Edexcel International AS Chemistry

Your notes

Inorganic & Organic Chemistry Core Practicals

Contents

Your notes

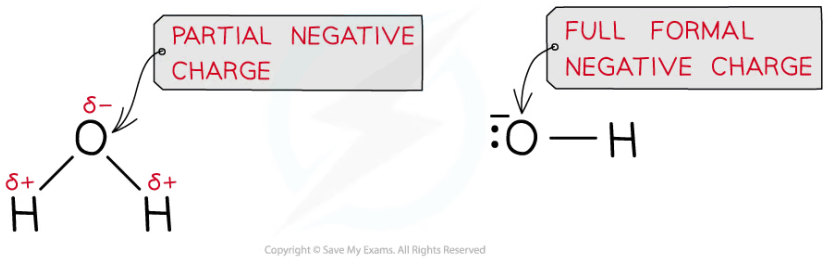
Core Practical 5: Investigating Halogenoalkane Hydrolysis

- Halogenoalkanes can be **hydrolysed** with an **aqueous** solution of sodium hydroxide (NaOH) or potassium hydroxide (KOH)
- This reaction is very slow at room temperature, so the reaction mixture is warmed
- The rate of this reaction depends on the type of halogen in the halogenoalkane
  - The stronger the C-X bond, the slower the rate of the reaction
  - In terms of bond enthalpy, C-F > C-Cl > C-Br > C-I
  - Fluoroalkanes do not react at all, but iodoalkanes have a very fast rate of reaction

$$\underset{\text{BROMOETHANE}}{CH_3CH_2Br} + OH^- \longrightarrow \underset{\text{ETHANOL}}{CH_3CH_2OH} + Br^-$$

The halogen is replaced by the nucleophile, OH^-

- This reaction could also be done with water as the nucleophile, but it is very slow
  - The hydroxide ion is a better nucleophile than water as it carries a full negative charge
  - In water, the oxygen atom only carries a partial charge



A hydroxide ion is a better nucleophile as it has a full formal negative charge whereas the oxygen atom in water only carries a partial negative charge; this causes the nucleophilic substitution reaction with water to be much slower than the aqueous alkali

Measuring the rate of hydrolysis

- Acidified silver nitrate can be used to measure the rate of hydrolysis of halogenoalkanes
- Set up three test tubes in a 50 °C water bath, with a mixture of ethanol and acidified silver nitrate

- Add a few drops of a chloroalkane, bromoalkane and an iodoalkane to each test tube and start a stop watch
- Time how long it takes for the precipitates to form
- The precipitate will form as the reaction progresses and the halide ions are formed
- A white precipitate will form from the chloroalkane, a cream precipitate will form from the bromoalkane and a yellow precipitate will form from the iodoalkane
  - The yellow precipitate will form the fastest
  - This is because the C-I bond has the lowest bond enthalpy, so it is the easiest to break and will cause the I^- ions to form the fastest
  - The white precipitate will form the slowest
  - This is because the C-Cl bond has the highest bond enthalpy, so it is the hardest to break and will cause the Cl^- ions to form the slowest

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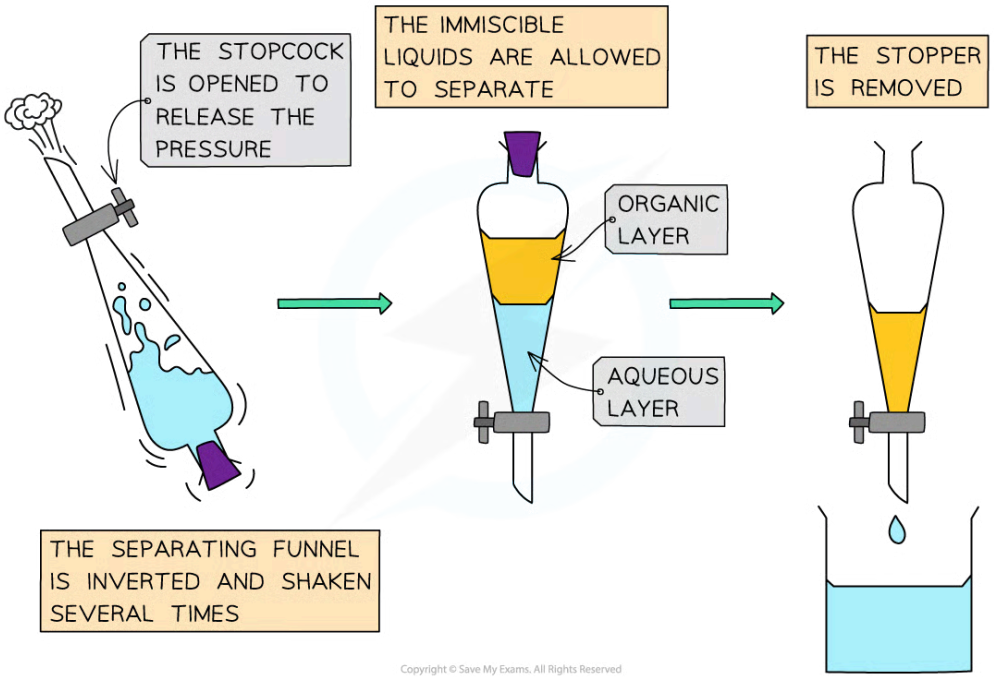
Core Practical 6: Chlorination of 2-Methylpropan-2-ol

Synthesis of a haloalkane from an alcohol

- Measure 8 cm^3 of 2-methylpropan-2-ol in a measuring cylinder
- Pour the 2-methylpropan-2-ol into a separating funnel
- Using a fume hood, add 20 cm^3 of concentrated hydrochloric acid to the separating funnel in small portions of 2 - 3 cm^3 making sure to release the pressure by opening the stopper after each addition
- Leave the separating funnel to stand in the fume hood for 20 minutes and gently shake the separating funnel at 2 minute intervals
- Allow the layers to separate and dispose of the lower aqueous layer by opening the tap
- Add sodium hydrogen carbonate solution in small portions to the funnel and gently shake the funnel, again, release the pressure at regular intervals
  - This removes acidic impurities
- Once these layers have separated, open the tap once more and dispose of the queues layer
- Pour the organic layer into a clean dry conical flask and add two spatulas of magnesium sulfate
  - This will remove water
- Once clear, decant the liquid into distillation apparatus
- Distill the liquid and collect the distillate in the range of 47 - 53°C



A separating funnel allows the product to be cleaned and isolated

Examiner Tips and Tricks

This practical requires a fume hood as concentrated hydrochloric acid is very volatile at room temperature and hydrogen chloride gas is released from the solution quickly when the cover to a container or bottle is removed.

When using a fume hood you must make sure the window is not open above the safety line, otherwise, the exhaust system will not be strong enough to draw in the fumes. Therefore toxic, poisonous, harmful or irritant fumes will escape into the laboratory

If heating is involved in the experiment taking place in the fume hood, having the window open above the safety line will mean that carbon monoxide or soot could also escape.

Your notes

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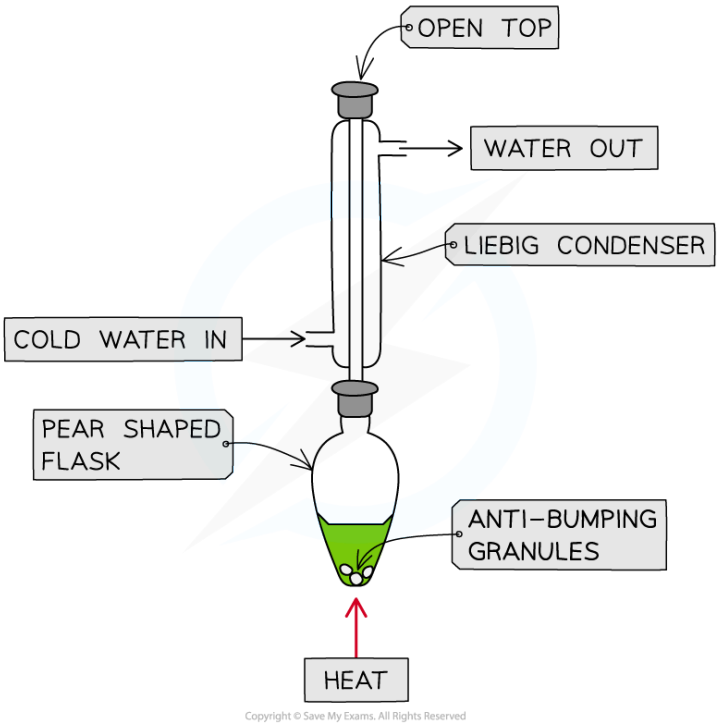
Core Practical 7: Oxidation of Propan-1-ol

Oxidation of propan-1-ol

- Primary alcohols can be oxidised to form **aldehydes** which can undergo further oxidation to form **carboxylic acids**
  - When propan-1-ol is oxidised, propanal is produced and when oxidised further propanoic acid will be formed

Synthesis and purification of propanal and propanoic acid

- Carefully add 20 cm^3 of acidified potassium dichromate(VI) solution, $K_2Cr_2O_7$ (aq), to a 50 cm^3 pear-shaped flask and cool the flask in an iced water bath
- Set up the reflux apparatus keeping pear shaped flask cool
- Place **anti-bumping granules** into the pear shaped flask
- Measure out 1 cm^3 of propan-1-ol
- Using a pipette, slowly add the propan-1-ol drop wise into the reflux condenser
- When the propan-1-ol has been added remove the ice bath and allow to warm up to room temperature
- Position the flask over an electric heater or in a water bath and heat for 20 minutes
  - Propan-1-ol is flammable, therefore. naked flames should not be used when heating which is why the use of an electric heater or water bath is an important safety precaution
- Purify the product using distillation apparatus



Reflux apparatus for the oxidation of propan-1-ol to propanoic acid

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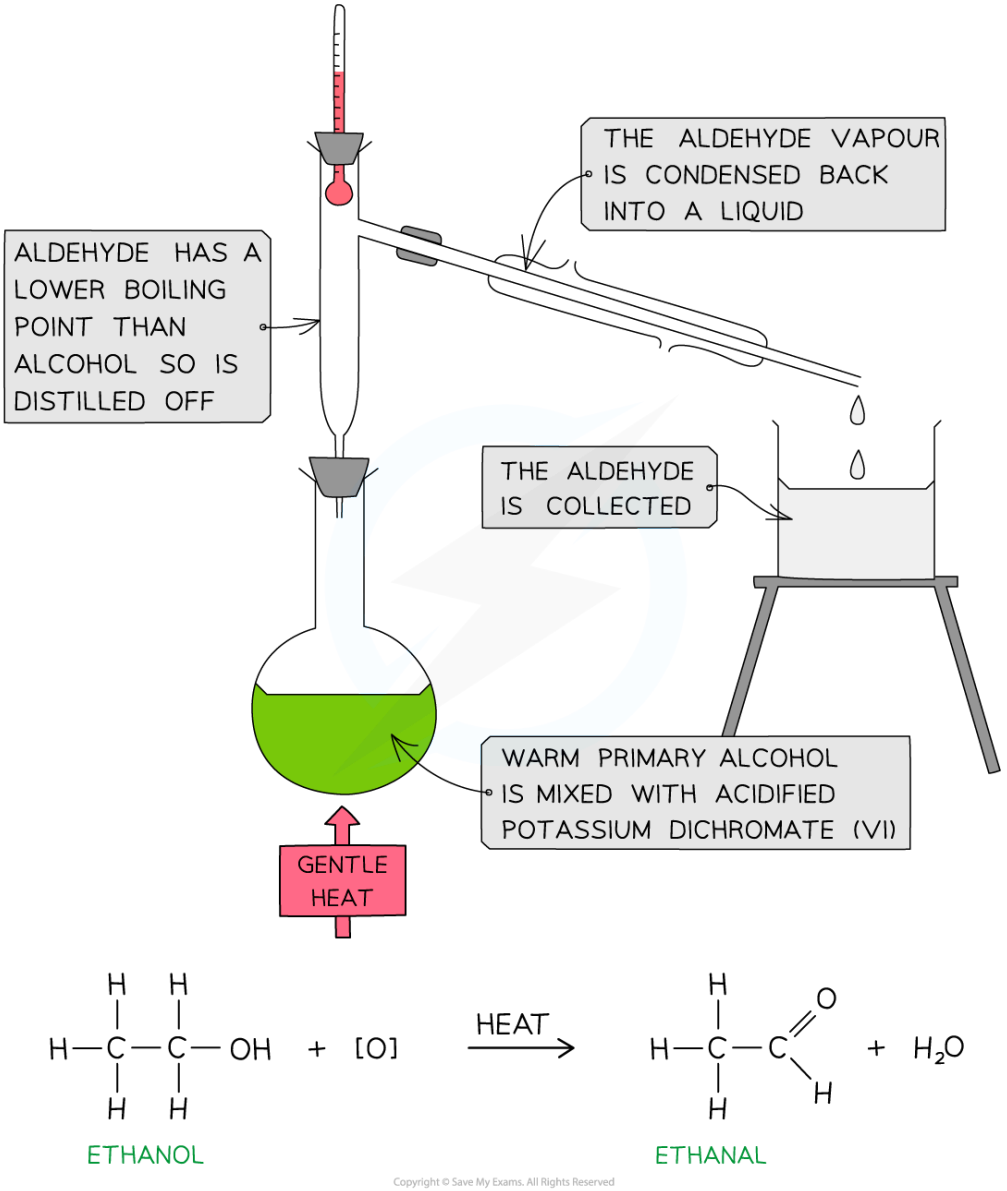


Oxidation of propan-1-ol by acidified $K_2Cr_2O_7$ to form propanal by distillation

Hazards, risks and precautions

FLAMMABLE

HARMFUL TO HEALTH

OXIDISING

Your notes

- The alcohols used are flammable and often harmful to health, e.g, propan-1-ol, butan-1-ol, pentan-1-ol
- The alcohols should be kept away from naked flames, e.g. a Bunsen burner
- Avoid contact with the skin and breathing in the vapour
- A fume cupboard can be used for harmful alcohols
- Potassium dichromate is a strong oxidising agent and should be handled with care
- Spillages should be mopped up right away with plenty of water

Your notes

Core Practical 8: Analysis of Inorganic and Organic Unknowns

Test Tube Reactions

- Simple test tube reactions can be done to identify the following ions:
  - Ammonium ions (NH_4^+)
  - Halide ions (X^-) - covered in 2.3.4 Halide Ion Reactions
  - Carbonate ions (CO_3^{2-}) and hydrogencarbonate ions (HCO_3^-)
  - Sulfate ions (SO_4^{2-})
- If the sample to be tested is a solid, then it must be dissolved in deionised water and made into an aqueous solution

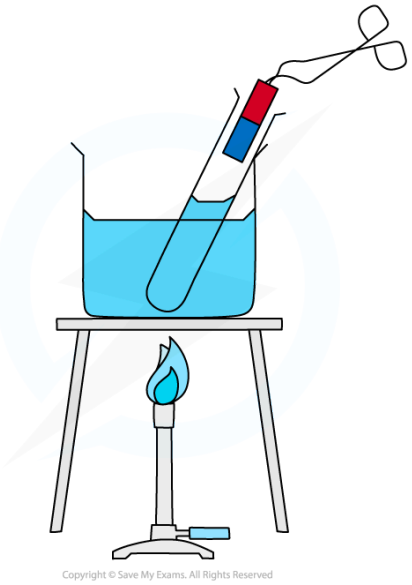
Testing for Ammonium Ions

- About 10 drops of a solution containing ammonium ions, such as ammonium chloride, should be added to a clean test tube
- About 10 drops of sodium hydroxide should be added using a pipette

$$\text{Overall equation: } NH_4Cl\,(aq) + NaOH\,(aq) \rightarrow NH_3\,(g) + H_2O\,(l) + NaCl\,(aq)$$

$$\text{Ionic equation: } NH_4^+\,(aq) + OH^-\,(aq) \rightarrow NH_3\,(g) + H_2O\,(l)$$

- The test tube should be swirled carefully to ensure that it is mixed well
- The test tube of the solution should then be placed in a beaker of water, and the beaker of water should be placed above a Bunsen burner, so that it can become a water bath
- As the solution is heated gently, fumes will be produced
- A pair of tongs should be used to hold a damp piece of red litmus paper near the mouth of the test tube, to test the fumes
- The red litmus paper will change colour and become blue in the presence of ammonia gas



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

Testing for Carbonate Ions

- A small amount (around 1 cm^3) of dilute hydrochloric acid should be added to a test tube using a pipette
- An equal amount of sodium carbonate solution should then be added to the test tube using a clean pipette

$$\text{Overall equation: } 2HCl\,(aq) + Na_2CO_3\,(aq) \rightarrow 2NaCl\,(aq) + CO_2\,(g) + H_2O\,(l)$$

$$\text{Ionic equation: } 2H^+\,(aq) + CO_3^{2-}\,(aq) \rightarrow CO_2\,(g) + H_2O\,(l)$$

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

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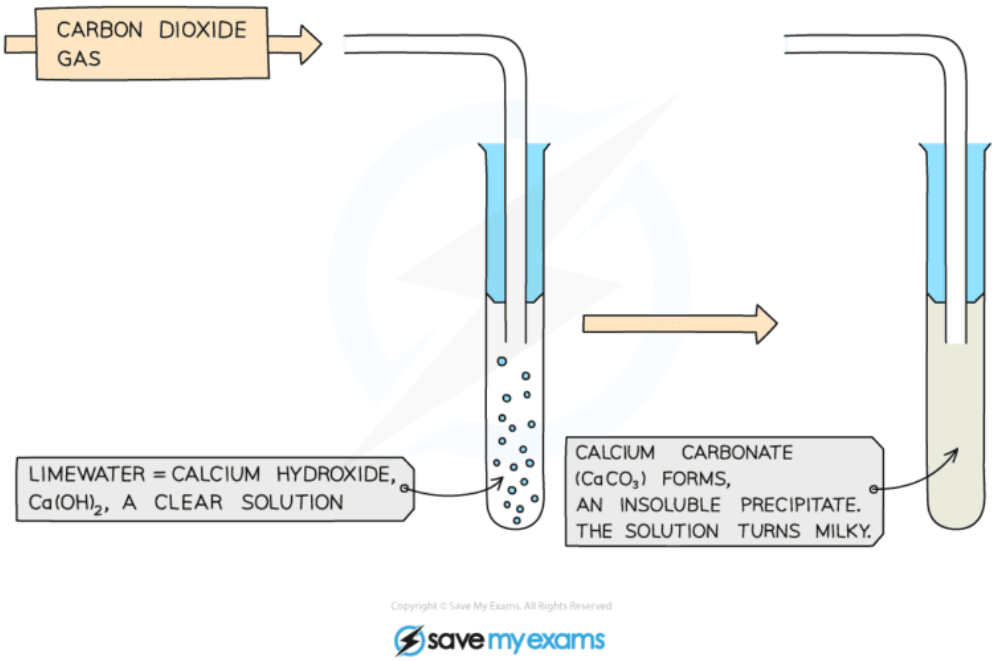

When carbon dioxide gas is bubbled into limewater it will turn cloudy as calcium carbonate is produced

- A similar reaction is seen with sodium hydrogen carbonate, but the equations are:

Overall equation: $HCl\,(aq) + NaHCO_3\,(aq) \rightarrow NaCl\,(aq) + CO_2\,(g) + H_2O\,(l)$

Ionic equation: $H^+\,(aq) + HCO_3^-\,(aq) \rightarrow CO_2\,(g) + H_2O\,(l)$

Testing for Sulfate Ions

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

$$\mathbf{Ba^{2+}\,(aq) + SO_4^{2-}\,(aq) \rightarrow BaSO_4\,(s)}$$

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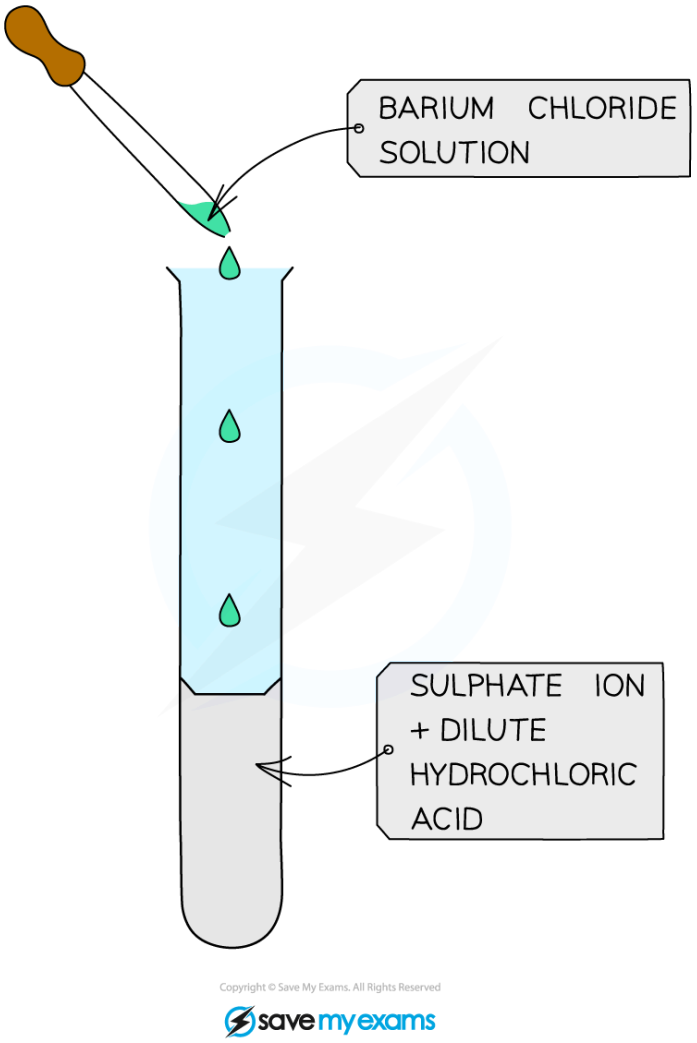

A white precipitate of barium sulfate is a positive result for the presence of sulfate ions

Examiner Tips and Tricks

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

Flame tests

- Metal ions produce a **colour** if heated strongly in a flame
- Ions from **different** metals produce **different colours**
- The flame test is thus used to identify metal ions by the colour of the flame they produce
- Dip the loop of an **unreactive** metal wire such as nichrome or platinum in concentrated acid, and then hold it in the blue flame of a Bunsen burner until there is no colour change

- This cleans the wire loop and avoids **contamination**
  - This is an important step as the test will only work if there is just **one type** of ion present
  - Two or more ions means the colours will mix, making identification erroneous
- Dip the loop into the solid sample and place it in the edge of the **blue** Bunsen flame
- Avoid letting the wire get so hot that it glows red otherwise this can be confused with a flame colour

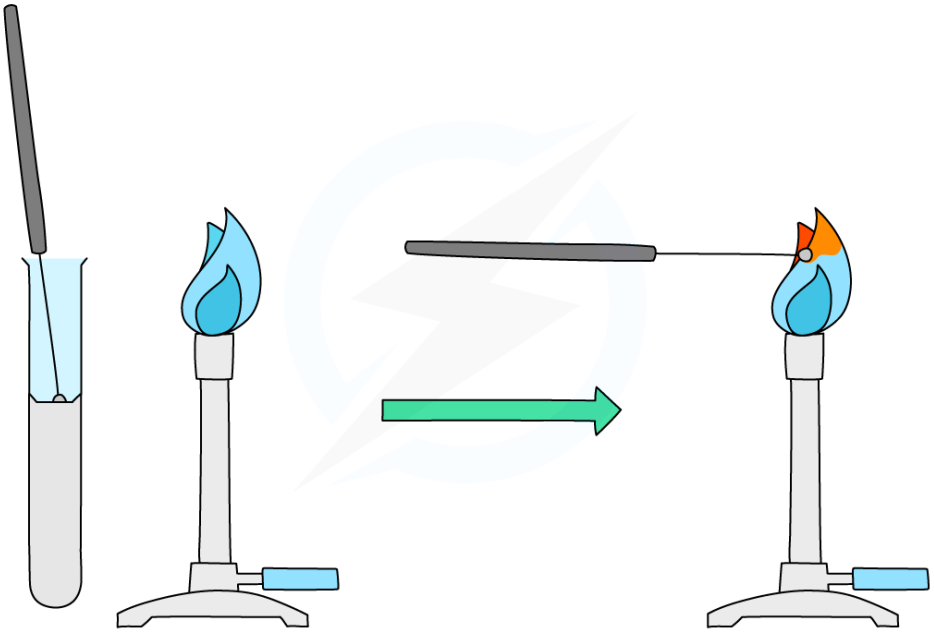

Diagram showing the technique for carrying out a flame test

Colours Observed in Flame Tests

Your notes

Metal ion	Colour observed
Li^+	Scarlet red
Na^+	Yellow
K^+	Lilac
Rb^+	Red
Cs^+	Blue
Mg^{2+}	No flame colour
Ca^{2+}	Brick red
Sr^{2+}	Red
Ba^{2+}	Apple green

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