# Important Points to Remember

- ➢ To ensure that drying process for a solid is complete, heat to constant mass. This ensures all water has been evaporated.
- ➢ Since alcohols are highly flammable, it is dangerous to heat them on a naked flame. Always use a water bath to heat alcohols and mixtures containing alcohols.
- ➢ $CO_2$, $NH_3$, HCl, $SO_2$ are water soluble gases (are polar); CO and $O_2$ are only slightly/sparingly soluble.
- ➢ $N_2$, $H_2$, $CH_4$, He are insoluble gases (non-polar).
- ➢ To increase reliability, repeat experiment trial multiple times and take average reading.
- ➢ Give burette readings to the nearest 0.05 $cm^3$, since burette can only measure up to ± 0.05 $cm^3$. It cannot take readings more accurate than this.
- ➢ In electrolysis, some deposited metal could possibly fall off the electrode while washing it, leading to a lower reading of mass than expected.
- ➢ While weighing a mass to prepare standard solution, weigh a larger mass to reduce percentage error and make experiment more accurate.
- ➢ Always use distilled water in experiments since tap water may contain dissolved ions which alter its boiling point and may interfere with experimental results.
- ➢ To keep temperature of a solution constant, place it in a thermostatically controlled water bath.
- ➢ If a heated solution is left without a lid, water may evaporate, changing the concentration of reactants.
- ➢ In any condenser, cold water always enters at the bottom.
- ➢ To control exothermic reactions, add reactants dropwise.
- ➢ Certain reaction enthalpies cannot be measured directly since it is impossible to know when the reaction is complete.
- ➢ If a reaction involved heating (eg. decomposition), it is difficult to measure the temperature while applying heat.
- ➢ Before starting an experiment where temperature is measured, allow system to equilibrate and reach the same temperature.
- ➢ In an experiment where temperature is measured, stirring the mixture will make results more reliable since this ensures uniform heating of solution.
- ➢ Initial trial experiments are often done to calibrate instruments.
- ➢ When asked how one variable varies with respect to another, answer in terms of directly/inversely proportional.
- ➢ Soda lime (mixture of CaO and $Ca(OH)_2$) is alkaline in nature.
- ➢ When a hot solid is being made to react with a gas, the gas may be passed over it before the solid is begun to be heated and after it has finished reacting. This is to prevent the solid from reacting with other gases in air.
- ➢ Instruments must be wiped to ensure they are clean and dry to prevent water/dirt does not interfere with readings.

- ➢ During titration, ensure that titre readings are concordant. If they are not consistent/close to each other, repeat titration until concordant readings are obtained.
- ➢ If indicator and one of the reactants used is the same colour, change the indicator to one which displays a different colour to improve the accuracy of results.
- ➢ To eliminate anomalous readings, repeat the experiment for those values.
- ➢ While doing enthalpy change experiments, it is important to measure the temperature before and after the reaction.
- ➢ In a decomposition reaction, if heating is very strong, time measurement will be very small, increasing the percentage error and reducing accuracy of reaction. At the same time, underheating may not ensure that all of the solid has decomposed.
- ➢ How to know when reaction is complete:
    - Colour change of indicator/reactants/products
    - No more bubbles seen
    - No more mass increase seen
    - Temperature reaches its maximum and then starts to fall (for exothermic reactions)
    - Temperature reaches its minimum and then starts to rise (for endothermic reactions)
- ➢ Possible observations of reactions:
    - Solid/precipitate formed (if coloured then mention colour)
    - Solid disappears (if it reacts/dissolves in liquid)
    - Solid remains (if no visible change occurs to the solid)
    - Coloured gas evolved (mention colour); eg. Brown gas evolved ($NO_2$)
    - Bubbles of gas/Effervescence
    - Gas relights glowing splint (Oxygen)
    - Gas extinguishes glowing splint/Gas turns lime water milky (Carbon Dioxide)
    - Gas turns damp red litmus blue (Ammonia)
    - Gas smells of bad eggs ($H_2S$ – it is also flammable)
    - Gas bleaches litmus paper white (Chlorine
    - Condensation ($H_2O$) formed on sides of test tube (dehydration of hydrated salt)

# Methods

1. **Weighing by difference using a weighing boat:**
    i. Boat + substance is weighed
    ii. Substance is transferred
    iii. Empty boat reweighed

Table to record the process – E.g. to weigh AgNO3

| | /g |
|---|---|
| Mass of boat + AgNO3 before transfer | |
| Mass of boat after transfer | |
| Mass of AgNO3 transferred | |

Note: don't reverse the order of weighing since some of the substance might remain in the boat as a residue after the transfer, giving an inaccurate reading of mass transferred.

2. **Preparing 250.0cm3 of a solution, starting from a stated mass of solid in a 100cm3 beaker:**
    - Dissolve the solid / in the beaker using a small volume of distilled water.
    - Transfer / add to a 250cm3 volumetric flask and rinse beaker with distilled water.
    - Top the 250 cm3 volumetric flask up to mark with distilled water.
    - Invert the volumetric flask with stopper and shake to mix.

3. **Preparing clean burette for titration**
    - Run some distilled water through the burette to ensure it is clean
    - Rinse / wash (50 cm3 burette) with (stated conc.) of solution, before filling with the solution (to remove water).
    - Place the solution in the burette to fill (using funnel) AND then run some solution out through the jet by opening the tap (to eliminate air bubbles).

4. **To calculate mean titre to be used in calculations:**
    - Select at least 2 titre values <= 0.10 cm3 apart.

5. **Carrying out accurate titration**
    Example:

    **step 6** Carry out **one** accurate titration of all the contents in the conical flask with 0.00200 mol $dm^{-3}$ aqueous sodium thiosulfate, $Na_2S_2O_3(aq)$, using starch indicator.

    Describe the procedure for carrying out the one accurate titration in step 6.

M1

- add / run Na2S2O3(aq) into the conical flask.
- until permanent colour change seen / end-point reached.

M2

- add dropwise towards the end to ensure the end-point is accurate.

6. **Table for recording titration results for five samples A–E:**

| | (titration) A | (titration) B | (titration) C | (titration) D | (titration) E |
|---|---|---|---|---|---|
| final (burette) reading / $cm^3$ | | | | | |
| initial (burette) reading / $cm^3$ | | | | | |
| titre / $cm^3$ | | | | | |

7. **How the reliability of results could be improved.**
   - Repeat experiment (x3) and take an average.

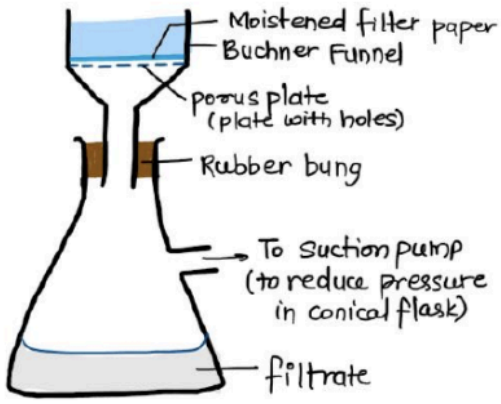
8. **How glassware should be dried before use.**
   - Warm in an oven

9. **Suggest what should be done before recording maximum temperature reached to improve the experimental procedure.**
   - stir the solution before taking reading

# Apparatus

# APPARATUS SETUP

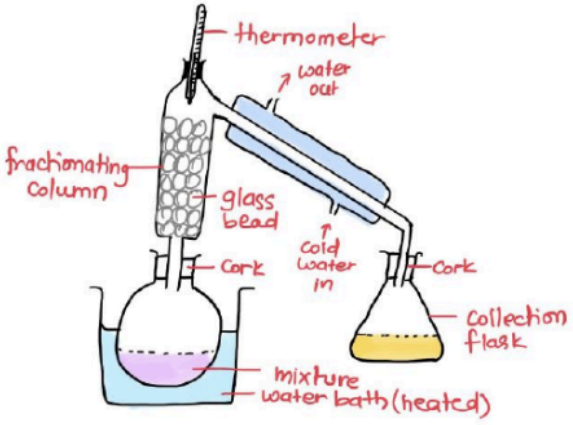
## FILTRATION UNDER REDUCED PRESSURE




A vacuum in the flask underneath the paper allows atmospheric pressure on the sample to force the liquid through the filter paper.

Advantage – faster filtration

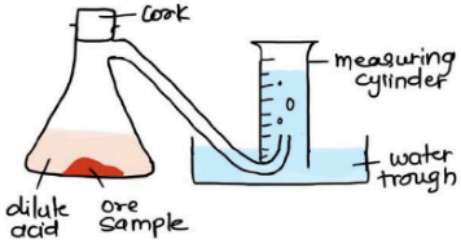
## FRACTIONAL DISTILLATION




Liquid with lower boiling point is collected first

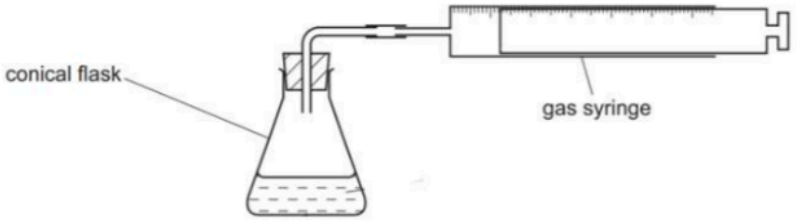
<u>IMPORTANT</u>: The bulb of the thermometer should be at the front of the tube opening where the gas leaves the fractionating column.

## COLLECTING GAS OVER WATER



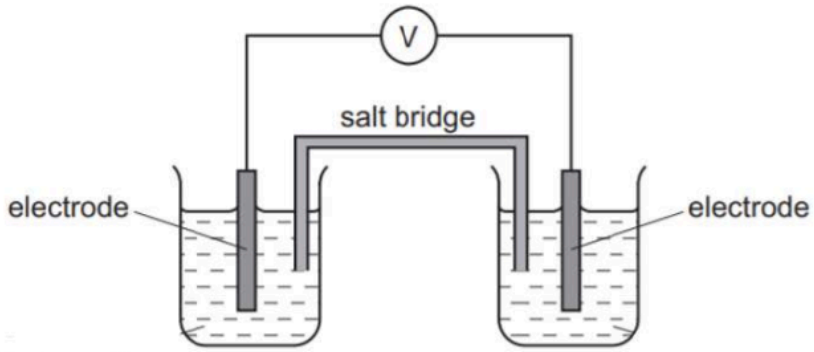

- Gas should not be water soluble
- Inverted measuring cylinder should be fully filled with water at the beginning of the experiment
- Never use first sample of gas since this may contain air from apparatus and not just pure gas
- **Risk of suck back** – the creation of a vacuum/region of low pressure in the container due to collection of gas over water causes the water to be sucked back into the reaction container, which is not desirable

## COLLECTING GAS IN A GAS SYRINGE

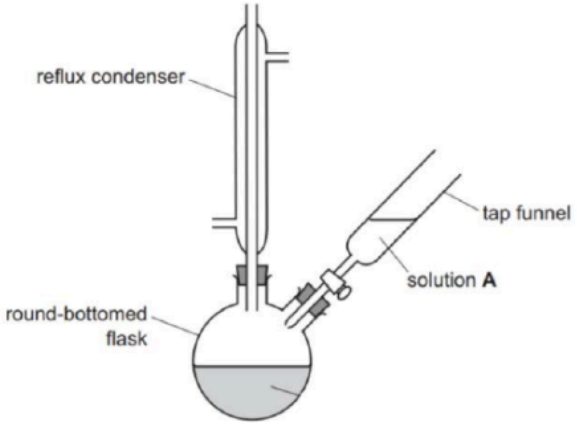



Never use first sample of gas from a reaction since this may contain air from apparatus and not just pure gas

## SETUP FOR ELECTROLYSIS




## PERFORMING A REACTION UNDER REFLUX



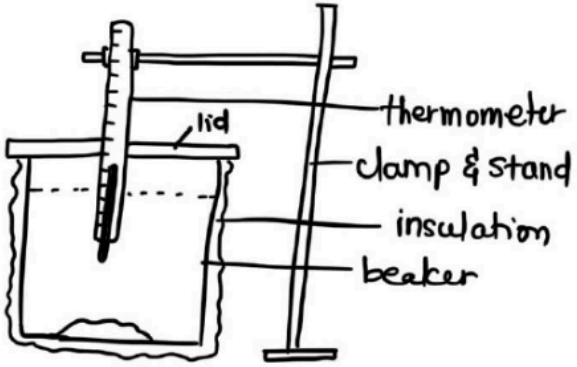

**Don't put a bung** at the top of the condenser since heating causes gases to expand.

So the pressure would build up, causing the apparatus to shatter.

Reflux is used to heat mixture to

a. Increase rate of reaction
b. Prevent loss of vapour

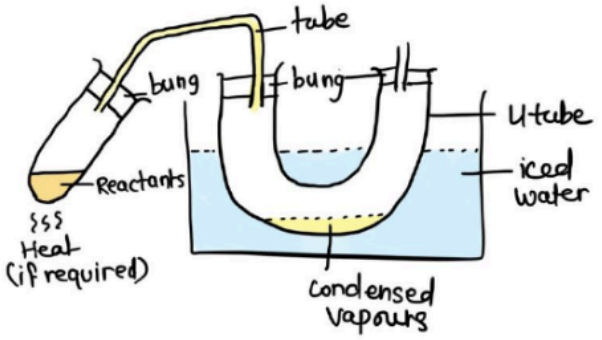
## Measuring temperature change of a reaction




Stir the liquid if possible with the thermometer to ensure accurate readings

Insulate beaker to prevent heat loss

## COLLECTING A CONDENSED GAS FROM A REACTION




Allows collection of gases with different boiling points (kind of reverse of fractional distillation)

Gas with lower boiling point will condense first, other gas will escape.

Must leave other end of U tube open to prevent pressure build up.

## General rules

- Measuring cylinder is used to add a volume of reagent in excess.
- Volumetric pipette is used for 10 – 25 cm3 (10, 15, 20, 25)
- Burette is used for <10cm3 and >25cm3
- To measure 10.00cm3, use 10.0cm3 volumetric pipette; to measure 10.0cm3, use 10cm3 volumetric pipette (1d.p. less)

Examples

- **To transfer 10.00cm3 of a solution:** 10cm3 volumetric pipette
- **To transfer 1cm3 of a solution:** Dropping pipette (as 1 cm3, not 1.00 cm3 is asked for)
- **To prepare 500.0cm3 of 0.00200 mol dm–3 $Na_2S_2O_3(aq)$ after the required mass of $Na_2S_2O_3x5H_2O(s)$ has been weighed out:** 500 cm3 volumetric flask
- **To transfer 50.0cm3 of solution:** Burette
- **To measure 1.15cm3:** Burette

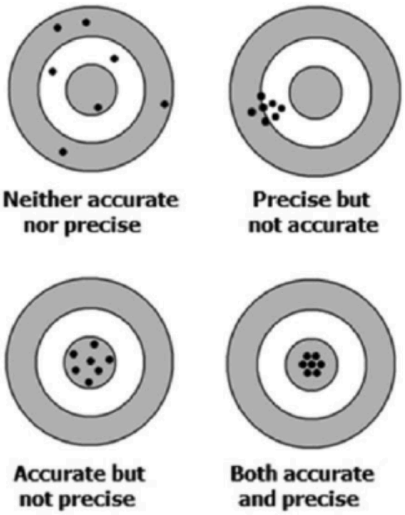
1. **Function of salt bridge:** To allow movement of ions between half cells

2. **Suggest a change to apparatus that would reduce the heat lost:** Use a lid

# Errors

**Systematic error:** measurement errors caused by measurements differing from the true value by a consistent amount for each measurement that is made.

**Random error:** measurement error caused by measurements varying unpredictably from one measurement to the next.




**Sources of error (examples):**

- Heat loss in experiments where enthalpy is being measured (not all heat from heater is transferred to reaction mixture/some heat produced in reaction is lost to surroundings).
- In some titrations, it is difficult to judge the end point of the reaction since colour changes may be subjective/difficult to discern.
- Insufficient capacity of apparatus may cause reactants to splash out/escape (e.g: filling a beaker to the very brim).

**To make readings more precise/accurate:**

- Changes in apparatus: use burette instead of measuring cylinder, use thermocouple instead of lab thermometer, use pH meter instead of pH paper.
- Repeating readings to identify and eliminate anomalies and make results more reliable.
- Performing experiments with larger quantities of reactants, to reduce percentage error.
- Changes in method: using weighing boat method to measure mass transferred, using lower meniscus for accurate volume measurements.

NOTE: Uncertainty is ± half of the smallest scale division of an instrument.

$$\%\ uncertainty = \frac{uncertainty \times no.of\ times\ measurement\ is\ taken\ using\ instrument}{measurement\ or\ quantity} \times 100$$

1. **Burette**
   - ½ smallest division = 0.05cm3
   - For titre readings: final - initial, so total error = 2 x 0.05 = 0.1cm3

Explanation: If the least count of a burette is 0.1 cm3 then the uncertainty/error in measurements could be ±0.05 cm3. Uncertainty is ± half of the smallest scale division of an instrument. When the burette is used to measure a change in volume of 20 cm3, that means that volume is being read twice – initial volume and final volume. Thus, to find percentage uncertainty:

$$\%\ uncertainty = \frac{0.05 \times 2}{20} \times 100$$

2. **Syringe**

Titration can be used to determine the concentration of dissolved oxygen in samples of river water.

The procedure for the experiment is given.

**step 1** Use five 50 $cm^3$ graduated syringes, **A**, **B**, **C**, **D** and **E**, to collect five separate 30.0 $cm^3$ samples of river water.

**step 2** In the laboratory, carefully add 5.0 $cm^3$ of 0.220 mol $dm^{-3}$ manganese(II) sulfate, $MnSO_4(aq)$, into syringe **A** and mix well.

**step 3** Add 5.0 $cm^3$ of alkaline aqueous potassium iodide into syringe **A** and mix well.

**step 4** Add 10.0 $cm^3$ of dilute sulfuric acid into syringe **A** and mix well.

**step 5** Transfer the contents of syringe **A** into a 150 $cm^3$ conical flask. Rinse syringe **A** using 10 $cm^3$ of distilled water and add washings to the conical flask.

**step 6** Carry out **one** accurate titration of all the contents in the conical flask with 0.00200 mol $dm^{-3}$ aqueous sodium thiosulfate, $Na_2S_2O_3(aq)$, using starch indicator.

Given that: The graduations on each syringe are every 1.0cm3. Calculate the percentage error in the measurement of 5.0cm3 of alkaline aqueous potassium iodide by the syringe.

Solution:

$\frac{2 \times 0.5}{5.0} \times 100$ = 20(.0%)

- ½ smallest division = 0.5cm3

- In this case, solutions are being added one by one into the syringe: so you look at the initial level, and the final level for each solution added. The difference in initial and final levels gives the volume of solution added, so total error = 2 x 0.5.

3. **Effect on measurement uncertainty and error percentage, when a larger volume of solution is measured by the same apparatus.**
    - Effect on uncertainty of the measurement = no effect
    - Effect on percentage error of the measurement = smaller effect

NOTE: when labelling condensers, label the errors for water entry and exit as ‘water in’ and ‘water out’.

4. **The experiment is repeated and the value of q0 is calculated to be 78.1mg–1. The total percentage error from the experimental procedure is 6.5%. The data book value of q0 is 86.0mg–1. Use this information to determine whether the error in the repeated experiment could bw accounted for by experimental errors or is caused by other factors.**

    percentage error of experimental result compared to data book value
    $= \frac{(86.0 - 78.1)}{86.0} \times 100\% = 9.2\%$
    **AND**
    9.2% > 6.5%
    **AND**
    other factors contributed towards error

Justifying why results are reliable:
- Because measurements are close to each other.
- Because all points lie either on or very close to the line of best fit.
- Because there are no anomalous points.

# Reasoning Questions

1. **Suggest why a solution of silver nitrate, AgNO3(aq), is kept in a dark brown glass bottle rather than a colourless glass bottle.**
    - Sunlight decomposes silver nitrate

2. **Titration with Ag+(aq) is used to determine concentration of Cl-(aq) in seawater. Spectroscopic analysis of the sample of sea water accurately determined the concentration of Cl−(aq) to be lower than that determined by titration. Suggest why titration gave a higher value.**
    - Ag+ ions react with other substances in the sample of sea water.

3. **Iced water in a conical flask is used to significantly reduce rate of reaction. Suggest 2 reasons why rate of reaction is significantly reduced when the reaction mixture is transferred to this conical flask.**
    - Lowers the temperature of reaction mixture
    - Decreases concentration in reaction mixture

NOTE: Most common control variables:
- Volumes & concentrations of solutions
- Temperature

4. **State whether the data from the experiment is reliable. Justify your answer.**
    - No and there is an anomalous point on the graph OR
    - Yes and all the points lie on/close to the line of best fit

5.

**step 1** Use five 50 $cm^3$ graduated syringes, **A**, **B**, **C**, **D** and **E**, to collect five separate 30.0 $cm^3$ samples of river water.

**step 2** In the laboratory, carefully add 5.0 $cm^3$ of 0.220 mol $dm^{-3}$ manganese(II) sulfate, $MnSO_4(aq)$, into syringe **A** and mix well.

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**step 4** Add 10.0 $cm^3$ of dilute sulfuric acid into syringe **A** and mix well.

**step 5** Transfer the contents of syringe **A** into a 150 $cm^3$ conical flask. Rinse syringe **A** using 10 $cm^3$ of distilled water and add washings to the conical flask.

**step 6** Carry out **one** accurate titration of all the contents in the conical flask with 0.00200 mol $dm^{-3}$ aqueous sodium thiosulfate, $Na_2S_2O_3(aq)$, using starch indicator.

Repeat steps 2–6 for the samples in syringes **B**–**E**.

a. **Suggest why the reaction mixture is mixed well in steps 2–4.**
    - to ensure the reaction is / are complete in the syringe.

**b.** Freshly distilled water does not contain any dissolved oxygen. A student decides to run the procedure on a sample of freshly distilled water and at the end obtains a value of 2.26 × 10–5moldm–3 dissolved oxygen. Suggest why the student did not get a value of 0moldm–3. Assume the procedure was carried out correctly.

- there was a small amount of dissolved oxygen in the other solutions / reagents / reactants used in the experiment.
  (they had given the entire reaction equation, and only oxygen from that set of reagents reacted)

**c.** Suggest how the value of 2.26 × 10–5moldm–3 could be used to improve the answer obtained for concentration, in moldm–3, of dissolved oxygen in the river water.

- subtract the result 2.26 10–5 mol dm–3 of the distilled water experiment from the final result for concentration of dissolved oxygen in river water.

**d.** Suggest why this method is unsuitable for samples of tap water that have been purified by chlorination and so contain Cl2(aq).

- chlorine is an oxidising agent OR chlorine reacts in the same way as oxygen OR chlorine reacts with Mn2+ / S2O32– / I– /reactants

**6.**

The activation energy, $E_A$, for the reaction between dilute hydrochloric acid, $HCl(aq)$, and aqueous sodium thiosulfate, $Na_2S_2O_3(aq)$, can be determined by an initial rates method.

$$2HCl(aq) + Na_2S_2O_3(aq) \rightarrow 2NaCl(aq) + H_2O(l) + S(s) + SO_2(g)$$

The solid sulfur formed is seen as a white suspension in the reaction mixture. The reactants are mixed and the time, $t$, for a fixed quantity of sulfur to be formed is recorded.

A measure of the initial rate of the reaction is $\frac{1}{t}$.

Standard solutions of $0.100\,mol\,dm^{-3}$ $Na_2S_2O_3(aq)$ and $0.500\,mol\,dm^{-3}$ $HCl(aq)$ are supplied.

Measurements are taken for a series of temperatures using the following procedure.

**step 1** A thermostatically controlled water bath is set up.

**step 2** A $100\,cm^3$ conical flask is labelled **A** and a second $100\,cm^3$ conical flask is labelled **B**.

**step 3** $10.00\,cm^3$ of $0.100\,mol\,dm^{-3}$ $Na_2S_2O_3(aq)$ is added to flask **A**. Flask **A** is placed in the water bath.

**step 4** $10\,cm^3$ of $0.500\,mol\,dm^{-3}$ $HCl(aq)$ is added to flask **B**. Flask **B** is placed in the same water bath.

**step 5** Wait for 10 minutes.

**step 6** Flask **A** is removed from the water bath and placed on a tile marked with a black cross.

**step 7** The contents of flask **B** are added to flask **A** and a timer started.

**step 8** The timer is stopped when the black cross is no longer visible. The time is recorded.

**a.** Suggest a reason why it is necessary to wait for 10 minutes in step 5.

- To ensure the solutions in flasks A and B are at the same temperature of the water bath before mixing.

**b.** The procedure does not mention how a value for the temperature of the mixture during the reaction is obtained. State the temperature measurements that should be taken and at which stage in the procedure they should be taken.

- take the temperature of reaction mixture in flask A at the start of reaction / step 7 OR immediately before step 7 OR step 6 / immediately before the reaction OR immediately after step 5
  AND at the end of reaction / step 8 / when cross is no longer visible / immediately after step 8.

**c.** State how to use the temperature measurements to determine an accurate value for the temperature of the mixture during the reaction.

- calculate the mean temperature during the reaction.

**d.** Suggest a change to one controlled variable that the student could make so that the time measured for a given temperature is shorter.
- increase the concentration of one or both of the reactants.

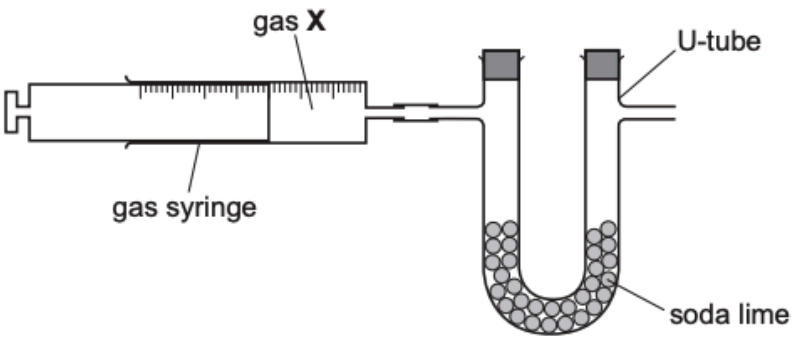
**7. Explain why the first sample of gas collected should not be used for calculations of volume, etc.**
- The sample will contain air from the apparatus OR gas mixture not pure gas.

NOTE: unit of activation energy = kJ mol–1.

**8. Give 2 reasons why gas collection over water may not be suitable.**
- There is danger of suck back
- Gas is soluble in water

**9.**

The student is told to use the U-tube shown to find the mass of a sample of gas **X**.



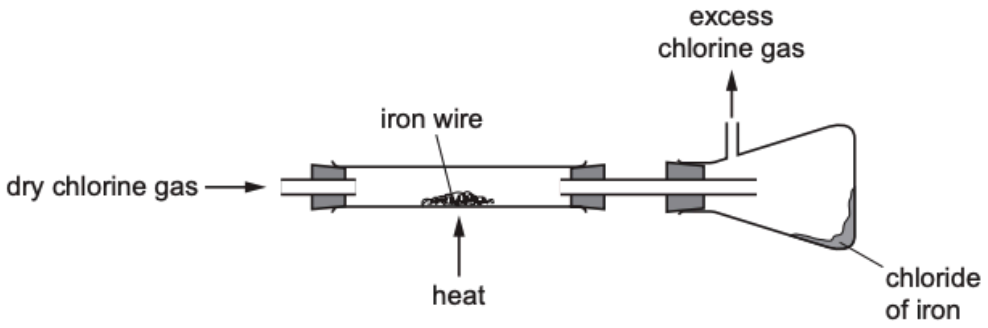

A 100.0 $cm^3$ sample of pure gas **X** is placed in a gas syringe. The gas syringe is attached to a U-tube containing small lumps of solid soda lime, a mixture of sodium hydroxide and calcium hydroxide. All of gas **X** is slowly passed into the U-tube and the mass of gas **X** absorbed determined.
The temperature and the pressure of the room are recorded.

**a. State the measurements that are needed to determine the mass of gas X absorbed.**
- Mass of U-tube + soda lime before the experiment AND Mass of U-tube + soda lime after the experiment

**b. Suggest why soda lime is used to absorb gas X.**
- Soda lime is alkaline / a neutralisation reaction will occur.

**10.**

Each student in a class of nine students performs an experiment to find the formula of a chloride of iron. Each student prepares the chloride of iron by passing a stream of chlorine gas over a sample of iron wire as shown in the diagram.




**(a)** Each student carries out the following steps in the order shown in the list. Some of the measurement steps are missing from the list.

- Weigh the reaction tube containing a quantity of iron wire.
- Set up the apparatus as shown in the diagram.
- Start the flow of dry chlorine gas.
- Heat the iron wire until it has completely reacted.
- Allow the apparatus to cool, with chlorine gas still flowing.
- Weigh the side-arm conical flask containing the chloride of iron.

**a. State the two additional measurement steps that each student must perform in order to find the formula of the chloride of iron.**

- Mass of empty reaction tube
- Mass of empty side-arm conical flask

**b. The flow of dry chlorine gas must start before the iron wire is heated. Explain why.**

- The iron could react with oxygen or air (instead of the chlorine).

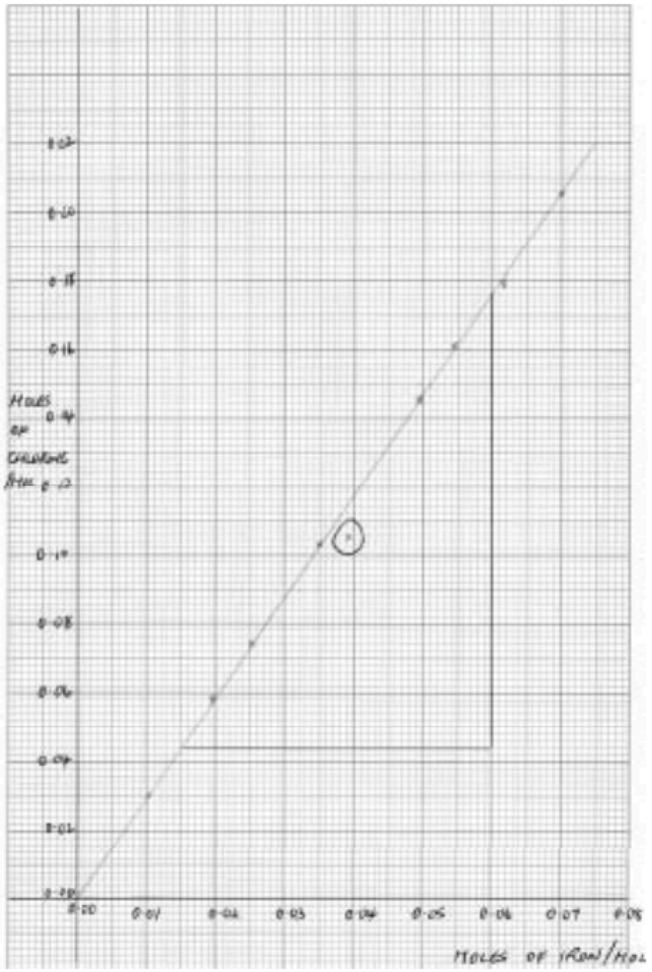
**c. State an assumption that has to be made for the measurements made in this experiment to be valid.**

- the mass of air in the flask is equal to mass of chlorine in the flask
- all the iron (wool) has reacted
- any residue in the reaction tube is iron

Assumption 1: Before the reaction, the side-arm flask contains air. After the reaction, it contains chlorine gas + iron chloride. When calculating the mass of iron chloride, we assume that: replacing air with chlorine does not change the mass of gas inside the flask. If this were not assumed: The mass increase of the flask could be partly due to chlorine gas, not just solid iron chloride. That would cause an overestimation of the chlorine that reacted

Assumption 3: After heating, some solid may remain in the reaction tube. We assume this residue is unreacted iron, not: iron chloride or iron oxide (from oxygen contamination). If this assumption is false: Some iron chloride might be left behind. The mass of iron chloride collected in the flask would be too low. This would give an incorrect mass (and moles) of chlorine.

**d.** Graph obtained for mol of chlorine vs. mol of iron:



| Student | Mass of iron / g | Mass of iron chloride / g | Mass of chlorine / g | Moles of iron | Moles of chlorine |
|---|---|---|---|---|---|
| 1 | 0.57 | 1.64 | 1.07 | 0.0102 | 0.0301 |
| 2 | 1.10 | 3.16 | 2.06 | 0.0197 | 0.0580 |
| 3 | 1.40 | 4.03 | 2.63 | 0.0251 | 0.0741 |
| 4 | 1.95 | 5.61 | 3.66 | 0.0349 | 0.103 |
| 5 | 2.18 | 5.89 | 3.71 | 0.0391 | 0.105 |
| 6 | 2.75 | 7.90 | 5.15 | 0.0493 | 0.145 |
| 7 | 3.05 | 8.77 | 5.72 | 0.0547 | 0.161 |
| 8 | 3.45 | 9.80 | 6.35 | 0.0618 | 0.179 |
| 9 | 3.90 | 11.18 | 7.28 | 0.0699 | 0.205 |

**Circle the point on the graph you consider to be most anomalous. Suggest one reason why this anomaly may have occurred during this experimental procedure.**

- Viable reason why the number of moles of chlorine is too small: e.g. not all the mass of iron chloride formed was collected in the conical flask OR Iron chloride dust particles escaped with excess chlorine gas.

**11. In experiment (a) iron chloride is prepared by iron + dry chlorine gas, forming $FeCl_3$. In experiment (f), it is prepared by iron + HCl, forming $FeCl_2$.**

| reaction | electrode potential, $E^{\ominus}$/V |
|---|---|
| $Fe^{2+}(aq) + 2e^- \rightleftharpoons Fe(s)$ | –0.44 |
| $2H^+(aq) + 2e^- \rightleftharpoons H_2(g)$ | 0.00 |
| $Fe^{3+}(aq) + e^- \rightleftharpoons Fe^{2+}(aq)$ | +0.77 |

**Explain, using the electrode potential values in the table, why the methods in (a) and (f) do not produce the same chlorides of iron.**

- In method (f), conversion of Fe to Fe2+ takes place as EΘ cell is +0.44V AND Conversion of Fe2+ to Fe3+ is not feasible as EΘcell is –0.77V. (In (a), you give heat, so even negative Ecell is fine.)

**12. Suggest how water can be removed from ethoxyethane.**

- Fractional distillation

**13.**

Grignard reagents have the general formula RMgX, where R is a hydrocarbon group and X is a halogen. The Grignard reagent $C_6H_5MgBr$ is used as an intermediate in the reaction between bromobenzene, $C_6H_5Br$, and ethanal, $CH_3CHO$, to prepare 1-phenylethanol, $C_6H_5CH(OH)CH_3$. An organic solvent, ethoxyethane, is used.

The equations for the three reactions that take place during the preparation are shown.

reaction 1 $Mg + C_6H_5Br \rightarrow C_6H_5MgBr$

reaction 2 $C_6H_5MgBr + CH_3CHO \rightarrow C_6H_5CH(CH_3)OMgBr$

reaction 3 $C_6H_5CH(CH_3)OMgBr + H^+ \rightarrow C_6H_5CH(OH)CH_3 + Mg^{2+} + Br^-$

The preparation involves the following steps.

**step 1** Set up the apparatus shown in Fig. 1.1 with approximately 1.25 g of Mg powder and 5 $cm^3$ of ethoxyethane in the round-bottomed flask.

**step 2** Add 0.0500 mol of liquid $C_6H_5Br$ to the round-bottomed flask dropwise using the tap funnel. Leave until reaction 1 is complete.

**step 3** Dissolve 3.00 $cm^3$ of $CH_3CHO$ in 15 $cm^3$ of ethoxyethane and add this solution to the round-bottomed flask using the tap funnel. Leave until reaction 2 is complete.

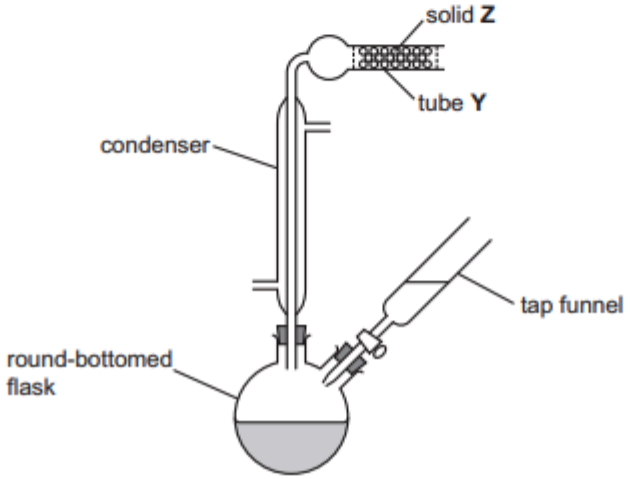
**step 4** Remove the condenser, tube **Y** and the tap funnel from the round-bottomed flask.

**step 5** Add 40 $cm^3$ of dilute hydrochloric acid, $HCl(aq)$, to the round-bottomed flask so that reaction 3 takes place.

**step 6** Transfer the contents of the round-bottomed flask to a separating funnel. Allow the liquids to settle so that two layers are formed.

**step 7** Open the tap of the separating funnel and run the lower layer into a beaker labelled **A**. Run the upper layer into a beaker labelled **B**.

**step 8** Allow the ethoxyethane to evaporate from the beaker containing $C_6H_5CH(OH)CH_3$.




**a. Suggest why solid Z is used.**

- to prevent water getting into the apparatus

**b. Suggest why the apparatus does not have a bung at the end of tube Y.**

- to avoid pressure build up in the apparatus

**14. In a separating funnel with ethoxethane and water, which layer is on top and why?**

- Ethoxyethane
- It is less dense than water

NOTE:

| | |
|---|---|
| Enthalpy change of combustion | Always negative |
| Enthalpy change of hydration | Always negative |
| Lattice energy | Always negative |
| Enthalpy change of formation | positive/negative/zero |

**15.**

The enthalpy change of combustion, $\Delta H_c$, of butane, $C_4H_{10}$, can be determined using the apparatus shown in Fig. 2.1.

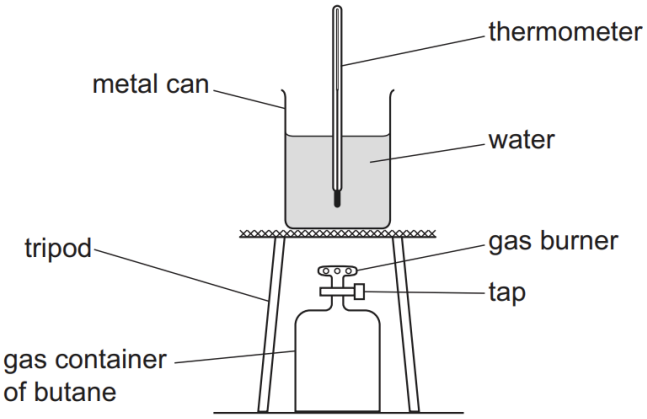



**Fig. 2.1**

The following steps are carried out.

**step 1** Use a 500 $cm^3$ measuring cylinder to transfer 320 $cm^3$ of water into a metal can.

**step 2** Place a thermometer into the water. Record the initial temperature of the water in the metal can.

**step 3** Weigh the gas container with burner and record the initial mass.

**step 4** Set up the apparatus as shown in Fig. 2.1.

**step 5** Light the burner and allow the flame to heat the water in the metal can for three minutes.

**step 6** Switch off the burner and record the maximum temperature reached.

**step 7** When cool, reweigh the gas container with burner and record the final mass.

| initial temperature of water / °C | maximum temperature of water / °C | change in temperature of water, $\Delta T$ / °C | initial mass of gas container with burner / g | final mass of gas container with burner / g | mass of butane burned / g |
|---|---|---|---|---|---|
| 19.3 | 76.6 | | 183.56 | 181.46 | |

$q = mc\Delta T$ to calculate the energy, $q$, in J, gained by the water.

Calculate the enthalpy change of combustion, $\Delta H_c$, of butane, in kJ mol$^{-1}$.

**The experiment was repeated but the burner was switched off after only two minutes.**

**a. Suggest why this might contribute to a reduction in the accuracy of ΔHc of butane**

- percentage error (of temperature change or mass change of butane) increases

**b. Suggest why this might contribute to an increase in the accuracy of ΔHc of butane.**

- less heat loss

**16. Suggest why a conical flask might be shaken**

- To ensure a uniformly mixed sample

# Numericals

1. **From 500.0cm3 of 0.139moldm−3 solution, calculate the volume of that solution needed to make 100.0cm3 of 0.00556moldm−3 dilution.**

$$C_1V_1 = C_2V_2$$

$$\Rightarrow 0.139 \times \frac{V_1}{1000} = 0.00556 \times \frac{100}{1000}$$

$$\Rightarrow V_1 = \boxed{4.0}\ cm^3$$

Sea water contains about 20 g $dm^{-3}$ of chloride ions, $Cl^-(aq)$.

The exact concentration of $Cl^-(aq)$ in sea water can be determined by titration with aqueous silver ions, $Ag^+(aq)$, using aqueous potassium chromate(VI), $K_2CrO_4(aq)$, as an indicator.

When aqueous silver nitrate, $AgNO_3(aq)$, is added to a sample of sea water, silver ions react with chloride ions to form a precipitate of silver chloride.

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

When all of the $Cl^-(aq)$ has reacted with $Ag^+(aq)$, the presence of unreacted $Ag^+(aq)$ is detected by chromate(VI) ions, $CrO_4^{2-}(aq)$. A red precipitate of $Ag_2CrO_4(s)$ is seen.

$$2Ag^+(aq) + CrO_4^{2-}(aq) \rightarrow Ag_2CrO_4(s)$$

The amount of $Ag^+(aq)$ reacting with $Cl^-(aq)$ in the sample of sea water can be calculated in order to determine the concentration of $Cl^-(aq)$ in the sample of sea water.

A student uses the following method.

**step 1** Use a weighing boat to weigh by difference approximately 10.6 g of $AgNO_3(s)$ into a 100 $cm^3$ glass beaker.

**step 2** Use the sample of $AgNO_3(s)$ in the glass beaker to prepare 250.0 $cm^3$ of $AgNO_3(aq)$.

**step 3** Transfer this solution into a dark brown glass bottle. Label this solution **X**.

**step 4** Collect a sample of sea water and remove any solid material present.

**step 5** Transfer 10.00 $cm^3$ of the sea water into a conical flask.

**step 6** Add 1 $cm^3$ of $K_2CrO_4(aq)$ to the conical flask.

**step 7** Rinse a burette in preparation for the titration.

**step 8** Fill the burette with solution **X**.

**step 9** Slowly add solution **X** to the conical flask until the white precipitate turns red. This is the end-point.

Mean titre of solution X = 22.40 cm3

**Use the mean titre to calculate the concentration of chloride ions in the sample of sea water. Assume the mass of solid silver nitrate used in step 2 was 10.62g.**

M1 Mol $AgNO_3$ used = 10.62 / 169.9 = 0.0625… (mol)

M2 Mol $Ag^+(aq)$ reacting with $Cl^-(aq)$
= M1 × 22.40/250
= $5.60 \times 10^{-3}$ (mol)

M3 Conc $Cl^-(aq)$ = M2 × 1000 / 10
= 0.560 (mol $dm^{-3}$)

NOTE: red ppt is just an indication that all the Cl– has reacted.

# Safety Precautions

- When handling toxic solutions:
    - Wear safety goggles
    - Wear chemically resistant gloves
- Wear heat resistant gloves while handling heated apparatus.
- Do not dispose solutions which are toxic to aquatic life/pose environmental hazards down the sink.
- Keep alcohols and flammable compounds away from naked flames.
- In experiments in which toxic gases are evolved that pose a health hazard, perform experiment in fume cupboard.
- Wear a mask while performing experiments where compounds are harmful by inhalation/if swallowed.
- Use insulation while performing an experiment involving temperature changes being measured.
- Ensure that instruments have no zero error. If a zero error exists, be sure to add/subtract the error as appropriate to avoid systematic error.