



# HL IB Chemistry

  
Your notes

## Tool 1: Experimental Techniques

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- \* Safety, Ethical & Environmental Issues in Chemistry
- \* Measuring Variables in Chemistry
- \* Applying Techniques in Chemistry



Your notes

## Safety, Ethical & Environmental Issues in Chemistry

### Safety, Ethical & Environmental Issues in Chemistry

- Throughout the course, you will perform a variety of different experiments
- You need to be aware of the **hazards** and **risks** that accompany practical work and be able to suggest ways to **minimise** these risks
- Risks may be **chemical** or **environmental**
  - **Environmental** risks arise due to the potential harm that the materials used and the products formed can have on our environment
- Some investigations may have **ethical** implications

### Hazards & Risks






- The **hazard** of a chemical substance relates to the inherent properties of the substance
- The **risk** is associated with how you plan to use it, the chance of it causing harm and the severity of the harm that it can cause
- Take hydrochloric acid as an example:
  - The **hazard** associated with hydrochloric acid depends on its concentration:
    - Concentrated HCl is classified as corrosive
    - Mildly concentrated HCl is classified as an irritant
    - Dilute HCl is not classified as hazardous
  - The **risk** associated with using hydrochloric acid depends on
    - How much HCl you use
    - How you use it
      - For example, if you pour HCl into a burette above eye level, it is more likely to get into your eyes than if you fill the burette below eye level
  - The risk of harm can be **reduced** by how we use the acid, for example:
    - Hydrochloric acid could potentially cause harm to your eyes
    - This risk of this, however, depends on how likely it is the acid will get in your eyes
    - We can reduce this risk by using eye protection
    - **Note:** Using eye protection does not affect the **hazard**, but does decrease the **risk** of harm

### Hazard warning symbols


- **Hazard warning symbols** are standardised symbols used on labels and safety data sheets to communicate the hazards associated with a chemical
- There are nine hazard warning symbols, each representing a different type of hazard
- These symbols are usually depicted inside a red diamond with a white background

**Table showing the most common hazard symbols**

Hazard Symbol	Meaning	
---------------	---------	--

	<p>Health hazard</p>	<p>includes warning on skin rashes, eye damage and ingestion</p>
	<p>Corrosive</p>	<p>can cause skin burns and permanent eye damage</p>
	<p>Flammable</p>	<p>can catch fire if heated or comes into contact with a flame</p>
	<p>Acute toxicity</p>	<p>can cause life-threatening effects, even in small quantities</p>
	<p>Hazardous to the environment</p>	<p>substances that are a threat to aquatic life and the environment</p>



	<p>Oxidising</p>	<p>oxidising agents that can cause fire or explosions</p>
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## Risk Assessments

- Risk assessments can be used to help reduce the risk of certain hazards in a laboratory
- To carry out a risk assessment, the following steps are taken:

### Identify the hazards:

- Write a list of all of the chemicals used and identify those substances which have potential hazards associated with them

### Assess the risks:

- Evaluate the likelihood and severity of harm resulting from exposure to the identified hazards
- Factors to consider include:
  - The amount used
  - The age and experience of the person using it
  - Whether it will be heated
  - The type of product given off, e.g. is it a gas that could be inhaled?
- You can categorise risks into low, medium, or high levels

### Implement control measures:

- Develop control measures to reduce the risks to an acceptable level, e.g.:
  - Elimination, e.g. completely remove the hazard
  - Substitution, e.g. replace a hazardous chemical with a less hazardous one
  - Use of good laboratory practices, e.g.
    - Keeping a cap on a bottle after removing some of the substance
    - Keeping the substance away from a source of heat
  - Use safety equipment, e.g. use of fume hoods to prevent the inhalation of toxic gases
  - Use of Personal Protective Equipment (PPE), e.g. use gloves when handling concentrated acids to protect skin from exposure if spilt
  - Plan for an emergency, e.g. what to do if some of the substance is spilt on the floor or gets on the skin
- **Material safety data sheets** (MSDS) are available to help carry out risk assessments when using chemicals and provide information on the safe handling and use of chemicals

### Examiner Tip

When you are planning a practical procedure for internal assessment and you are suggesting a safety precaution you need to say more than just 'wear a lab coat and goggles'.

This is standard practice in any laboratory. You need to identify what the hazard is, and to justify the safety precaution taken e.g.

'HCl gas is produced which is toxic and corrosive, so this step needs to be carried out in a fume cupboard'



Your notes

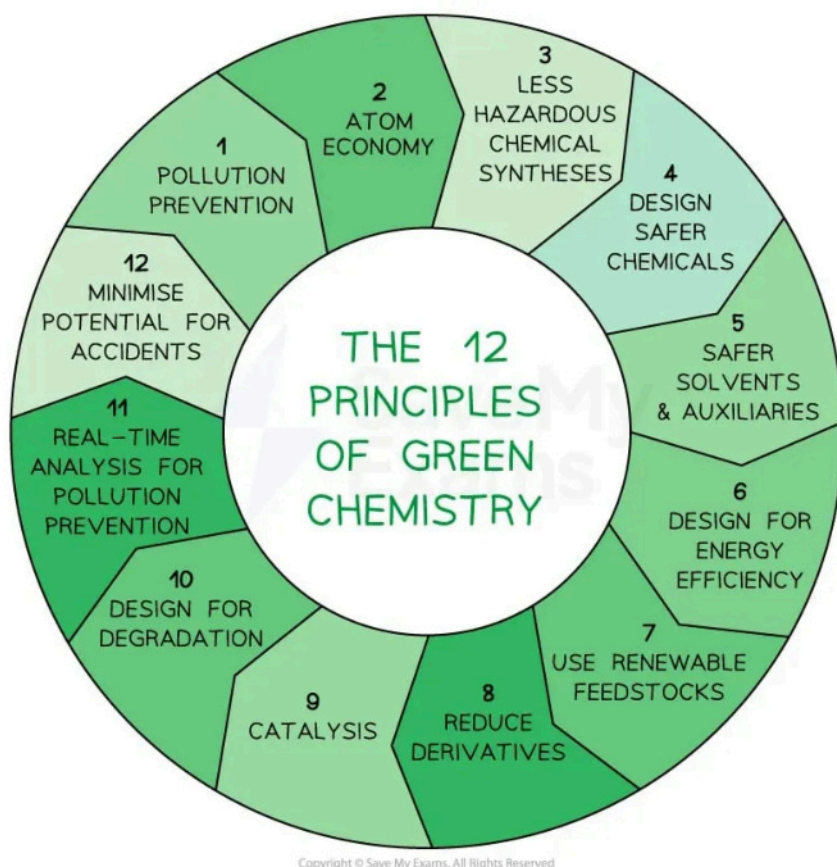
### Proper Handling and Storage of Chemicals:

- You should always look at the **material safety data sheets** (MSDS) when handling or storing chemicals
- Make sure that you **label** any pieces of equipment that you use to handle chemicals
  - For example, when preparing a standard solution for use in titrations, ensure that the volumetric flask used to prepare the solution is properly labelled with the identity and concentration of the solution, together with any necessary hazard symbols

### Environmental Impact Assessment

- You need to consider the potential **environmental** impact of your investigation
- Make sure that any waste is **disposed** of correctly, including any unused reactants or products formed during the course of the investigation
- You should consider whether there are any alternative methods or **Green Chemistry** approaches that are more environmentally friendly and reduce the use of hazardous substances

### The 12 Principles of Green Chemistry



*The 12 Principles of Green Chemistry guide chemists in designing chemical processes that are more sustainable, environmentally friendly, and socially responsible*

- Applications of these **12 Principles of Green Chemistry** are beneficial to the environment and can be incorporated into laboratory work by:
  - **Reducing** the volumes and concentrations of reagents
  - **Minimise waste** by the use of micro-scale laboratory work and the reuse of excess reactants and products
  - **Choose solvents** carefully and use aqueous solutions or simple alcohols/alkanes wherever possible
  - Use a **catalyst** where possible to minimise waste and energy consumption
    - For more information about the environmental impact of using a catalyst, see our revision notes on [Energy Profiles With & Without Catalysts](#)
  - Determine the **atom economy** and look at ways to increase this
    - For more information about atom economy, see our revision notes on [Atom Economy](#)

## Ethical Considerations

- **Ethical** implications are not so obvious in chemistry investigations but still need to be considered

- For example, if an investigation involves human subjects, consent from the participants needs to be obtained
- For investigations involving animal testing, ethical guidelines and protocols for animal experimentation must be followed



Your notes



Your notes

## Measuring Variables in Chemistry

### Measuring Variables in Chemistry

- You need to know how to accurately measure variables to allow the collection of valid and high-quality data
- Sometimes, you will be required to make a decision as to what piece of equipment to use based on which is the most appropriate for that particular task

#### Measuring mass

- Mass is measured using a digital balance which normally gives readings to two decimal places
- Balances must be tared (set to zero) before use
- The standard unit of mass is kilograms (kg) but in chemistry, grams (g) are most often used
  - 1 kilogram = 1000 grams

#### Measuring the volume of liquids

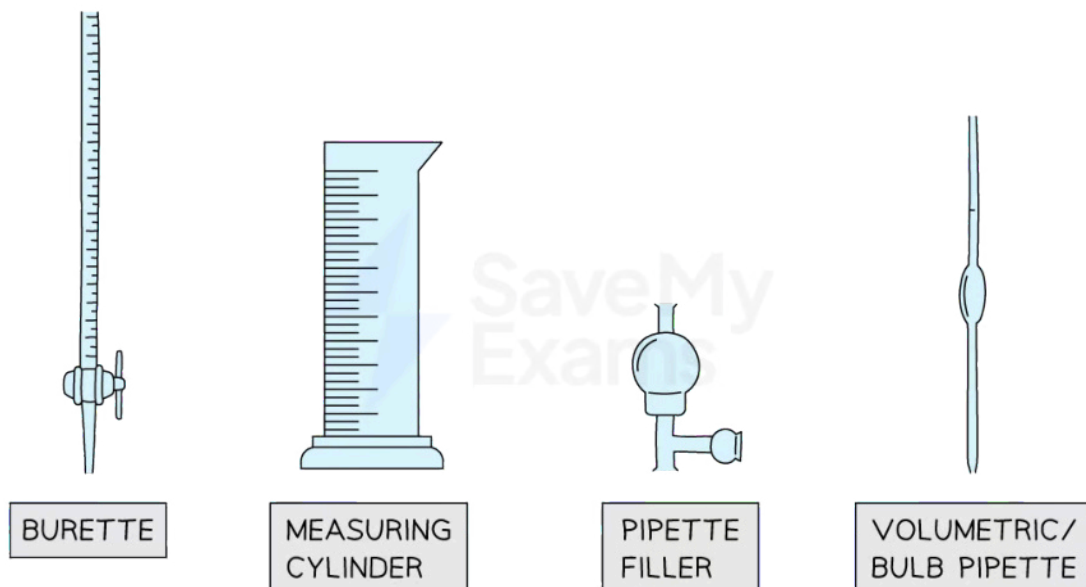
- The volume of a liquid can be determined using several types of apparatus, depending on the level of accuracy needed
- For **approximate** volumes where high accuracy is not an important factor, measuring (or graduated) cylinders are used
  - These are graduated (have a scale so can be used to measure) and are available typically in a range of sizes from 10 cm<sup>3</sup> to 1 litre (1 dm<sup>3</sup>)
- Volumetric pipettes are the most **accurate** way of measuring a **fixed** volume of liquid, usually 10 cm<sup>3</sup> or 25 cm<sup>3</sup>
  - They have a scratch mark on the neck which is matched to the bottom of the **meniscus** to make the measurement
- Burettes are the most **accurate** way of measuring a **variable** volume of liquid between 0 cm<sup>3</sup> and 50 cm<sup>3</sup> (e.g. in a titration)
  - The tricky thing with burettes is to remember to read the scale from top to bottom as 0.00 cm<sup>3</sup> is at the top of the column
- Whichever apparatus you use, you may see markings in ml (millilitre) which is the same as a cm<sup>3</sup>



### Equipment used to measure the volume of liquids



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*Diagram of a burette, a measuring cylinder, a pipette filler and a volumetric pipette*

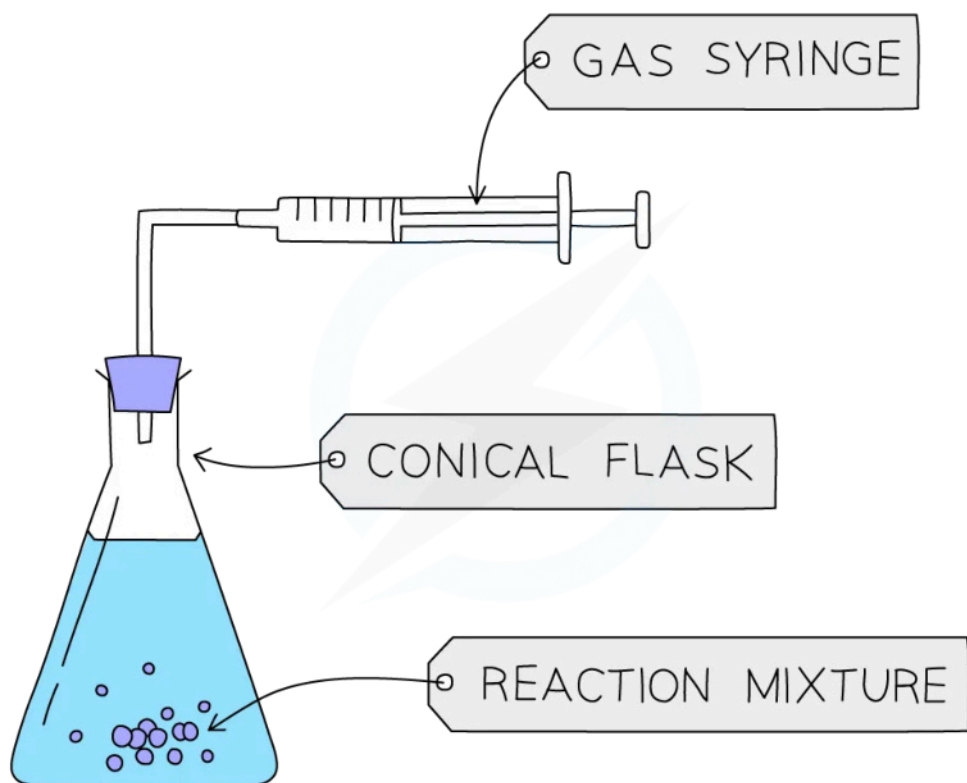
### Measuring the volume of gases

- The volume of a gas sometimes needs to be measured and is done by collecting it in a graduated measuring apparatus
- A **gas syringe** is usually the apparatus used
- A graduated measuring cylinder or burette inverted in water may also be used, provided the gas is not water-soluble
- If the gas happens to be heavier than air and is coloured, the cylinder can be used upright

#### **Measurement of the volume of gas using a gas syringe**



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*Diagram of the set-up for an experiment involving gas collection*

## Measuring time

- Time can be measured using a stopwatch or stop-clock which are usually accurate to one or two decimal places
- The units of time normally used are seconds or minutes although other units may be used for extremely slow reactions (e.g. rusting)
  - 1 minute = 60 seconds
- An important factor when measuring time intervals is human reaction time
  - This can have a significant impact on measurements when the measurements involved are very short (less than a second)

### Examiner Tip

- Be careful when recording time not to mix up seconds and minutes in the same table
  - If a table heading shows Time / mins and you record a stopwatch display of 1.30, meaning 1 minute and 30 seconds, that is wrong as it should be 1.5 mins
- To avoid any confusion, if the time intervals are less than a minute, it is best to change the recorded units to seconds
  - So the 1.30 stopwatch display would therefore be recorded as 90 seconds

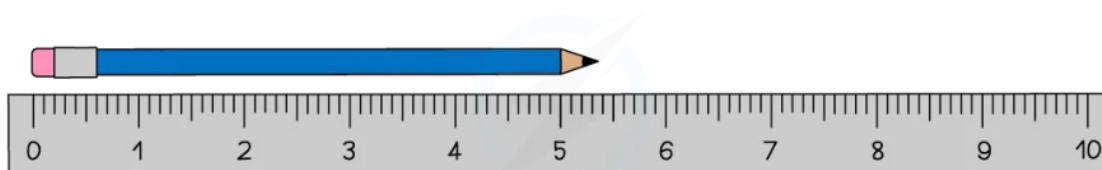
## Measuring temperature

- Temperature is measured with a thermometer or digital probe
- Laboratory thermometers usually have a precision of a half or one degree
- Digital temperature probes are available which are more precise than traditional thermometers and can often read to 0.1 °C
- Traditional thermometers rely upon the uniform expansion and contraction of a liquid substance with temperature; digital temperature probes can be just as, if not, more accurate than traditional thermometers
- The units of temperature are **degrees Celsius (°C)**

## Measuring length

- Rulers can be used to measure small distances of a few centimetres (cm).
  - They are able to measure to the nearest millimetre (mm)
- The standard unit of length is metres (m)
- Larger distances can be measured using a tape measure
- Many distances in chemistry are on a much smaller scale, for example, a typical atomic radius is around  $1 \times 10^{-10}$  m, so cannot be measured in this way

### Measuring length



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*A ruler can measure distances to the nearest mm*

## Measuring the pH of a solution

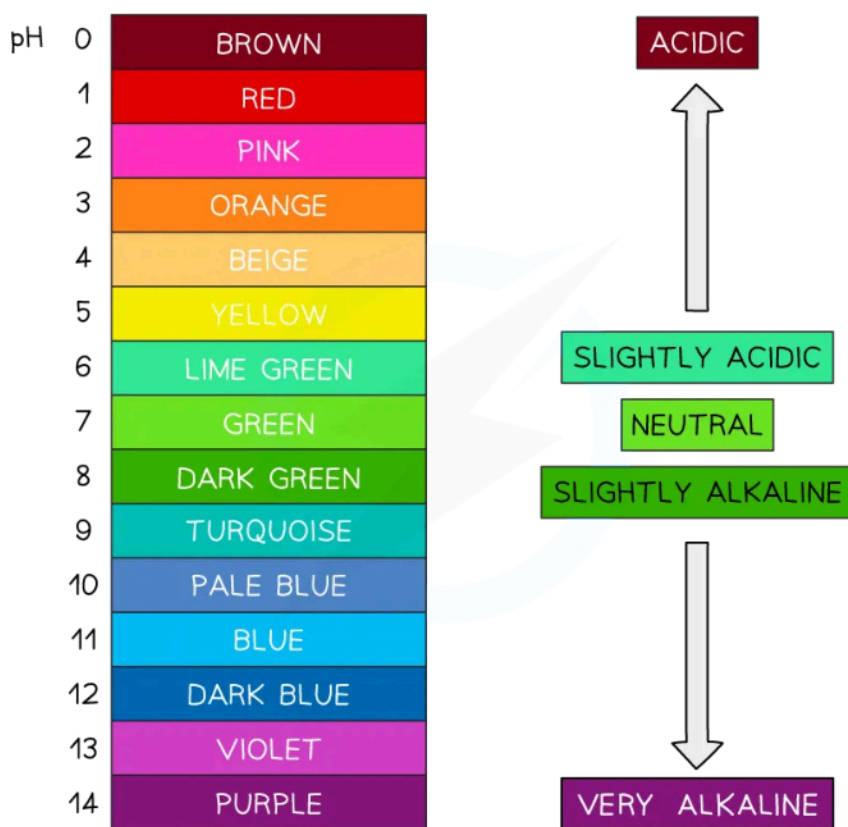
- pH can be measured using an **indicator** or a **digital pH meter**
- pH meters contain a special electrode with a thin glass membrane that allows hydrogen ions to pass through; the ions alter the voltage detected by the electrode



Your notes

- An **indicator** is a substance which changes colour depending on the pH of the solution to which it is added
- There are **natural** indicators and **synthetic** indicators which have different uses
  - Generally, natural indicators are wide range indicators that contain a mixture of different plant extracts and so can operate over a broad range of pH values
  - Synthetic indicators mostly have very narrow pH ranges at which they operate
    - They have sharp colour changes meaning they change colour quickly and abruptly as soon as a pH specific to that indicator is reached
- Indicators are intensely **coloured** and very sensitive so only a few drops are needed
- **Universal indicator** is a wide range indicator and can give only an approximate value for pH
  - It is made of a mixture of different plant indicators which operate across a broad pH range and is useful for estimating the pH of an unknown solution
  - A few drops are added to the solution and the colour is matched with a colour chart which indicates the pH which matches with specific colours
  - Universal indicator colours vary slightly between manufacturer so colour charts are usually provided for a specific indicator formulation

### Colours of universal indicator



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*pH scale with the universal indicator colours used to determine the pH of a solution*



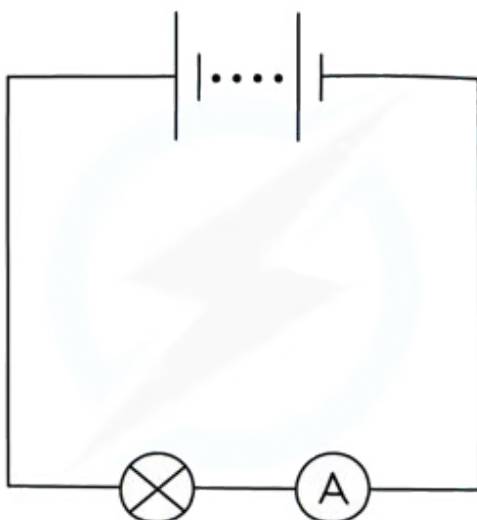
Your notes

**Examiner Tip**

- pH probes offer higher precision and accuracy compared with indicators, so they are more suitable for most applications
- Indicators with a sharp colour change are still a suitable choice for use in titrations as they give a clear endpoint, are simple to use and give valid results
- pH meters may respond more gradually to changes in pH so may not provide a clear, sharp signal at the endpoint

**Measuring electric current**

- Current is measured using an ammeter
- Ammeters should always be connected in series with the part of the circuit you wish to measure the current through



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*An ammeter can be used to measure the current around a circuit*

**Digital or Analogue?**

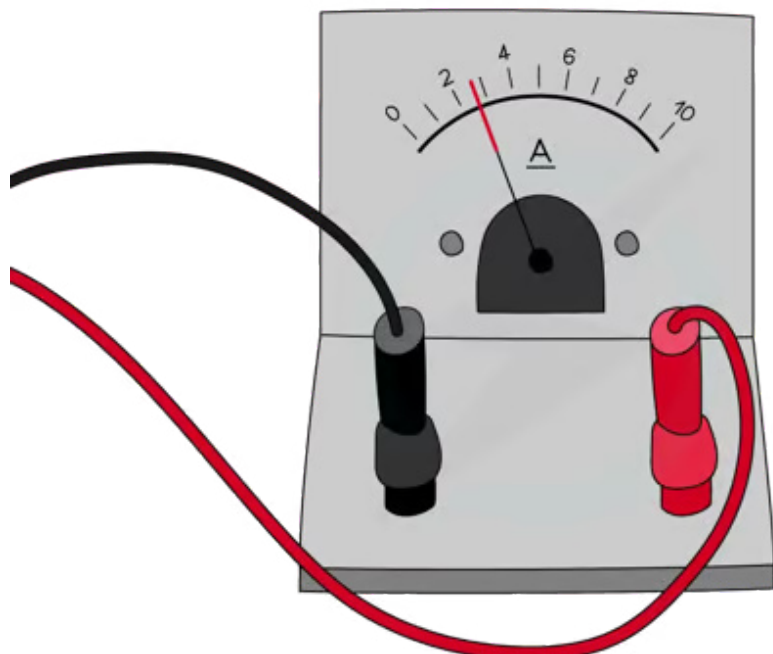
- Ammeters can be either
  - Digital (with an electronic display)
  - Analogue (with a needle and scale)

**Analogue Ammeters**

- Typical ranges are 0.1 - 1.0 A and 1.0 - 5.0 A for analogue ammeters
  - Always double-check exactly where the marker is before an experiment
  - If the marker is not at zero, you will need to subtract this from all your measurements
- They should be checked for zero errors before using

- They are also subject to parallax error
  - Always read the meter from a position directly perpendicular to the scale

### An analogue ammeter



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*Analogue ammeters have a needle and scale for measuring electric current*

### Digital Ammeters

- Digital ammeters can measure very small currents, in mA or  $\mu\text{A}$
- Digital displays show the measured values as digits and are more accurate than analogue displays
- They're easy to use because they give a specific value and are capable of displaying more precise values
- However, digital displays may 'flicker' back and forth between values and a judgement must be made as to which to write down
  - Make sure the reading is zero before starting an experiment, or subtract the "zero" value from the end results
  - Digital ammeters should be checked for [zero errors](#)

### A digital ammeter



Your notes



*Digital ammeters have an electric read-out for measuring electric current*

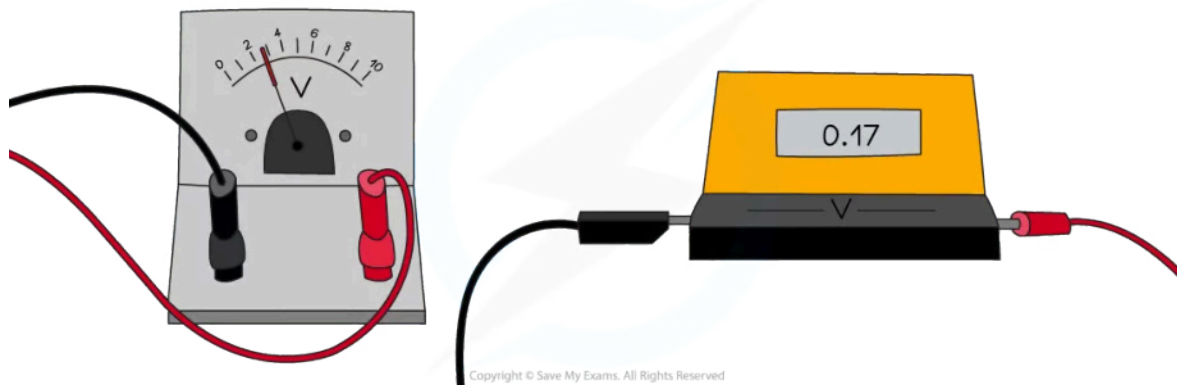
## Measuring the electric potential difference

- Electric potential difference is measured using a voltmeter, which can be either
  - Digital (with an electronic display)
  - Analogue (with a needle and scale)
- Voltmeters are connected in **parallel** with the component being tested
  - The potential difference is the **difference** in electrical potential between two points, therefore the voltmeter has to be connected to **two points** in the circuit

### Analogue or Digital?

- **Analogue voltmeters** are subject to
  - Always read the meter from a position directly perpendicular to the scale parallax errors
- Typical ranges are 0.1–1.0 V and 0–5.0 V for analogue voltmeters although they can vary
  - Always double-check exactly where the marker is before an experiment, if not at zero, you will need to subtract this from all your measurements
  - They should be checked for zero errors before using

### An analogue and digital voltmeter



***Voltmeters can be either analogue (with a scale and needle) or digital (with an electronic read-out) for measuring the electric potential difference***

- **Digital voltmeters** can measure very small potential differences, in mV or  $\mu\text{V}$
- Digital displays show the measured values as digits and are more accurate than analogue displays
- They're easy to use because they give a specific value and are capable of displaying more precise values
  - However, digital displays may 'flicker' back and forth between values and a judgement must be made as to which to write down
- Digital voltmeters should be checked for zero errors
  - Make sure the reading is zero before starting an experiment, or subtract the "zero" value from the end results



Your notes



## Applying Techniques in Chemistry



Your notes

### Applying Techniques in Chemistry

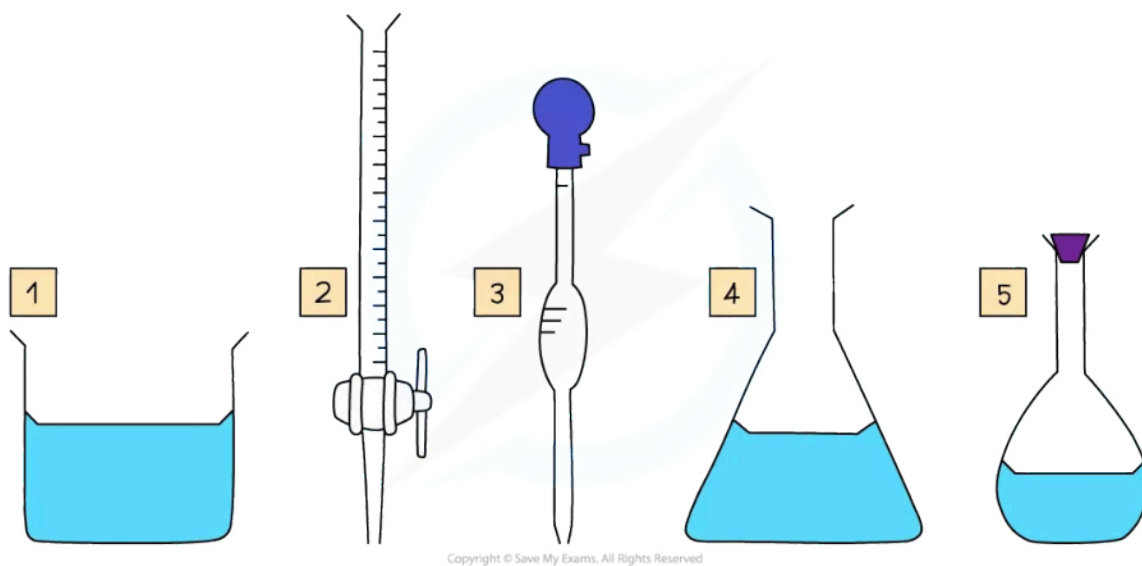
- There are a number of practical experiments and techniques that you need to be aware of, within the following categories:
  - Volumetric analysis techniques
  - Separation techniques
  - Purification techniques
  - Other techniques and experiments

#### Volumetric analysis

- Volumetric analysis techniques including:
    - Preparing a standard solution
    - Carrying out dilutions
    - Performing titrations (acid-base titration and redox titration)
  - Volumetric analysis** is a process that uses the volume and concentration of one chemical reactant (**a standard / 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**
    - 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
- Key pieces of apparatus used to prepare a volumetric solution and perform a simple titration**



Your notes



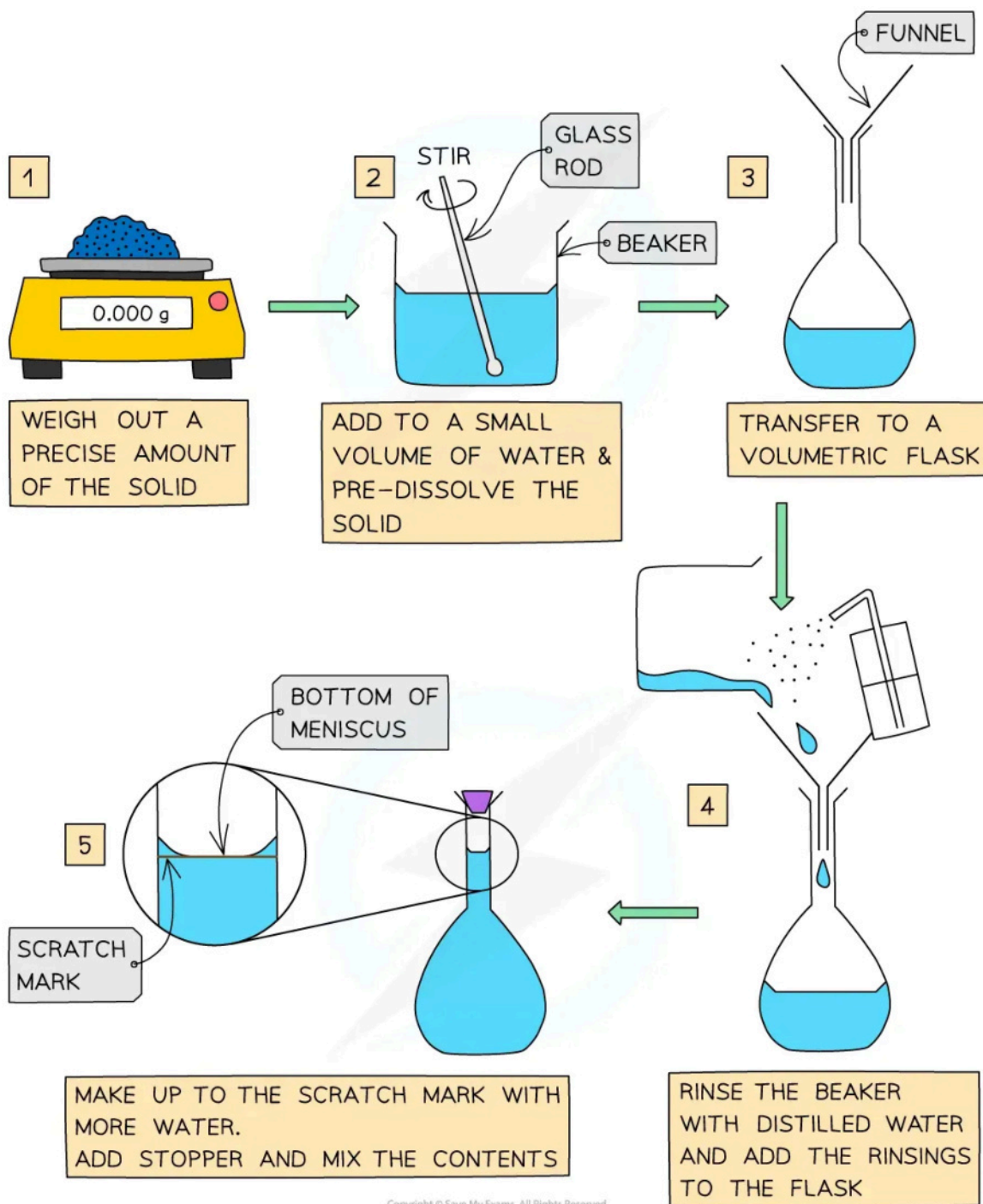
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*Key pieces of apparatus for volumetric analysis include a beaker, burette, volumetric pipette, conical flask and a standard / volumetric flask*

### Making a standard / volumetric solution

- Chemists routinely prepare solutions needed for analysis, whose concentrations are known precisely
  - These solutions are termed **standard solutions** or **volumetric 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

#### How to prepare a standard solution



**A standard solution is made by adding a measured mass of solid to a measured volume of deionised water**



Your notes

### Worked example

Calculate the mass of sodium hydroxide, NaOH, required to prepare 250 cm<sup>3</sup> of a 0.200 mol dm<sup>-3</sup> solution.

#### Answer:

- Find the number of moles of NaOH needed from the concentration and volume:
  - number of moles = concentration (mol dm<sup>-3</sup>) x volume (dm<sup>3</sup>)
  - $n = 0.200 \text{ mol dm}^{-3} \times 0.250 \text{ dm}^3$
  - $n = 0.0500 \text{ mol}$
- Find the molar mass of NaOH:
  - $M_r = 22.99 + 16.00 + 1.01 = 40.00 \text{ g mol}^{-1}$
- Calculate the mass of NaOH required:
  - mass = moles x molar mass
  - mass =  $0.0500 \text{ mol} \times 40.00 \text{ g mol}^{-1} = 2.00 \text{ g}$

### Carrying out dilutions

- The **concentration** of a solution is the amount of **solute** dissolved in a **solvent** to make 1 dm<sup>3</sup> of **solution**
  - The solute is the substance that dissolves in a solvent to form a solution
  - The solvent is often water
- 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
  - mass per unit volume
  - parts per million
- A concentrated solution can be diluted to form a dilute solution
  - For example, diluting 500 cm<sup>3</sup> of a stock 1.0 mol dm<sup>-3</sup> standard solution to a 0.5 mol dm<sup>-3</sup> standard solution
    - Take the 500 cm<sup>3</sup> of the 1.0 mol dm<sup>-3</sup> standard solution
    - Add 500 cm<sup>3</sup> of deionised water
    - There is now 1000 cm<sup>3</sup> of a 0.5 mol dm<sup>-3</sup> standard solution
- Serial dilutions are a sequence of dilutions
  - The initial stock solution is typically diluted by a factor of 10, e.g. 100 cm<sup>3</sup> of the stock solution added to 900 cm<sup>3</sup> of deionised solution
  - This process is then repeated until a solution of the desired concentration is achieved

### Performing titrations

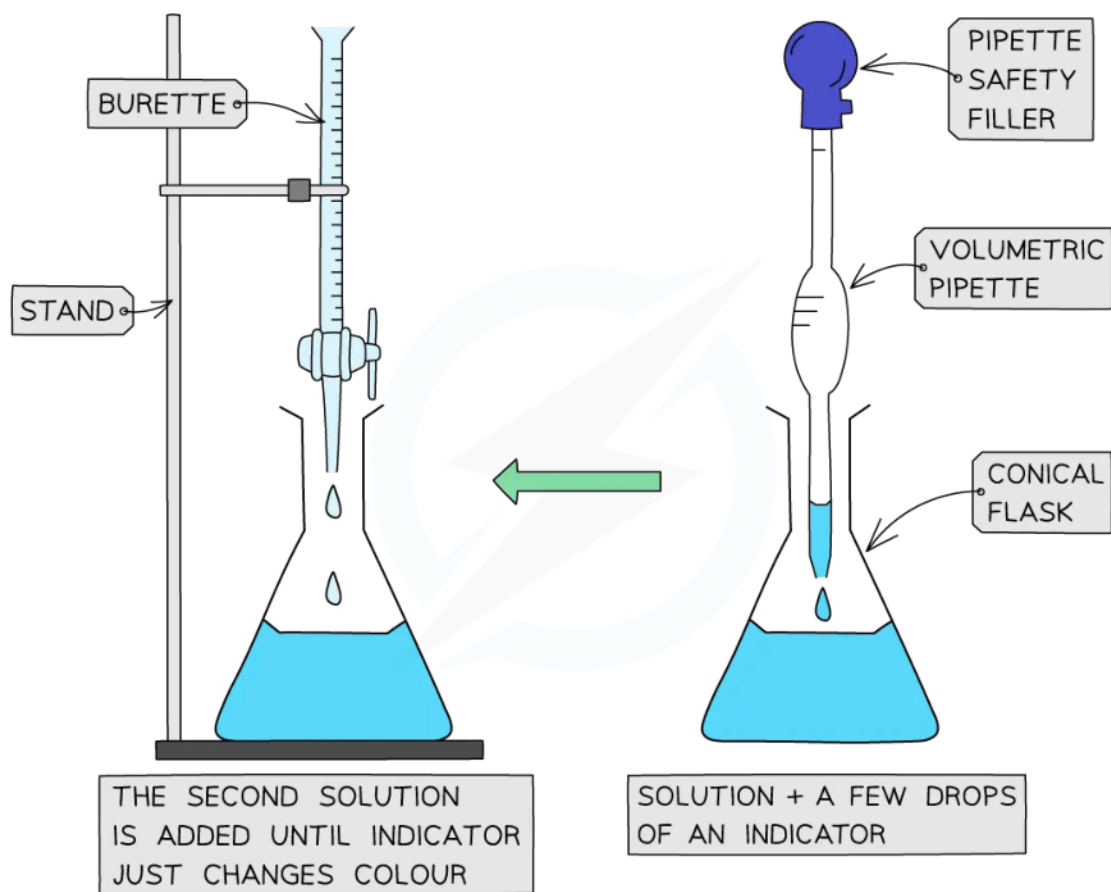
- Titrations include acid-base titrations and redox titrations
  - Acid-base titrations involve an acid and a base



Your notes

- Redox titrations are more specific reactions involving the reduction and oxidation occurring simultaneously, e.g. the  $\text{Fe}^{2+} / \text{MnO}_4^-$  titration
- The key piece of equipment used in the titration is the burette
- Burettes** are usually marked to a precision of  $0.10 \text{ cm}^3$ 
  - 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 **endpoint** or **equivalence point** occurs when the two solutions have reacted completely
  - In the case of most acid-base titrations, this can be observed with the use of an **indicator**
  - For more information about choosing indicators, see our revision note on [Choosing an Acid-Base Indicator](#)
  - There are some examples of redox titrations where no indicator is required, e.g. the  $\text{Fe}^{2+} / \text{MnO}_4^-$  redox titration

### Using an indicator in titrations



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*Only a few drops of indicator are added, if necessary, because they are typically weak acids and can influence the results*

- The steps in a titration are:

- Measuring a known volume (usually 20.0 or 25.0 cm<sup>3</sup>) 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 cm<sup>3</sup>
- If necessary, a few drops of **indicator** are added to the solution in the conical flask
  - A **white tile** is sometimes placed under the conical flask while the titration is performed, to make it easier to see the colour change
- The tap on the burette is carefully opened and the solution is added, portion by portion, to the conical flask until the indicator starts to change colour
  - After each portion, the conical flask should be swirled
- As you start getting **near to the endpoint**, 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 a **permanent colour change**
- Multiple runs are carried out until concordant results are obtained

### Recording and processing titration results

- Both the **initial** and **final burette readings** should be recorded and shown to a precision of  $\pm 0.05 \text{ cm}^3$ , the same as the uncertainty

#### A typical layout and set of titration results



Your notes

	Rough	Run 1	Run 2	Run 3
Initial burette reading $\pm 0.05$ ml	0.00	23.15	0.20	23.00
Final burette reading $\pm 0.05$ ml	23.75	45.95	23.00	46.10
Volume delivered $\pm 0.10$ 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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- The volume delivered (titre) is calculated and recorded to an uncertainty of  $\pm 0.10 \text{ cm}^3$ 
  - For more information about working with uncertainties, see our revision note on [Processing Uncertainties in Chemistry](#)
- Concordant results are then averaged, and non-concordant results are discarded
  - For more information about calculating average titres, see "What is the mean average?" in our [Applying General Mathematics in Chemistry](#) revision note
- Appropriate titration calculations are then performed, as shown in our revision note on [Concentration Calculations](#)

## Separation of mixtures

- The required separation techniques covered in our revision note on [Separating Mixtures](#) include:
  - Filtration
  - Simple and fractional distillation
  - Paper chromatography
    - The process of thin layer chromatography is the same as paper chromatography
    - The stationary phase is changed from chromatography paper to a sheet with a fine layer of silica or alumina
    - The mobile phase can still be any liquid solvent
    - Separation is still based on solubility

- It can be common to use UV light or locating agents, such as ninhydrin, to identify the spots
- Crystallisation



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## Purification techniques

- The specific purification techniques explicitly stated in the syllabus are:
  - Recrystallisation
  - Melting point determination

### Recrystallisation

- Recrystallisation involves dissolving an impure solid in a suitable solvent and then allowing the compound to crystallise out of the solution
  - The recrystallisation product should have a higher purity
- This process relies on the differences in solubility between the desired compound and the impurities present in the original solid
- For more information about recrystallisation, see our revision note on [Separating Mixtures](#)

### Melting point determination

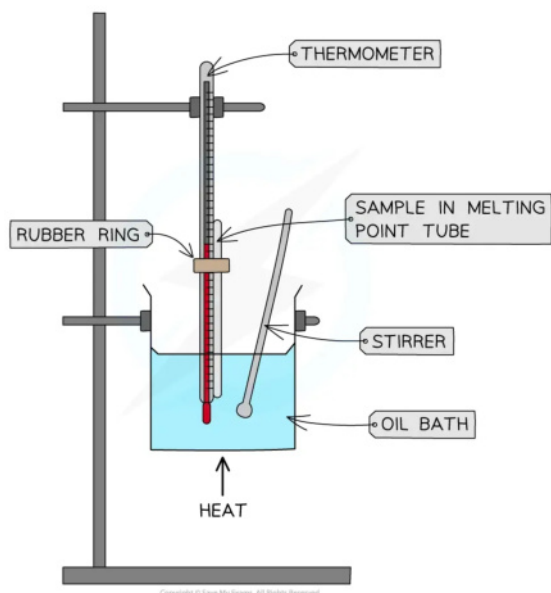
- The melting point of a solid is indicative of its purity and identity
- A melting point can be matched to a known substance as a means of identification or confirmation of a desired product
- The proximity of a melting point to the actual data book value can express purity
  - Impurities tend to lower 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 and when it completely melts
- The skills needed in performing a melting point test are largely dependent on the specific melting point apparatus you are using:

#### Different apparatus used to determine the melting point of a sample

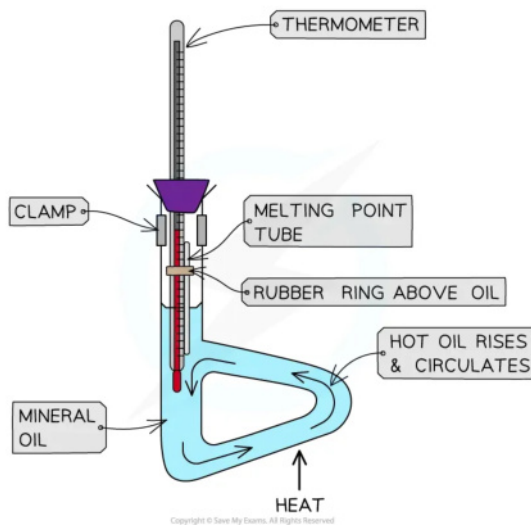




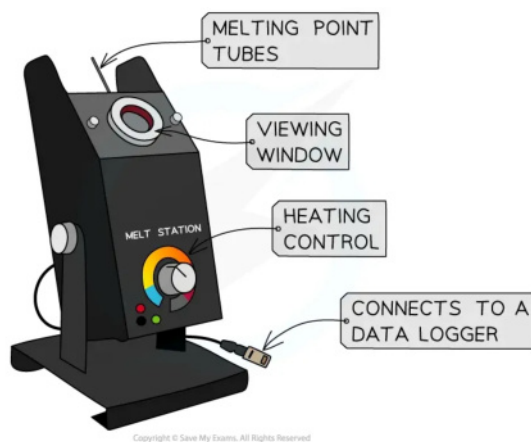
Your notes



**Oil bath method**



**Thiele tube method**



**Melt station method**

- 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)



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- 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

## Other experiments and techniques

- Other specific experiments and techniques explicitly stated in the syllabus are:
  - Calorimetry
    - For more information about calorimetry, see our revision note on [Calorimetry](#)
  - Electrochemical cells
    - For more information about experiments involving electrochemical cells, see the relevant revision notes in our [Electron Transfer Reactions](#) topic
  - Drying to constant mass
  - Reflux
  - Colorimetry / spectrophotometry
  - Physical and digital molecular modelling

## Drying to constant mass

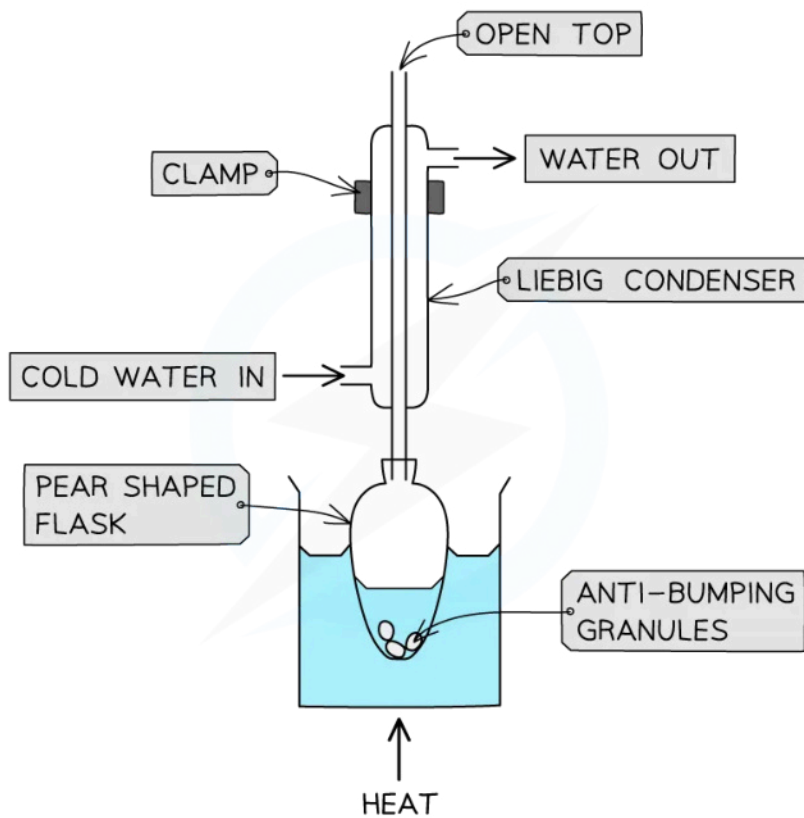
- This is used to determine the amount of water (or volatile components) in a substance
  - The initial mass of the substance is recorded, using a balance
  - The substance is placed in an oven / drying chamber and heated at a specific temperature
  - At regular intervals, the substance is taken out of the oven, allowed to cool and reweighed
  - These steps are repeated until the recorded mass of the substance remains constant
- This is a common technique associated with water of crystallisation in hydrated transition metal compounds

## Heating under reflux

- Organic reactions often occur slowly at room temperature
- Therefore, organic reactions can be completed by heating under reflux to produce an **organic liquid**
- This allows the mixture to react as fully as possible without the loss of any reactants, products or solvent
  - In **distillation**, you are trying to separate a chemical or product from a mixture
  - When **heating under reflux**, you aim to keep all the chemicals inside the reaction vessel
- Example reactions where heating under reflux could be used include:
  - The production of a carboxylic acid from a primary alcohol using acidified potassium dichromate
  - The production of an ester from an alcohol and acid in the presence of an acid catalyst
- The reaction mixture is placed into a pear-shaped or round-bottomed flask
- Anti-bumping granules are added to promote smooth boiling
- The flask is placed in a heating mantle or it can be immersed in a water bath for heating
- Quickfit apparatus is then set up with the condenser clamped vertically in place
  - The joints of the Quickfit apparatus are commonly greased as with distillation
- A steady and constant stream of water passes through the condenser in a 'water jacket' - it enters at the bottom of the condenser and the drainage pipe removes the water from the top of the condenser
- The flask is indirectly heated and the reaction mixture is allowed to boil

- Finally, heating stops and the mixture is allowed to cool back to room temperature

### Heating under reflux practical equipment



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*The preparation of ethyl ethanoate involves heating under reflux for about 15 minutes*

### Colorimetry / spectrophotometry

- Colorimetry and spectrophotometry are techniques used to measure the concentration of substances in a solution based on the absorbance / transmittance of light at specific wavelengths
- Both techniques use the same basic method:
  - A light source emits a beam of light covering a wide range of wavelengths
  - The sample solution absorbs certain wavelengths of light, depending on its composition and concentration
  - The absorbance and transmittance of various wavelengths are then recorded
- The detector on a colorimeter measures the intensity of light which is directly related to the concentration of the solution
  - It is a relatively quick process although not as precise as spectrophotometry, especially with low concentrations or complex mixtures
- The detector on a spectrophotometer measures the absorbance of each wavelength of light



Your notes

- The resulting absorption spectrum is plotted, showing the characteristic absorption peaks of the sample
- The concentration is then determined by comparing this spectrum to a calibration curve
- Spectrophotometry is highly sensitive and accurate, making it suitable for analysing low concentrations and complex mixtures
- It is widely used in research, quality control, drug analysis, environmental monitoring and food testing
- For more information about calorimetry, see our revision note on [Measuring Rates of Reaction](#)



Your notes

### Physical and digital molecular modelling

- **Physical molecular modelling** is the creation of three-dimensional models using materials such as plastic balls and sticks (molymods)
  - It serves as a tool to understand molecular geometry, bond angles and the overall spatial arrangement of atoms within a molecule
- **Digital molecular modelling** uses specialist computer software to generate accurate and detailed 3D models of molecules
  - By giving specific data, such as bond lengths and angles, the software can produce highly accurate representations of molecules, including their electronic structures
  - It allows the study of more complex molecules, especially ones that are challenging to construct
  - It allows observations of molecular movements and reactions in real time
  - Digital molecular modelling provides access to various tools and simulations that can predict:
    - Molecular properties
    - Behaviour in different environments
    - Potential interactions with other molecules
    - These simulations aid researchers in drug design, material science and many other applications