETY PRECAUTIONS

Wear eye protection
Avoid skin contact
with the reactants
and products

Q and A

Q. Why use a polystyrene cup?

⇒ It acts as an insulator and reduces heat transfer to the surroundings.

Q. Why stir continuously?

⇒ To distribute heat evenly and obtain a uniform temperature.

Q. Why use excess hydrochloric acid?

⇒ To ensure the carbonate is the limiting reagent and reacts completely.

Q. Why is ΔH negative for $K_2CO_3 + HCl$?

⇒ The temperature increases, meaning heat is released to the surroundings.

Q. Why is ΔH positive for $KHCO_3 + HCl$?

⇒ The temperature decreases, meaning heat is absorbed from the surroundings.

common errors

error	Effect	How to minimise
Heat loss to surroundings	Q too small $\rightarrow \Delta H$ less negative	Polystyrene cup with lid extrapolate T -time graph
Heat capacity of cup ignored	Q underestimated	Use polystyrene (low heat capacity) or determine calorimeter constant
Incomplete dissolution of solid	ΔT too small $\rightarrow \Delta H $ too small	ensure all solid dissolves before recording
Evaporation of fuel (flame calorimetry)	Mass loss overestimated $\rightarrow \Delta H $ too small	Replace spirit lamp cap immediately after extinguishing
Density assumed = 1 g cm^{-3} and specific heat capacity also assumed	Small systematic error in mass	Weigh solutions directly for greater accuracy

Exam questions

Q1. A student reacts 25.0 cm^3 of $2.0 \text{ mol dm}^{-3} \text{ CuSO}_4(\text{aq})$ with excess zinc powder. Temperature rises from 19.5°C to 26.5°C . ($c = 4.18 \text{ J g}^{-1} \text{ K}^{-1}$; density = 1.00 g cm^{-3})

(a) Calculate q .

$$\begin{aligned}
 Q &= mc\Delta T \\
 &= 25 \times 4.18 \times (26.5 - 19.5) \\
 &= 25 \times 4.18 \times 7 \\
 &= \underline{\underline{731.5 \text{ J}}}
 \end{aligned}$$

(b) Calculate the moles of CuSO_4 .

$$\begin{aligned}
 n(\text{CuSO}_4) &= c \times v \\
 &= 2.0 \times \frac{25.0}{1000} \\
 &= \underline{\underline{0.05 \text{ mol}}}
 \end{aligned}$$

(c) Calculate ΔH per mole and give the sign, explaining what it represents.

$$\begin{aligned}
 \Delta H &= \pm \frac{Q}{n} \\
 &= \frac{731.5}{0.05} \\
 &= 14.6 \text{ kJ mol}^{-1} \\
 \text{Temperature increase} &\therefore \text{exothermic} \rightarrow -\Delta H \\
 &= \underline{\underline{-14.6 \text{ kJ mol}^{-1}}}
 \end{aligned}$$

Q2. 3.90 g anhydrous CuSO_4 ($M_r = 159.5$) dissolved in 25 cm^3 water; T rises 9.2°C . 6.24 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ($M_r = 249.5$) dissolved in 25 cm^3 water; T falls 1.8°C .

(a) Calculate ΔH_{soln} for anhydrous CuSO_4 .

$$Q = 25 \times 4.18 \times 9.2$$

$$= 961 \text{ J}$$

$$n = \frac{m}{M_r} = \frac{3.9}{159.5} = 0.02446 \text{ mol}$$

$$\Delta H_1 = \frac{Q}{n} = \frac{961 \div 1000}{0.02446} = \underline{\underline{-39.3 \text{ kJ mol}^{-1}}}$$

(b) Calculate ΔH_{soln} for hydrated CuSO_4 .

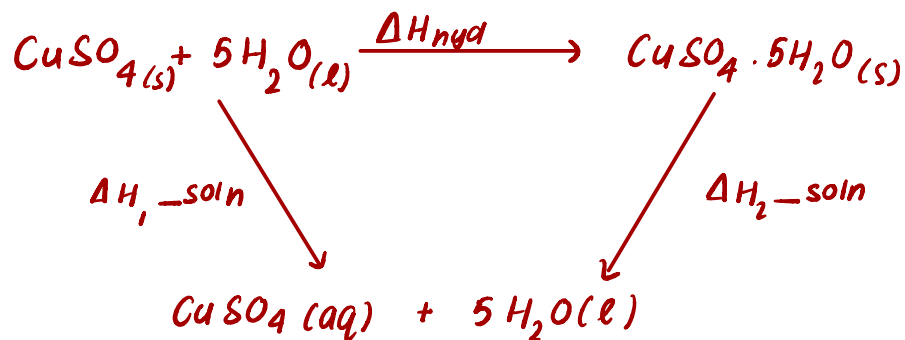
$$Q = 25 \times 4.18 \times 1.8$$

$$= 188 \text{ J}$$

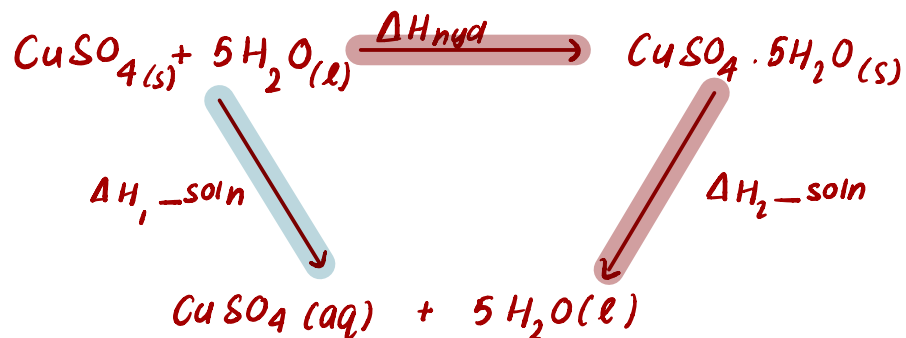
$$n = \frac{m}{M_r} = \frac{6.24}{249.5} = 0.02501 \text{ mol}$$

$$\Delta H_2 = \frac{188 \div 1000}{0.02501} = \underline{\underline{+7.5 \text{ kJ mol}^{-1}}}$$

(c) Draw and label a Hess's Law cycle.



d) Calculate the enthalpy of hydration.



$$\Delta H_{\text{hyd}} + \Delta H_2\text{-soln} = \Delta H_1\text{-soln}$$

$$\Delta H_{\text{hyd}} = \Delta H_1\text{-soln} - \Delta H_2\text{-soln}$$

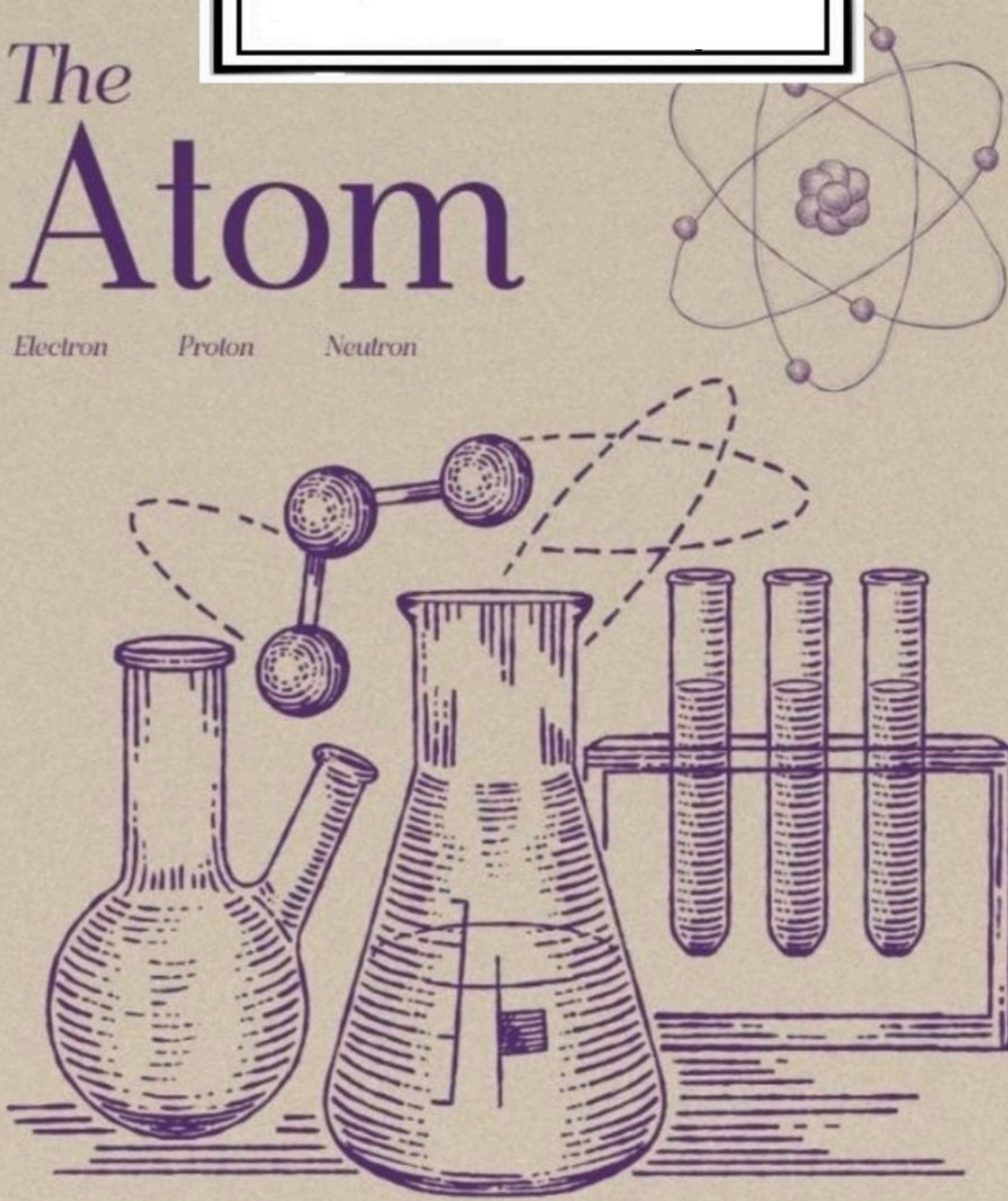
$$\Delta H_{\text{hyd}} = -39.3 - 7.5$$

$$\Delta H_{\text{hyd}} = \underline{\underline{-46.8 \text{ kJ mol}^{-1}}}$$

Core practical 03 & 04

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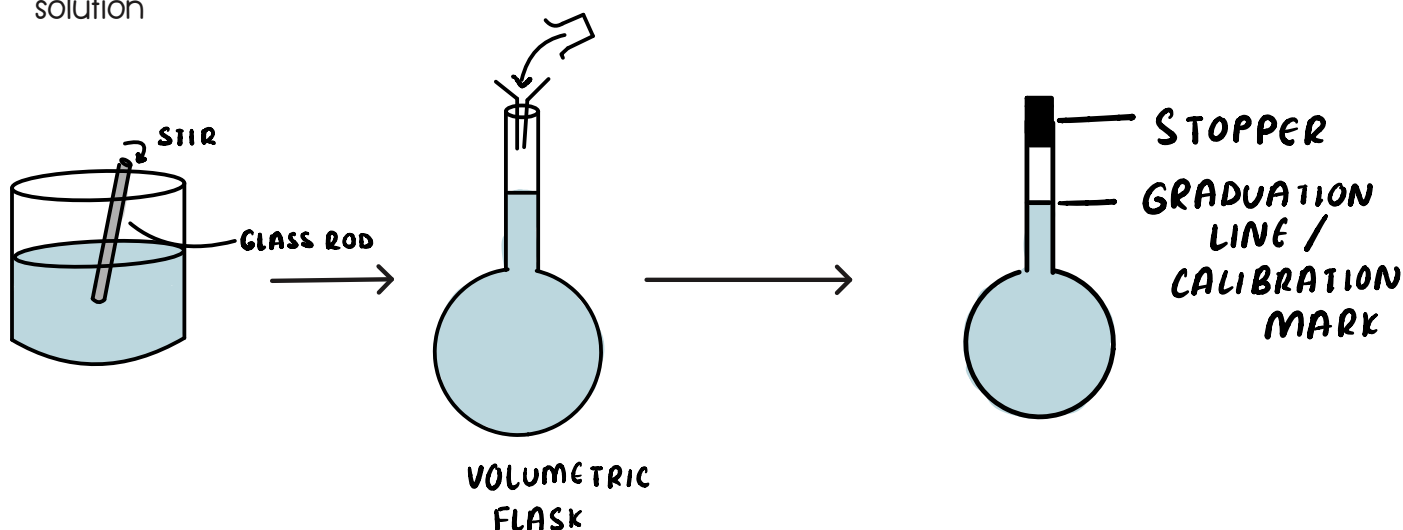
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CORE PRACTICAL 03 & 04: Titrations and standard solutions

STANDARD SOLUTION PROCEDURE

- Dissolve the solid in a beaker with distilled water. The amount of water added depends on the desired concentration of the standard solution.
- Stir the mixture using a glass rod until all the solid is dissolved in the water.
- Using a funnel, transfer the solution to a volumetric flask.
- After transferring, wash the beaker with distilled water and transfer these washings to the volumetric flask.
- Using a pipette, continue adding distilled water to the volumetric flask until the bottom of the meniscus is up to the mark.
- Close the volumetric flask using a stopper and keep inverting and shaking it to mix the solution



Precautions and Reasons for Taking Them:

- Rinse pipette with NaOH solution: Prevents dilution or contamination, ensuring accurate aliquot volumes.
- Rinse burette with sulfamic acid solution: Ensures correct concentration of titrant.
- Use minimal methyl orange: Excess indicator reacts with acid, skewing endpoint detection.
- Wash conical flask with distilled water between titrations: Removes residues, preventing cross-contamination.
- Invert volumetric flask after filling: Ensures homogeneous solution, avoiding concentration gradients.
- Read burette at eye level: Minimizes parallax errors for precise volume measurements.
- Use accurate mass balance: Ensures correct sulfamic acid mass, critical for standard solution concentration.



Safety precautions

Wear eye protection. Goggles are preferred as sodium hydroxide is particularly hazardous to the eyes take care when clamping and filling the burette that it does not crack or topple over.

TITRATION PROCEDURE

- Rinse the burette and pipette with HCl and NaOH, respectively (the apparatus should be rinsed with the solution they will contain. Water can leave residue behind).
- Pipette 25cm³ of NaOH into the conical flask. Place the flask over a white tile to make the colour change clearer.
- Fill the burette with the HCl (make sure no air bubbles form)
- Start titrating by adding the HCl slowly to the conical flask and swirling the flask
- Add an indicator (to the conical flask) to determine the endpoint of the titration.

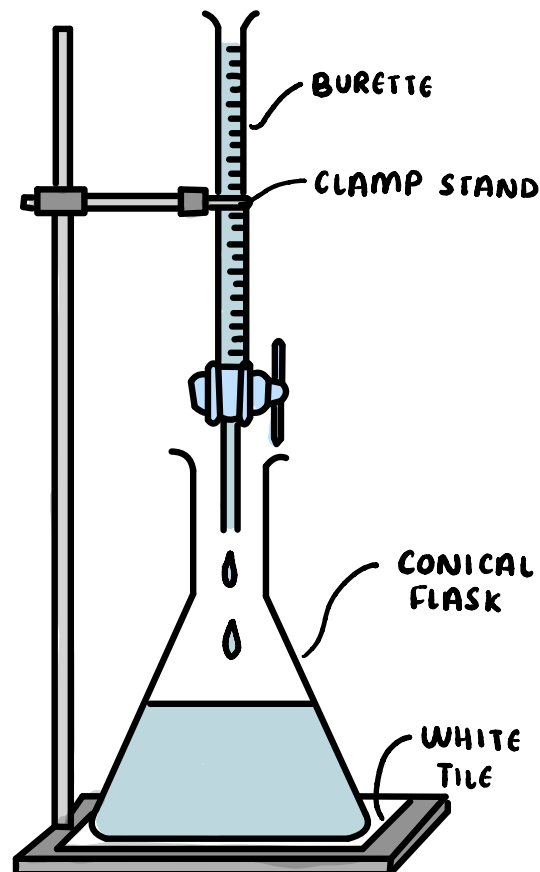
Phenolphthalein will turn pink (alkali) to colourless (neutral)

- the colour disappears at the endpoint.

Methyl orange will turn yellow (alkali) to orange (neutral) -

the colour is orange at the endpoint.

- Record the burette readings before and after adding the acid
- Repeat the titration until a minimum of two concordant results are obtained.



Precautions and Reasons for Taking Them:

- . Rinse pipette with HCl solution: Removes water or contaminants, preventing dilution or side reactions.
- . Rinse burette with NaOH solution: Ensures accurate concentration of titrant.
- . Read burette at eye level: Avoids parallax errors, ensuring precise volume measurements.
- . Swirl flask during titration: Ensures complete mixing, preventing premature endpoint detection.
- . Wash conical flask with distilled water between titrations: Removes residual chemicals, preventing contamination.
- . Ensure concordant titres: Increases reliability of mean titre, reducing random errors.



Safety precautions

Wear eye protection. Goggles are preferred as sodium hydroxide is particularly hazardous to the eyes take care when clamping and filling the burette that it does not crack or topple over.

Common errors

error	Effect	How to minimise
Burette not rinsed with acid	Acid diluted → titre too large	Always rinse burette with solution to be used
Funnel left in burette during titration	Drops fall in → titre reading too low	Remove funnel before starting
Air bubble in jet space	Bubble disappears mid-run → titre too large	Fill jet; tap burette to remove bubbles
Parallax error reading burette	Incorrect titre volume	Read at eye level; read bottom of meniscus
Pipette not rinsed with alkali	Alkali diluted → fewer moles → titre too small	Always rinse pipette with solution being measured
Measuring cylinder used instead of pipette	Large uncertainty → inaccurate volume	Use volumetric pipette for 25.00 cm ³

Exam questions

Q1. A student dissolves 1.33 g of Na₂CO₃ (M_r = 106) in water and makes up to 250 cm³. She titrates 25.00 cm³ of this against HCl(aq) using methyl orange. Titres: 24.80, 24.35, 24.40, 24.35 cm³.

(a) Calculate the concentration of Na₂CO₃.

$$n(\text{Na}_2\text{CO}_3) = \frac{m}{M_r} = \frac{1.33}{106} = 1.255 \times 10^{-3} \text{ mol}$$

$$c = \frac{n}{v} = \frac{1.225 \times 10^{-3}}{0.250} = 0.0502 \text{ mol dm}^{-3}$$

(b) Identify concordant results and calculate mean titre.

concordant results (within 0.10 cm³) = 24.35, 24.40, 24.35
24.80 ⇒ rejected

$$\therefore \text{mean} = \frac{24.35 + 24.40 + 24.35}{3} = 24.37 \text{ cm}^3$$

(c) Write the equation for the reaction.



(d) Calculate the concentration of HCl.

$$n(\text{Na}_2\text{CO}_3 \text{ per litre}) = c \times v = 0.0502 \times \frac{24.37}{100} = 1.224 \times 10^{-3} \text{ mol}$$

$$\begin{array}{ccc} \text{Na}_2\text{CO}_3 & : & \text{HCl} \\ 1 & : & 2 \\ \therefore n(\text{HCl}) & = & 1.224 \times 10^{-3} \times 2 \\ & = & 2.447 \times 10^{-3} \end{array}$$

$$c = \frac{n}{v} = \frac{2.447 \times 10^{-3}}{0.025} = 0.0979 \text{ mol dm}^{-3}$$

Q2. A burette has uncertainty $\pm 0.05 \text{ cm}^3$ per reading. Mean titre = 24.40 cm^3 .

(b) Calculate the percentage uncertainty in the titre.

$$\begin{aligned} \% \text{ uncertainty} &= \frac{0.05 \times 2}{24.40} \times 100 \\ &= 0.41\% \end{aligned}$$

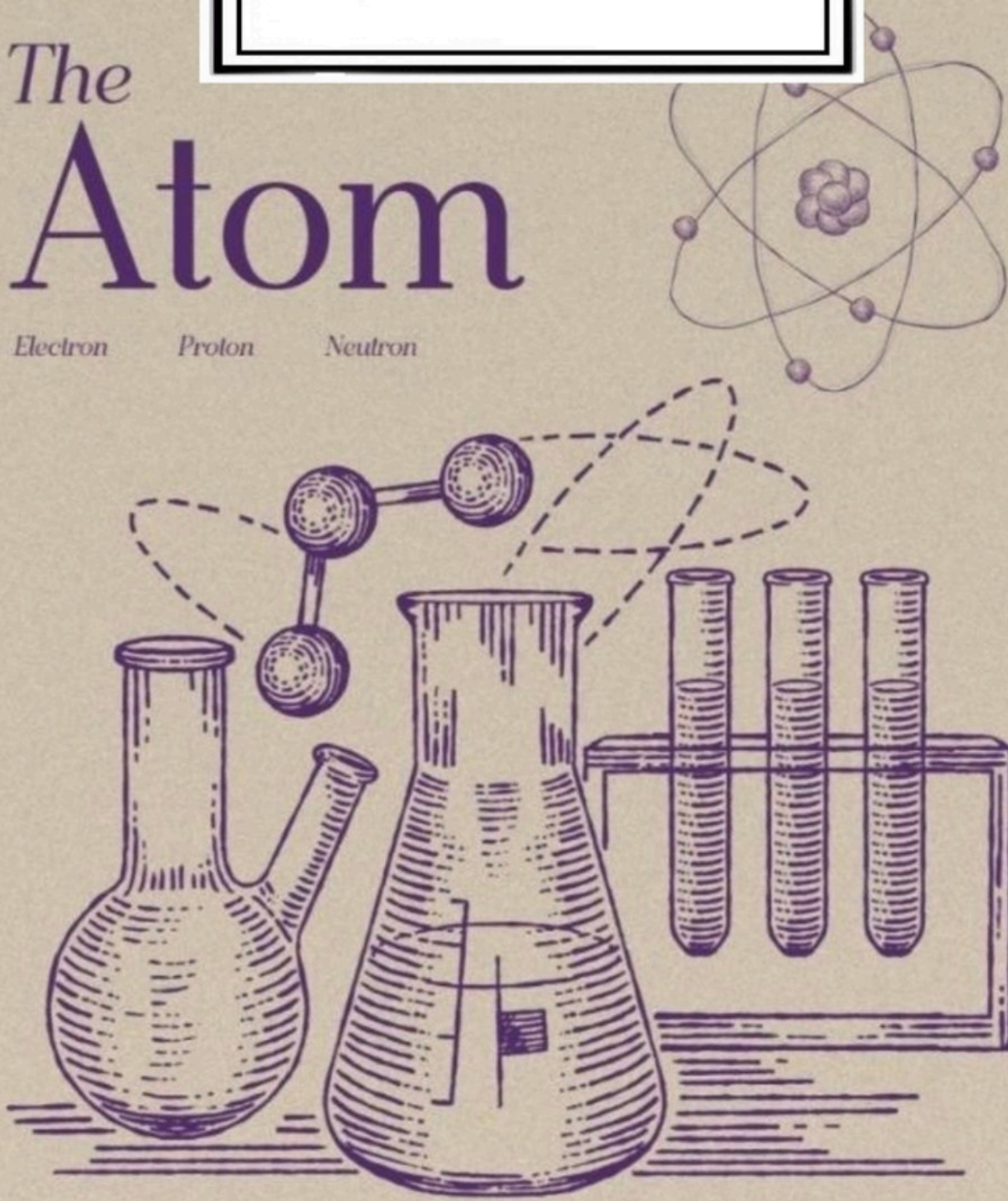
(c) An air bubble was in the jet space at the start and disappeared during the titration. Explain the effect on the titre and on the calculated concentration of the acid

- Bubble fills with acid during titration causing the volume of acid recorded is greater than volume delivered to flask.
- this results in titre being too large
- Hence, calculated concentration of acid appears higher than true value

Core practical 05

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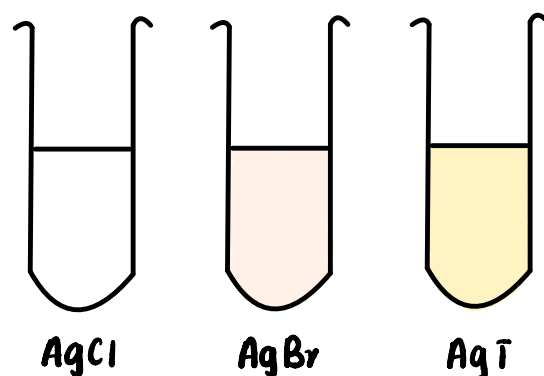
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CORE PRACTICAL 05: rate of hydrolysis of halogenoalkanes

procedure

- Set up three test tubes. Add 1 cm³ of ethanol to each — ethanol dissolves the halogenoalkane (they are insoluble in water).
- Add 2 drops of a different halogenoalkane to each tube: 1-chlorobutane, 1-bromobutane, 1-iodobutane.
- Place all three test tubes in a water bath (60°C) together with a test tube of 0.1 mol dm⁻³ AgNO₃ dissolved in ethanol.
- Allow all tubes to reach the same temperature (~5 minutes).
- Quickly add 1 cm³ of AgNO₃ in ethanol to each test tube simultaneously; start stopwatch.
- Record the time taken for a precipitate to appear in each tube and note the colour.



WHY ETHANOL? Halogenoalkanes are INSOLUBLE in water — ethanol dissolves them so the AgNO₃ can react. AgNO₃ must also be dissolved in ETHANOL (not water) for the same reason. Using water instead of ethanol is a common error — no observable reaction would occur

Observations

Halogenoalkane	Precipitate	Colour	Speed	Reason
1-iodobutane	AgI	Pale yellow	Fastest	C-I bond weakest → lowest E _a → fastest hydrolysis
1-bromobutane	AgBr	Cream	Second	C-Br bond intermediate strength
1-chlorobutane	AgCl	White	Slowest	C-Cl bond strongest → highest E _a → slowest hydrolysis

Common errors

Error	Effect	How to minimise
Using AgNO ₃ in water instead of ethanol	No reaction — halogenoalkanes insoluble in water	Always use AgNO ₃ dissolved in ethanol
Test tubes NOT placed in bath simultaneously	Temperature comparison invalid — unfair test	Place all three in bath at the same moment
Temperature of water bath varies	Time comparison meaningless	Use thermostatted bath; monitor temperature
AgNO ₃ added at different times	Incorrect titre volume	Add to all three tubes simultaneously; start stopwatch at moment of addition



Precautions and Reasons for Taking Them:

- Wear eye protection: Halogenoalkanes and ethanol are irritants and can harm eyes.
- Avoid skin contact: Halogenoalkanes are toxic and can cause burns or irritation.
- No naked flames: Ethanol and halogenoalkanes are highly flammable, posing a fire risk.
- Use a fume cupboard: Prevents inhalation of toxic halogenoalkane vapours.
- Maintain constant water bath temperature (50°C): Ensures consistent reaction rates for fair comparison.
- Label test tubes clearly: Prevents mix-ups, ensuring accurate identification of precipitates.
- Start stopwatch simultaneously with AgNO₃ addition: Ensures precise timing of precipitation.

Exam questions

Q1. A student investigates the rates of hydrolysis of three halogenoalkanes.

(a) State why ethanol is used rather than water.

Halogenoalkanes are insoluble in water; ethanol dissolves the halogenoalkane AND the AgNO₃ so they can come into contact and react

(b) Explain why all three test tubes must be placed in the water bath simultaneously.

To control temperature as a variable — all three reactions must start at the same temperature to make the rate comparison fair

(c) The student observes a pale yellow precipitate first, then cream, then white. Identify the halogenoalkane for each and explain the order in terms of bond enthalpies.

- Pale yellow = AgI from 1-iodobutane
- cream = AgBr from 1-bromobutane
- white = AgCl from 1-chlorobutane
- C—I has the lowest bond enthalpy as it has the weakest bond. Hence, it has the lowest activation energy resulting in fastest hydrolysis
- While C—Cl strongest bond therefore slowest rate of hydrolysis

Q2. b) Write an ionic equation for the reaction producing the pale yellow precipitate.



(c) Describe what you would observe when dilute ammonia is added to:

(i) the white precipitate and (ii) the pale yellow precipitate

- (i) White precipitate (AgCl) dissolves in dilute ammonia to give a colourless solution
- (ii) Pale yellow precipitate (AgI) does not dissolve in dilute or concentrated ammonia — remains as pale yellow solid

Q3. (a) A student uses aqueous AgNO_3 instead of AgNO_3 in ethanol. Explain the effect on the results

- Halogenoalkanes are insoluble in water.
- This results in no / very slow reaction with aqueous AgNO_3
- Hence, no precipitate observed
- Thus, cannot compare rates

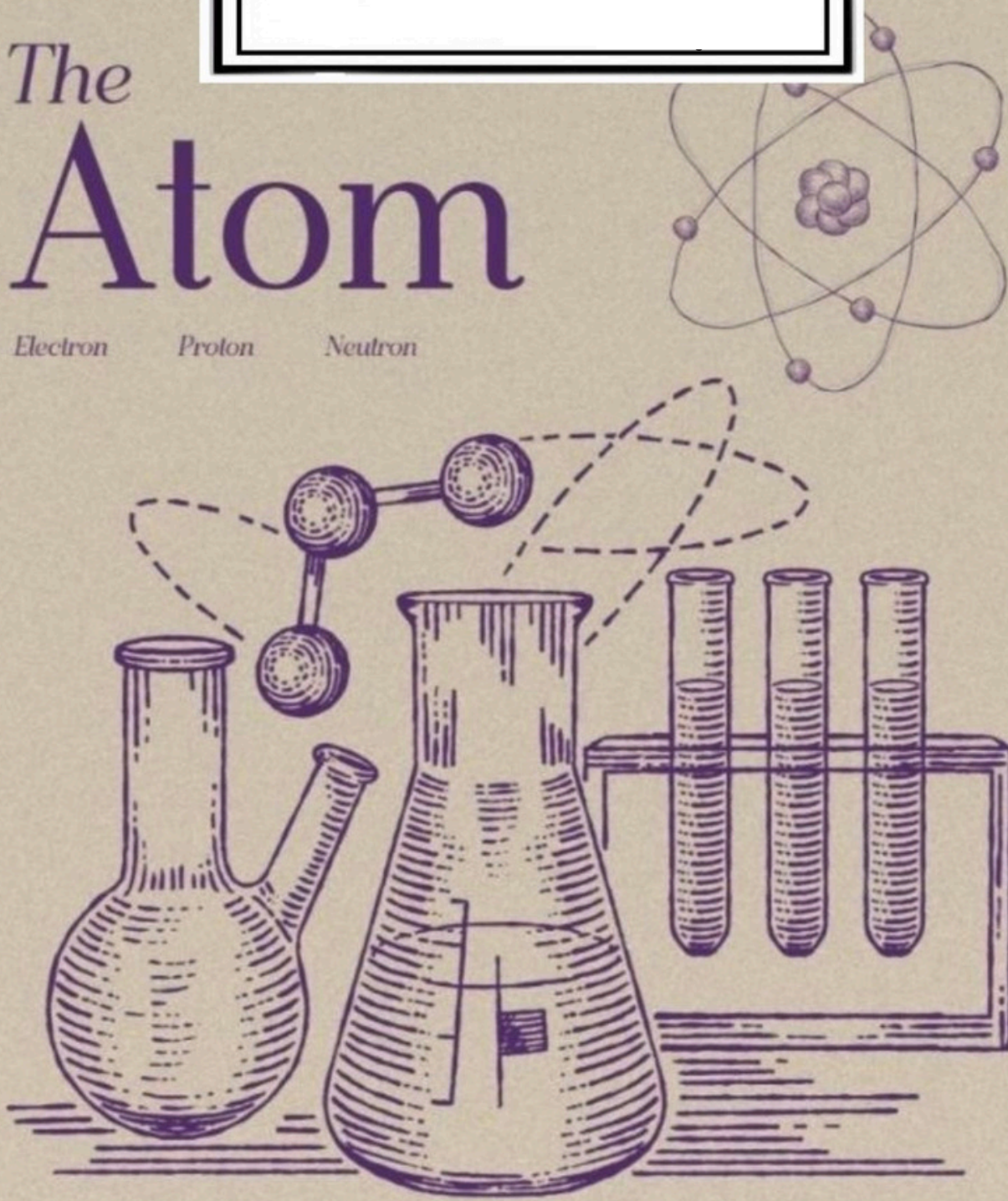
(b) State two variables that must be controlled to make the rate comparison a fair test.

- temperature (same water bath temperature)
- volume/concentration of AgNO_3 in ethanol
- volume/number of drops of each halogenoalkane
- all three added to AgNO_3 simultaneously

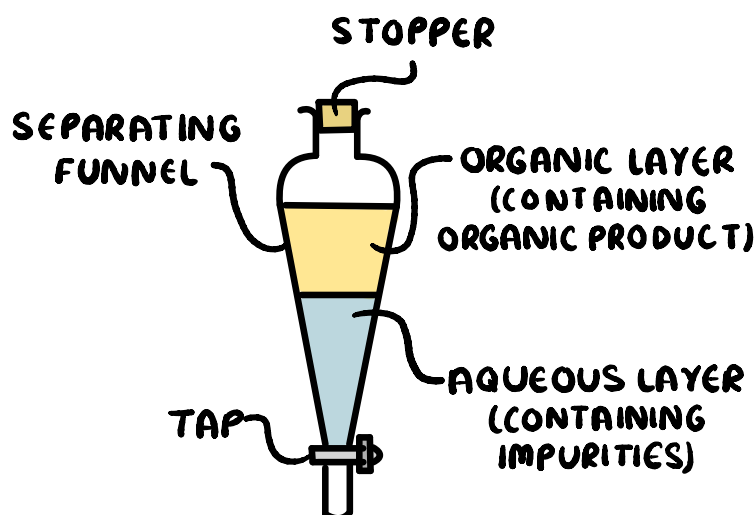
Core practical 06

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CORE PRACTICAL 06: Chlorination of 2-methylpropan-2-ol



procedure

- Measure 8 cm³ of 2-methylpropan-2-ol; weigh and record mass. Pour into separating funnel; reweigh to find exact mass used.
- In a fume cupboard, add 20 cm³ of concentrated HCl in 3 cm³ portions. After each addition, stopper and invert funnel; open tap to release pressure.
- Allow funnel to stand ~20 minutes; shake gently at intervals
- Allow layers to separate; remove and discard the lower aqueous layer.
- Add NaHCO₃ solution in 2 cm³ portions to neutralise residual acid. Release CO₂ pressure after each addition. Continue until no more CO₂ bubbles.
- Remove and discard the lower aqueous layer.
- Run organic layer into clean conical flask; add 2 spatulas of anhydrous Na₂SO₄. Stopper, shake, stand until liquid is clear (= dry).
- Decant into a clean distillation flask; distil; collect fraction boiling at 47–53°C.
- Weigh the product and calculate % yield.

Precautions and Reasons for Taking Them:

- Use a fume cupboard: Concentrated HCl and halogenoalkane vapours are toxic and corrosive.
- Wear goggles and gloves: Protects eyes and skin from corrosive HCl and irritant product.
- Avoid inhaling vapours: Prevents respiratory damage from toxic fumes.
- Release pressure frequently: Prevents flask/separating funnel rupture due to CO₂ or HCl gas buildup.
- No naked flames: Product is flammable, posing a fire risk.
- Use anhydrous drying agents: Removes water to prevent contamination of the organic product.
- Handle NaHCO₃ carefully: Neutralizes residual HCl safely, but gas evolution requires pressure release.

Purpose of certain step

1. Separate in separating funnel → Remove aqueous HCl layer from organic product
2. Wash with NaHCO_3 solution → Neutralise residual HCl; produces CO_2 — release pressure after each addition
3. Dry with anhydrous Na_2SO_4 → Remove dissolved water from organic layer — clear when dry
4. Distil; collect 47–53°C fraction → Remove remaining impurities; collect pure product

Common errors

Error	Effect	How to minimise
Not releasing pressure from funnel	Pressure builds → stopper blown out	Invert and open tap regularly
Discarding the organic layer	Product is lost	Check: organic layer is upper (lower density); test a drop in water
Incomplete drying	Water in product → impure; broad bp range	Add more drying agent; wait until liquid is clear
Collecting wrong boiling fraction	Impure product; incorrect compound	Thermometer at T-junction; collect only 47–53°C

Exam questions

Q1. (a) State why NaHCO_3 solution is used rather than NaOH solution to wash the organic product.

- NaOH is a strong alkali that could react with 2-chloro-2-methylpropane (elimination or substitution) and destroy the product.
- NaHCO_3 is a mild base that only neutralises HCl without attacking the product

(b) Write the ionic equation for the reaction of HCl with NaHCO_3 . Explain why the separating funnel must be inverted and the tap opened regularly.

- $\text{H}^+ + \text{HCO}_3^- \rightarrow \text{H}_2\text{O} + \text{CO}_2$ (or $\text{HCl} + \text{NaHCO}_3 \rightarrow \text{NaCl} + \text{H}_2\text{O} + \text{CO}_2$)
- CO_2 gas produced increases pressure inside funnel
- opening tap releases pressure to prevent stopper being blown out

(c) 5.0 g of 2-methylpropan-2-ol ($M_r = 74$) was used; 4.2 g of 2-chloro-2-methylpropane ($M_r = 92.5$) was collected. Calculate the percentage yield.

$$\text{theoretical moles} = \frac{m}{M_r} = \frac{5}{74} = 0.0676 \text{ mol}$$

$$\begin{aligned}\text{theoretical mass} &= n \times M_r \\ &= 0.0676 \times 92.5 \\ &= 6.25 \text{ g}\end{aligned}$$

$$\% \text{ yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100$$

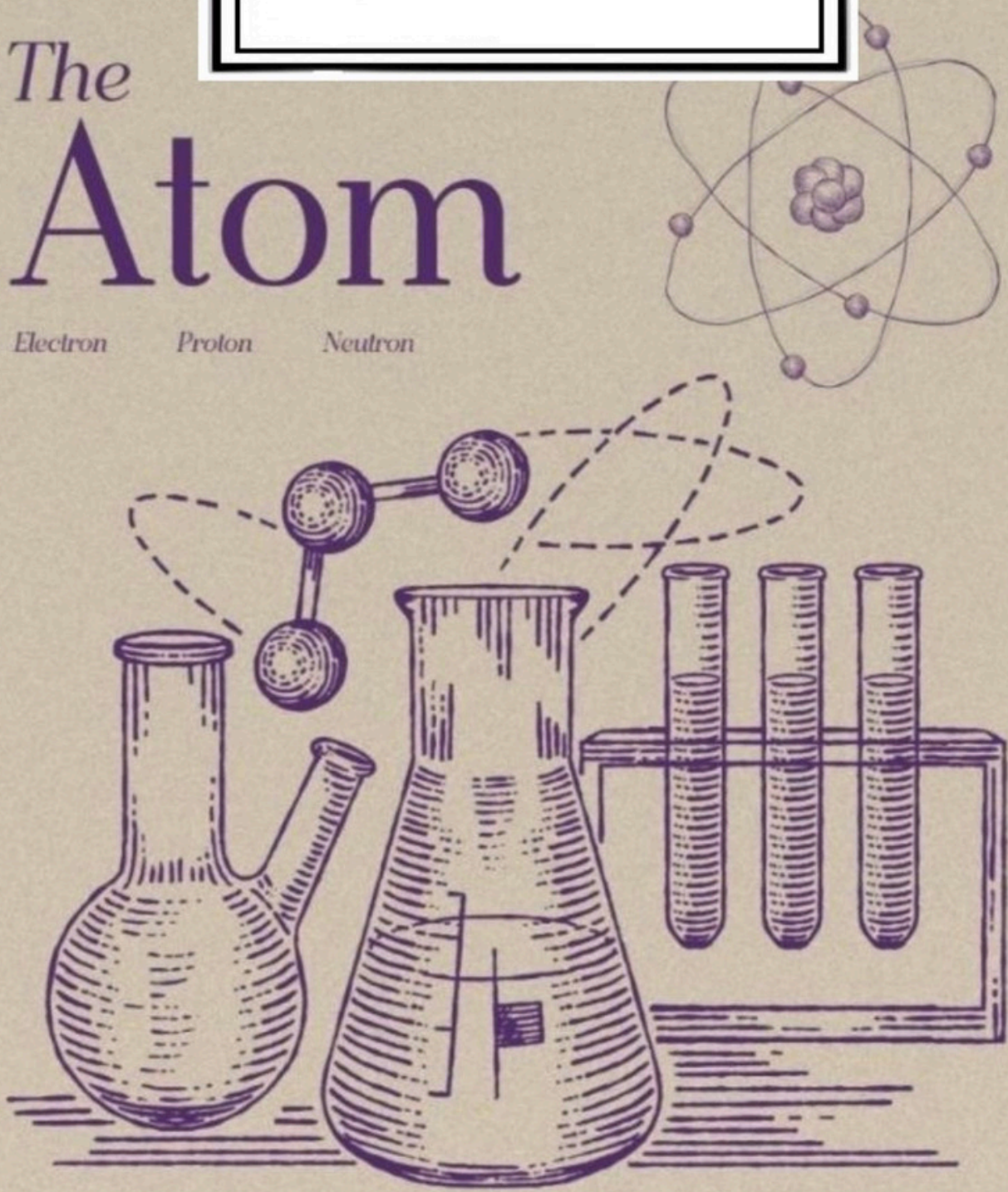
$$= \frac{4.2}{6.25} \times 100$$

$$= \underline{\underline{67.2\%}}$$

Core practical 01

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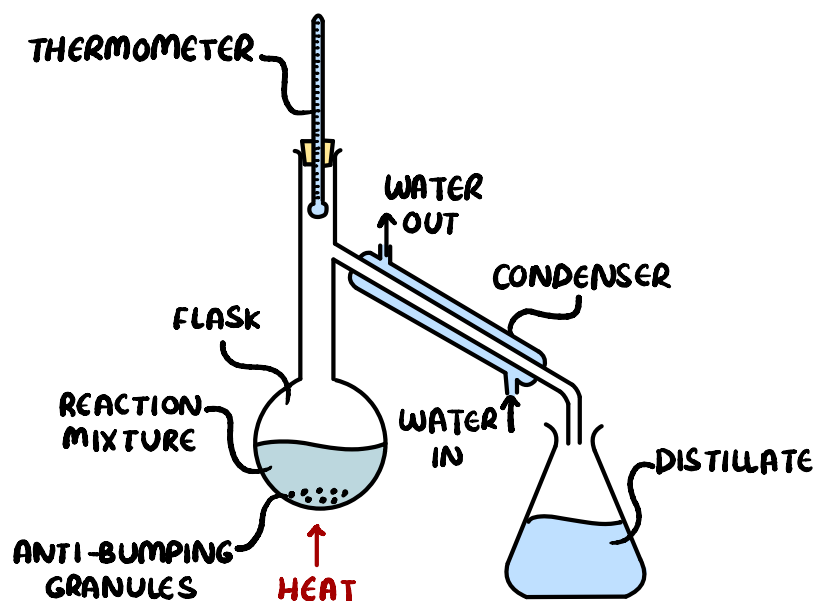
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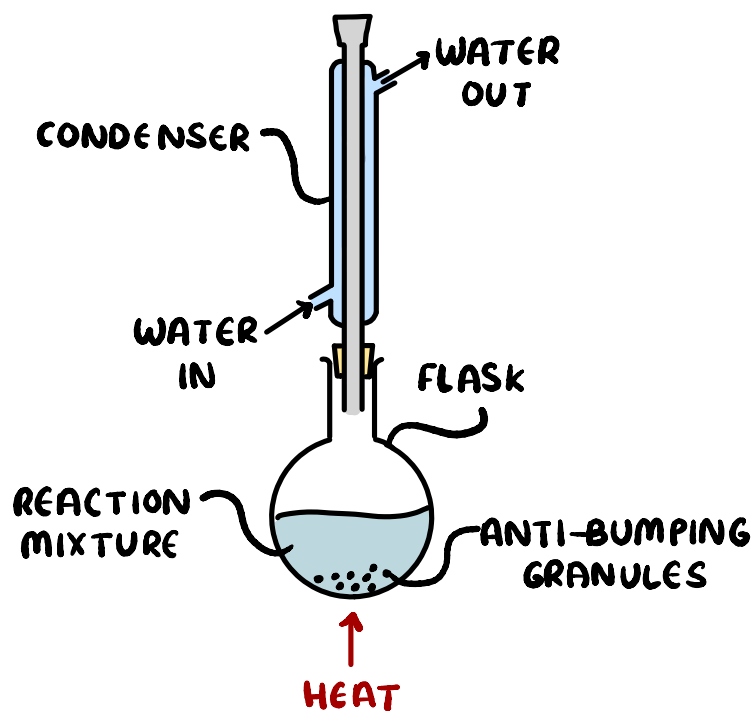
CORE PRACTICAL 07: oxidation of propan-1-ol

distillation

- Place $\sim 10 \text{ cm}^3$ dilute H_2SO_4 in a flask; add $\sim 3 \text{ g}$ $\text{K}_2\text{Cr}_2\text{O}_7$ and anti-bumping granules; shake to dissolve (do NOT warm).
- Add 1.5 cm^3 propan-1-ol drop-wise from a dropping pipette, shaking to mix.
- Assemble distillation apparatus: round-bottomed flask $\rightarrow$ thermometer bulb at T-junction $\rightarrow$ Liebig condenser (water in at bottom, out at top) $\rightarrow$ collection vessel.
- Heat gently; slowly distil $\sim 2 \text{ cm}^3$ into a test tube — this is crude propanal.
- Observation: orange $\text{Cr}_2\text{O}_7^{2-} \rightarrow$ green Cr^{3+} .



Heat under reflux



- Dissolve 6 g $\text{Na}_2\text{Cr}_2\text{O}_7$ in 5 cm^3 water; set aside.
- Put 1.5 cm^3 propan-1-ol in a 50 cm^3 round-bottomed flask; add $\sim 5 \text{ cm}^3$ water and anti-bumping granules.
- Fit vertical reflux condenser; add 2 cm^3 concentrated H_2SO_4 drop-by-drop down condenser while warm.
- Add dichromate solution down condenser drop-by-drop; energy released keeps mixture boiling.
- When all dichromate added, use low Bunsen flame for 10 minutes; do NOT allow vapour to escape.
- Rearrange apparatus for distillation; collect $2\text{--}3 \text{ cm}^3$ of product.

KEY POINTS

- Observation: orange ($\text{Cr}_2\text{O}_7^{2-}$) $\rightarrow$ green (Cr^{3+}) confirms oxidation has occurred.
- NEVER seal the end of the condenser — pressure build-up could cause the apparatus to explode.
- Use electric heater (not naked flame) — organic compounds are highly flammable.
- Anti-bumping granules make small bubbles $\rightarrow$ prevent vigorous uneven boiling.
- Water enters condenser at the BOTTOM — counterflow gives more efficient cooling.
- Thermometer bulb at T-junction — measures the boiling vapour temperature, not the liquid.
- Never draw connecting lines between flask, adaptor and condenser — they are separate pieces



Safety precautions

- Wear Goggles, Gloves, and Lab Coat: Sulfuric acid is corrosive, dichromate is toxic and carcinogenic.
- . Use a Fume Cupboard (if possible): Ethanal is volatile and has a choking smell.
 - . Avoid Open Flames: Ethanol and ethanal are flammable - use a water bath or electric heater.
 - . Neutralise Waste: Potassium dichromate waste is hazardous and must be collected for safe disposal.

Possible Anomalous Results / Errors

1. **Low Yield of Ethanal:** Loss due to its volatility, Overheating causes oxidation to ethanoic acid.
2. **Contaminated Ethanoic Acid:** Incomplete oxidation or improper separation, Presence of unreacted ethanol or water due to poor distillation.
3. **Orange Colour Persists:** Insufficient heating, incomplete reduction of $\text{Cr}_2\text{O}_7^{2-}$.

Exam questions

Q1. (a) State the reagents and conditions to oxidise propan-1-ol to propanal. State the observation

- Reagents: acidified $\text{K}_2\text{Cr}_2\text{O}_7$ ($\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4$)
- Conditions: limited/small amount of oxidant, warm gently, distil out product as it forms
- observation: orange $\rightarrow$ green ($\text{Cr}_2\text{O}_7^{2-} \rightarrow \text{Cr}^{3+}$)

(b) Explain why distillation must be used rather than reflux for the preparation of propanal.

- Propanal vapour would return to flask during reflux and be further oxidised to propanoic acid
- distillation removes it immediately to prevent further oxidation

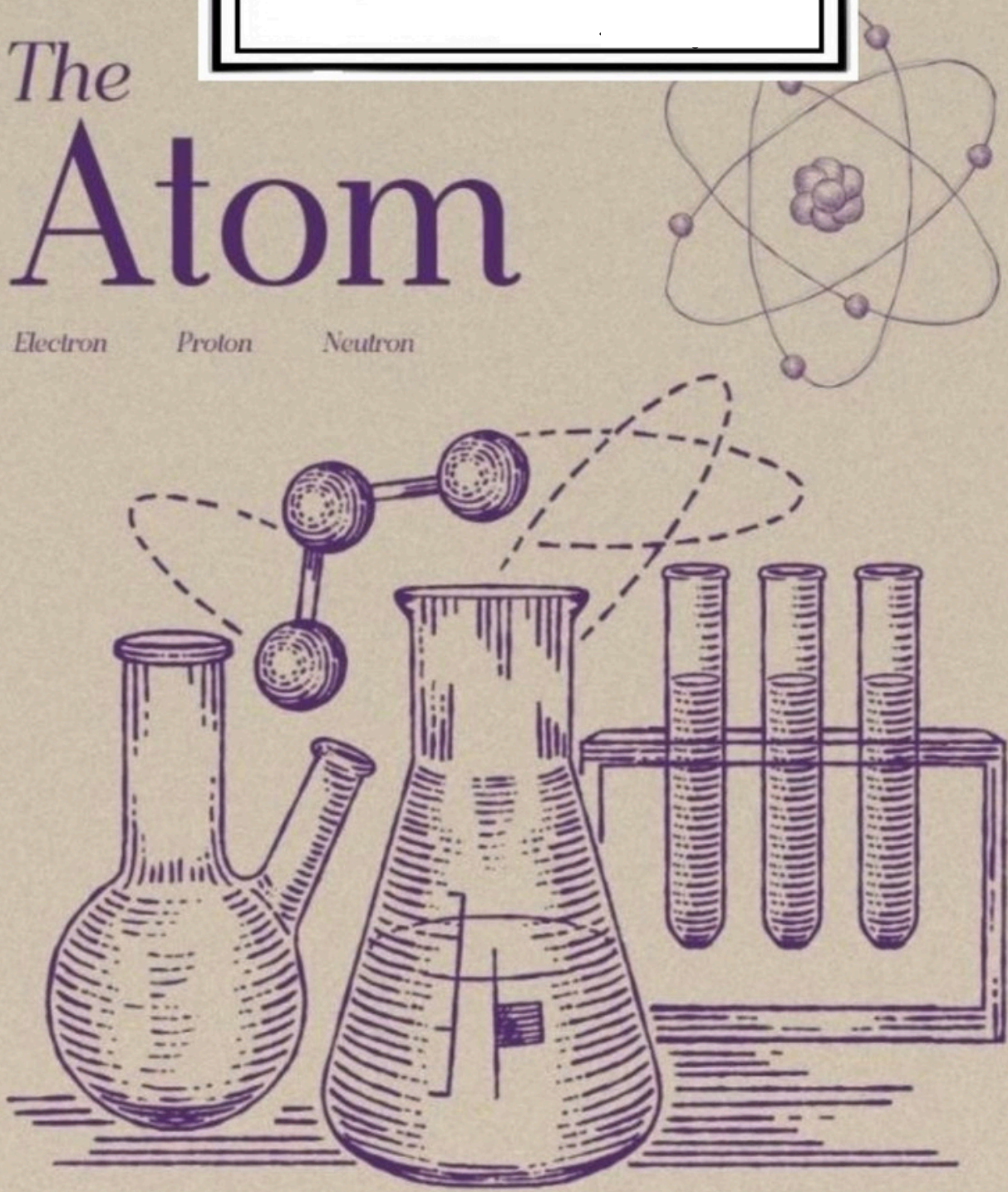
Q2. A student seals the top of the condenser with a bung. Explain why this is dangerous and what could happen.

- Organic vapours cannot escape. This causes pressure to build up
- flask or condenser could shatter/explode when pressure becomes too great

Core practical 08

The Atom

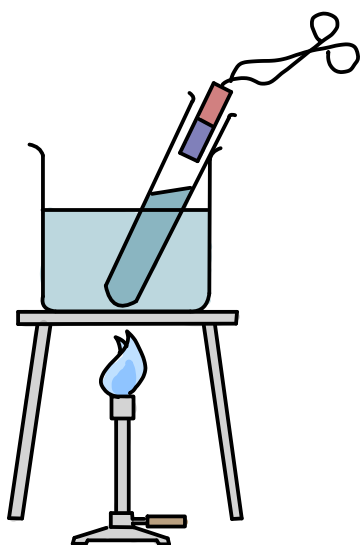
Electron Proton Neutron



CORE PRACTICAL 08: Test for anions, cations, gases and organic functional groups

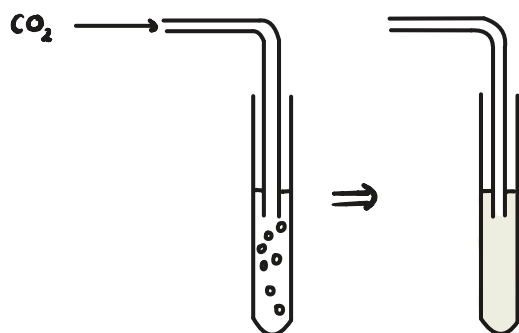
1. Ammonium Ion (NH_4^+):

- o Add sodium hydroxide solution and gently warm.
- o Test gas evolved with damp red litmus paper - it turns blue if ammonia is present.
- o Confirm with white fumes upon exposure to HCl



Carbonate (CO_3^{2-}) and Hydrogencarbonate (HCO_3^-):

- o Add dilute hydrochloric acid.
- o Observe effervescence (CO_2 gas).
- o Bubble gas through limewater - turns cloudy white (CaCO_3)



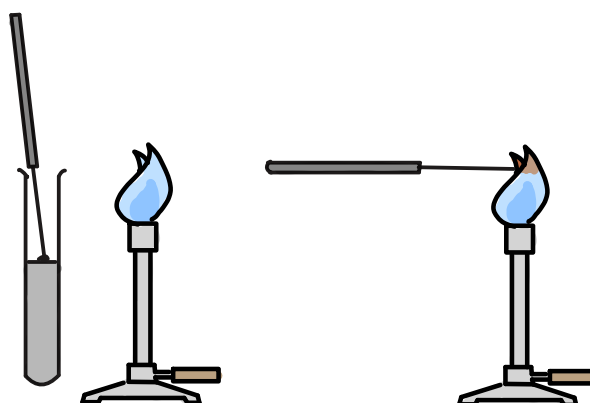
2. Flame Tests (Group 1 and 2 Cations):

- o Clean nichrome/platinum wire with HCl and heat in flame.
- o Dip in solid sample and place in Bunsen flame.

Reason: Electrons exist in discrete energy levels, so each element has different energy levels which are discrete. So only certain transitions are possible. When electrons gain energy, they jump to a higher energy level and then fall back to a lower energy level which emits a photon whose wavelength/frequency depends on the difference of the two energy levels. Some do not show any color like Mg as the wavelength of the photons emitted are in the UV spectrum.

OBSERVATIONS

- $\text{Li}^+ \rightarrow$ Red
- $\text{Na}^+ \rightarrow$ Yellow/orange
- $\text{K}^+ \rightarrow$ Lilac
- $\text{Rb}^+ \rightarrow$ Red/purple
- $\text{Cs}^+ \rightarrow$ Blue/violet
- $\text{Be}^{2+} \rightarrow$ No colour
- $\text{Mg}^{2+} \rightarrow$ No colour
- $\text{Ca}^{2+} \rightarrow$ (Brick) red
- $\text{Sr}^{2+} \rightarrow$ (Crimson) red
- $\text{Ba}^{2+} \rightarrow$ (apple) green



Sulfate (SO_4^{2-}) ions

- o Add dilute HCl, then BaCl_2 solution.
- o White precipitate of BaSO_4 confirms sulfate

Halide Ions (Cl^- , Br^- , I^-):

Add dilute nitric acid, then silver nitrate.

- . $\text{Cl}^- \rightarrow$ White ppt (soluble in dilute NH_3)
- . $\text{Br}^- \rightarrow$ Cream ppt (soluble in conc. NH_3)
- . $\text{I}^- \rightarrow$ Yellow ppt (insoluble in NH_3)

Test for Gases

1. Carbon Dioxide (CO_2): Bubble through limewater - turns milky.
2. Hydrogen (H_2): Insert a lit splint - 'pop' sound confirms presence.
3. Oxygen (O_2): Insert a glowing splint - relights in presence of O_2 .
4. Chlorine (Cl_2): Damp blue litmus paper turns red, then bleached white.
5. Ammonia (NH_3): Damp red litmus turns blue; white fumes with HCl.

Tests to identify organic compounds' functional groups

Alkenes

- Bromine water $\rightarrow$ a colour change from Brown to colourless
- Potassium Manganate and Sulfuric acid $\rightarrow$ a colour change from Purple to colourless

Alcohols (primary, secondary & tertiary)

- Adding Phosphorous (V) Chloride.
- Misty fumes (of HCl) are seen

Aldehydes

- Warmed with potassium dichromate(VI) and Sulfuric acid Orange to green
- Fehling's solution $\rightarrow$ Blue to brick red (precipitate)
- Benedict's solution $\rightarrow$ Blue to brick red
- Tollen's reagent $\rightarrow$ Silver mirror

Ketones $\rightarrow$ Fehling's solution, Benedict's solution, Tollen's reagent negative test

carboxylic acid $\rightarrow$ Add Carboxylic acid $\text{NaHCO}_3 \rightarrow$ Bubbles/effervescence (of CO_2)