Purity and chromatography

Learning objectives

- ❑ Explain what is meant by the purity of a substance
- ❑ Explain that useful materials are formulations of mixtures
- ❑ Using melting point data to distinguish pure from impure substances
- ❑ Describe and explain the process of filtration, crystallisation, simple distillation and fractional distillation
- ❑ Understand and interpret paper chromatography
- ❑ Describe the technique of thin-layer chromatography
- ❑ Identify substances by calculation and use of R_f values

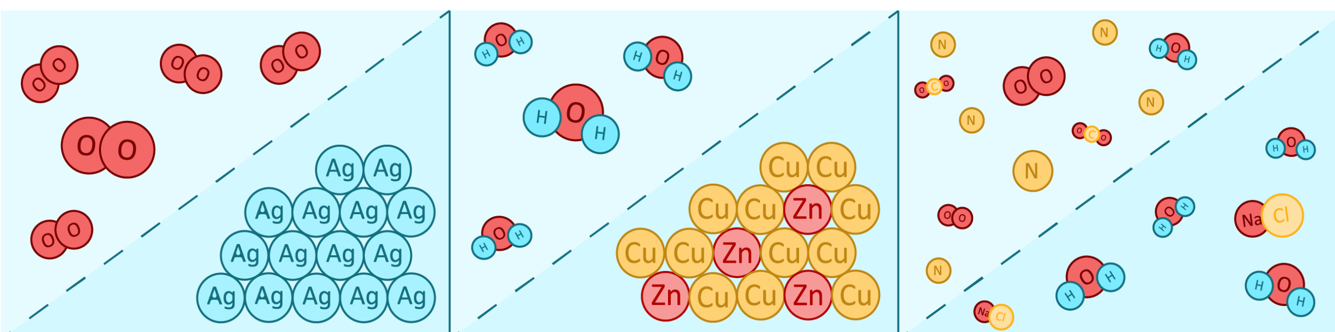
Pure substances

The meaning of pure

In everyday language pure means 'not mixed'. For example, a jumper might be **pure cotton**, or you have a glass of pure milk, rather than chocolate milk. However, in **chemistry, pure** means that a substance just made up of **one element or compound**. In chemical terms that cotton jumper and glass of milk are not pure. However, **pure gold** would be **chemically pure**, as it **only** contains the **element gold**.

> **Good to know**
> A **substance** commonly refers to an element or compound; we tend to say that a mixture is made up of substances.

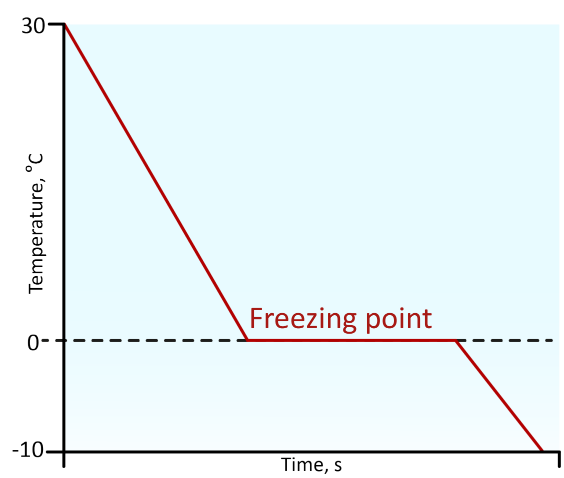
Useful terms:



An **element** is just one type of atom, for example; silver, Ag and Oxygen, O2	A **compound** is when two or more elements are bonded together; for example, water, H_2O or brass.	A **mixture** is made up of two or more substances that are not bonded together; like air, or saltwater.

Differentiating between pure substances and mixtures

To differentiate between a **pure substance** and a **mixture**, we need to look at freezing points. The **freezing point** of a pure substance is at a **specific temperature.** The cooling curve to the right shows the **freezing** of pure water. It happens at **precisely 0°C**. There is a **flat part** to the graph – this is where the pure liquid water freezes into pure ice.



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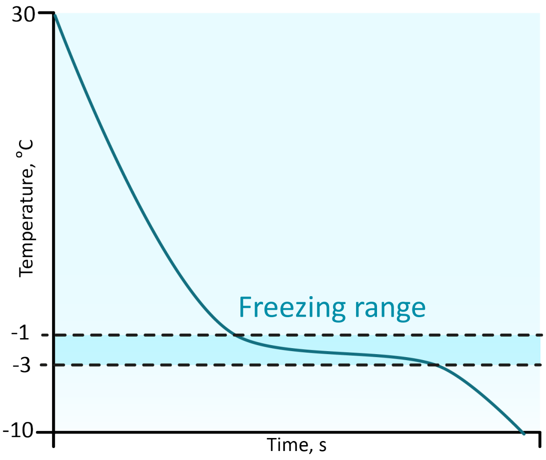
However, water in nature is **not pure**; it is a **mixture**. It has minerals in it, such as salt, magnesium and calcium. If we look at the **cooling curve for seawater**, it is **different**. Instead of freezing at a specific temperature, it freezes over a **range: between -1°C and -3°C.** The freezing part of the **curve is now sloped**.



Example

A student tests the **melting point** of a **sample of sodium.** It starts melting at **76-81°C**, but is not fully melted until the temperature is **83°C**. According to a **data book**, the melting point of sodium is **85°C**. Is this sample pure or impure?

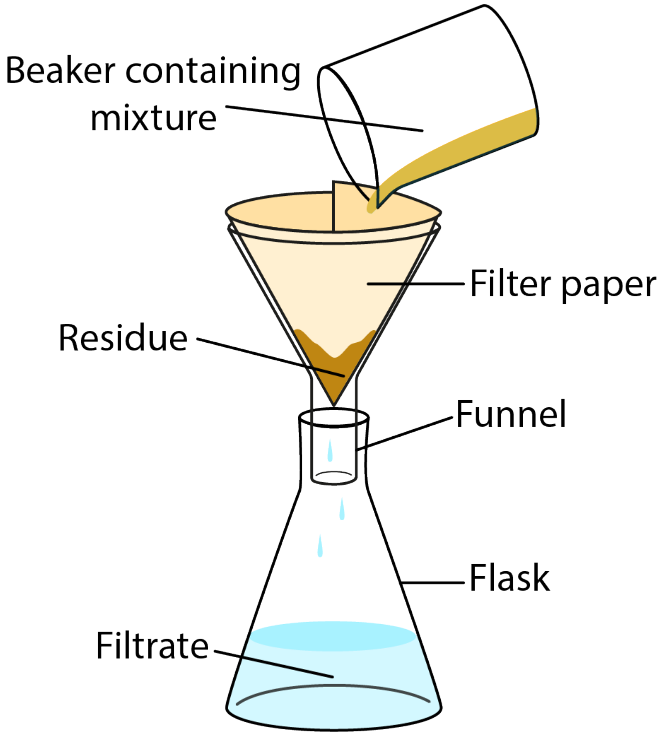
Impure. The sodium melts over a **range of temperatures**, and the melting point is **not** the same as the standard one. This indicates that there are **other substances** in the sample of sodium, making it a **mixture** rather than a pure element.

Mixture separation

It is **useful to separate mixtures,** for example we get salt to eat from separating it from the sea. On some islands, they make drinking water from seawater by separating it from the salt in the sea. There are four methods used to extract pure substances and separate mixtures: **filtration, crystallisation, simple distillation and fractional distillation.**

Filtration

Filtration separates a **solid that cannot dissolve in a liquid** from a liquid. Filter paper is placed in a funnel, and a container is put underneath. The mixture of the insoluble solid and liquid is poured **through the filter paper**. The filter paper **only allows the liquid to pass** into the container below. Once in the container, the liquid is known as the **filtrate**. The solid **remains in the filter paper**. The solid is now known as the **residue.** This method results in the separation of an insoluble solid and liquid.



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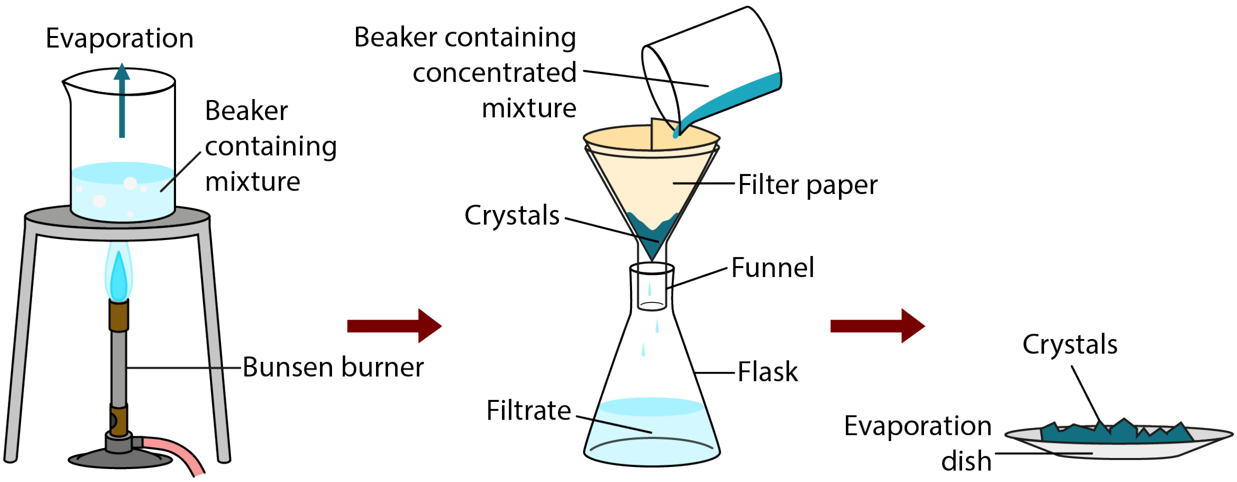
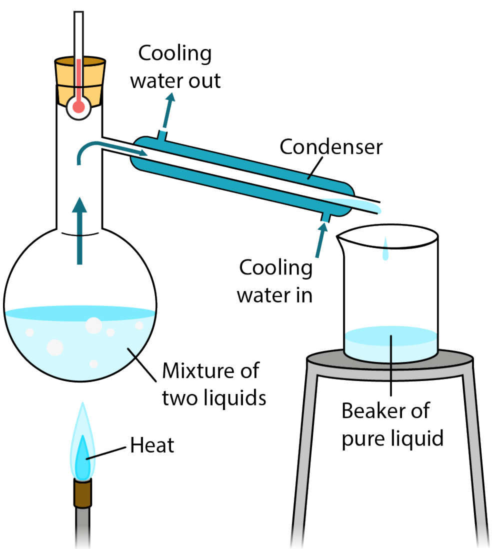
Crystallisation

Crystallisation separates a **dissolved solid** from a liquid. The mixture of the liquid and dissolved solid is **heated**. As the liquid heats, it will **evaporate into a gas**. Once all the liquid is evaporated, you will be left with **small crystals of the solid**. If you want larger crystals, **stop** heating the mixture when there is a **little liquid left**. Then use the **filtration method** to separate the crystals from the liquid, then allow the crystals to dry.



Simple distillation

Simple distillation separates a **solvent from a solution.** The dissolved **solute** has a much **higher boiling point** than the solvent. So, when the mixture is heated, the **solvent will evaporate first.** The mixture is poured into a round-bottomed flask that has a long neck. Coming out of this next is a thin tube with a condenser around it. As the mixture is heated the solvent with the **lower boiling point is evaporated**. As the gas of the solvent travels down the condenser, cold water causes it to condense and is collected in another flask as a liquid. The solvent is known as the **distillate** as it has been distilled.



Example

Simple distillation is used to make **pure water**. Having pure water is **very useful** for chemical reactions. Drinking water is **not** pure water, it might be safe to drink, but it still has **lots of dissolved minerals and ions**. Using the simple distillation method, we can produce **pure water, also known as distilled water.**

Solute, solvent and solution

- A **solute** is a substance that **dissolves** to make a solution.
- A **solvent** is a **liquid** that **dissolves** a solute.
- A **solution** is a **mixture** of two of more substances.
- In salt water, **salt** is the **solute**, **water** is the **solvent** and **together** they make a **solution.**

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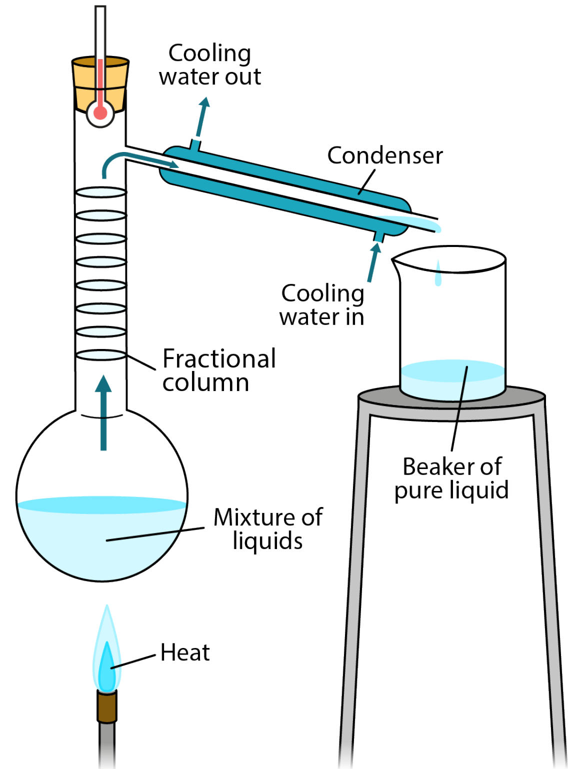
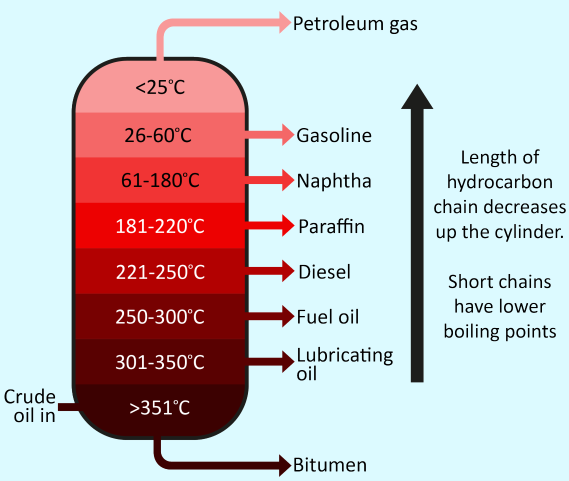
Fractional distillation

Fractional distillation separates a **mixture of liquids**. Similarly to simple distillation, the mixture of liquids is **heated** in a round-bottomed flask, with a **fractional column**. This time all the liquid is **evaporated, in stages** . In the neck of the flask, there are **glass beads.** These make the **gases spread out.** The gases with the **highest boiling points** will be towards the **bottom**, and the **gases** with the **lowest boiling points** will be at the **top**. Different gases can then be distilled off, at different temperatures. **First,** heat the mixture to the boiling point of the first substance with the lowest boiling point. **Collect this liquid** and then **raise the temperature** and **repeat**.



Example



Fractional distillation of crude oil

Crude oil is a **mixture** of **hydrocarbons** – chains of carbon and hydrogen. Different lengths of hydrocarbons have **different uses**, and **boiling points**. For example, methane, CH_4 and ethane, C_2H_8 are short chains and have such low boiling pointes they are gases. Large hydrocarbons have high melting points. To **separate** the different lengths crude oil is **heated**, different lengths of hydro-carbon **condense at different temperatures** and **leave the column at different heights.**

Formulations

Formulations are **mixtures** that have been designed as a **useful product**. These products are often **complex mixtures** with many different compounds and elements in them, each having a **purpose**. Shampoo is an example of a formulation. Its function is to clean hair. If you look at the back of the bottle, you will see a long list of compounds. Some will make it smell nice, and others will remove the dirt and grease from your hair – each has a purpose that makes the shampoo work and pleasant to use.

Examples of formulations

- Metal alloys
- Fuels
- Cleaning products
- Paints
- Medicines
- Fertilisers
- Food

Purity and chromatography

Water purification

Sewage water and freshwater need to be purified before we can drink it. This happens in five stages:

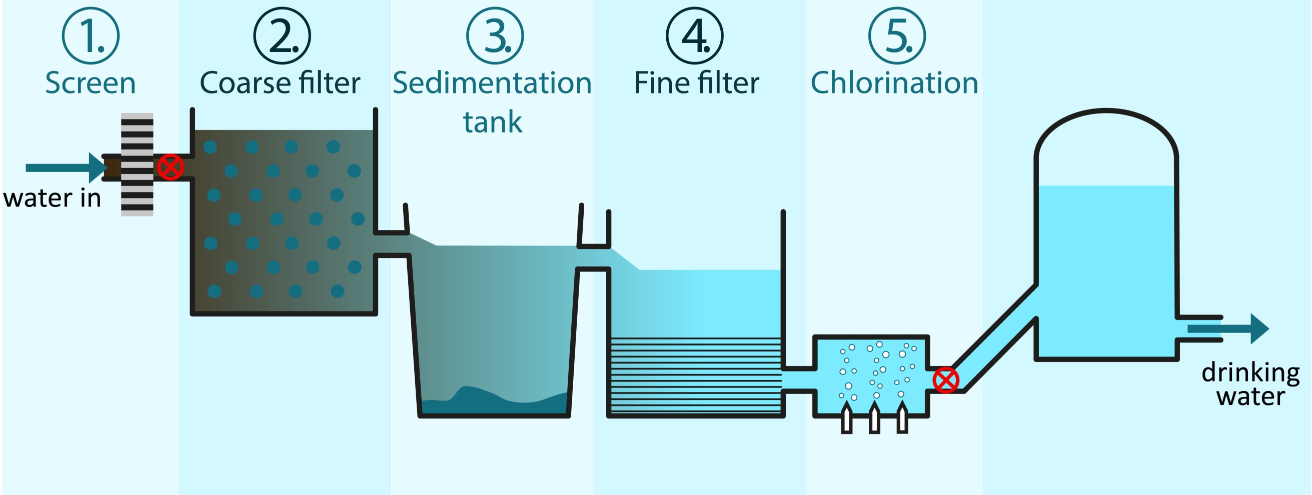
1. Screen – filters out large debris. E.g. branches and cans

2. Coase filter – removes insoluble grit particles. Made from clean sand and gravel.

3. Sedimentation aluminium sulphate within tank clumps smaller insoluble particles together which then sink

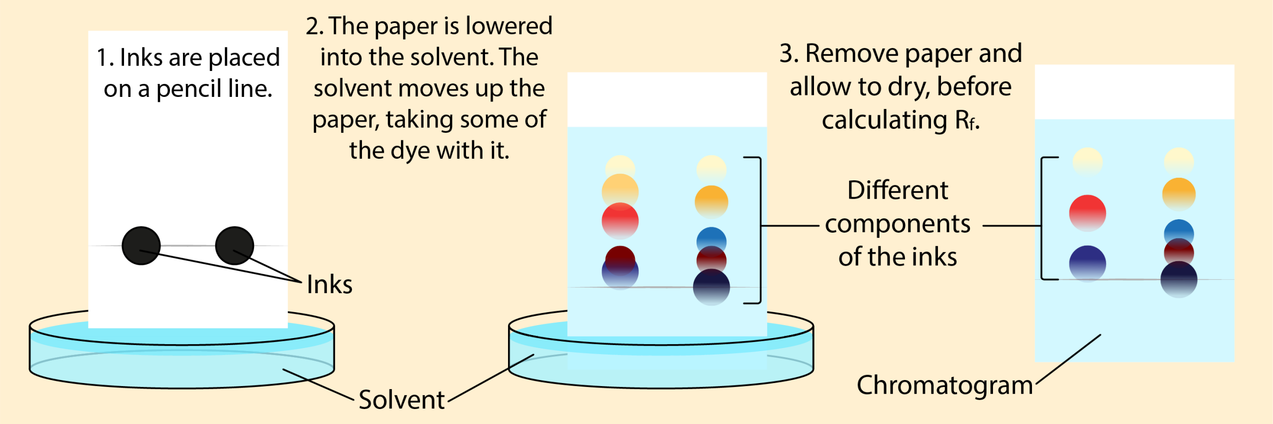
4. Fine filter - removes very small particles

5. Chlorination – chlorine gas is bubbled through to microorganisms



Chromatography

Chromatography is an analytic technique used to **separate a mixture of soluble substances** into its **individual substances**. It results in the possible identification of the substances in that mixture. There are many different types of chromatography. You need to know about **paper chromatography** and **thin layer chromatography**. In paper chromatography, the soluble substances are often food colourings, inks, dyes or plant pigments. We call the **solvent the mobile phase**. The **mobile phase is the substance** that moves, **carrying** the **different components** with it. The **paper is the stationary phase**, as it does not move. The stationary phase is the phase in which the **mobile phase passes through**.

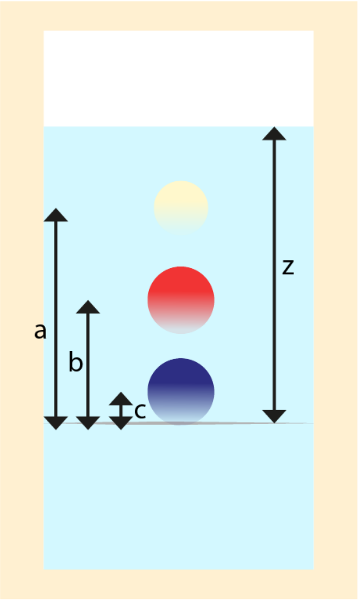


Purity and chromatography

Separation of a mixture by chromatography **results in a chromatogram**. Chromatograms are **visual end results** – the **spread** of the different induvial substances in the ink sample. The different individual substances in the mixture, move at **different rates** up the paper. This is because the different substances are attracted to the phases in different amounts. The ratio that a substance moved to the distance the solvent moved is known as the **R_f value:**

$$R_f = \frac{distance\ moved\ by\ individual\ substance}{distance\ moved\ by\ solvent}$$

Different substances have **different R_f values**. By calculating an R_f value, you can identify different substances in the sample. For the diagram on the left the R_f for the red substance is $\frac{b}{z}$ and for the yellow substance it is $\frac{a}{z}$.



If the sample is a **mixture,** the paper chromatography will result in **many different dots,** each representing the induvial substances. However, a **pure substance** will only result in **one dot**. This is useful as we can compare unknown mixtures to known pure substances, and work out what they are. If the individual substances in the mixture have the same R_f as the pure substance, it means that they are the same substance.

Example

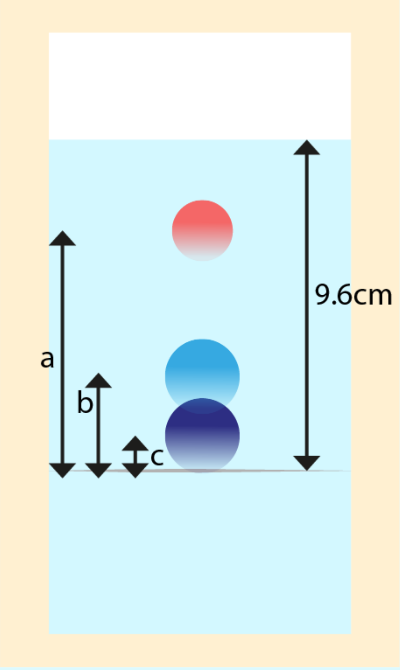
Work out the R_f values of each dot, if a = 8.1cm, b = 3.0cm and c = 0.9cm.

$$R_f = \frac{distance\ moved\ by\ individual\ substance}{distance\ moved\ by\ solvent}$$

a: $R_f = \frac{8.1}{9.6} = 0.84$

b: $R_f = \frac{3}{9.6} = 0.31$

c: $R_f = \frac{0.9}{9.6} = 0.09$



Purity and chromatography

Thin Later Chromatography

Thin Later Chromatography is another form of chromatography. Instead of paper, a **thin layer of an inert substance** (e.g. silica) is supported **on a flat plate** (e.g. glass). The distance a sample travels along the **stationary phase** can depend on size or polarity of the molecules:

- **Smaller molecules** are more mobile and will travel quicker
- **Polar molecules** will travel faster IF the solvent is polar

Worked exam questions

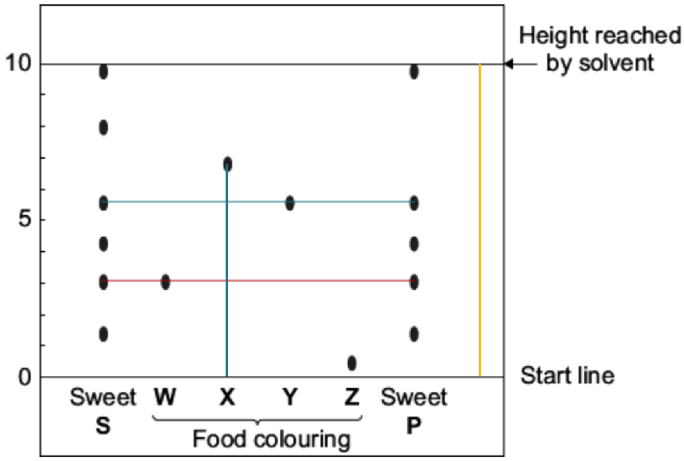
Q5. Read the article.

Problem food colourings

Scientists say they have evidence that some food colourings cause hyperactive behaviour in young children.

These food colourings are added to some sweets.

The results are shown on the chromatogram.



(a) Food colourings, such as **W**, **X**, **Y** and **Z**, are added to some sweets.

Suggest **one** reason why.

To add colour

(b) In chromatography, the R_f value = $\frac{\text{distance moved by the colouring}}{\text{distance moved by the solvent}}$

Use the scale on the chromatogram to help you to answer this question.

Which food colouring, **W**, **X**, **Y** or **Z**, has an R_f value of 0.7?

Step 1: measure the distance moved by the solvent – 5.0cm
Step 2: 0.7 is going to be the line that is about 70% the size of the distance moved by the solvent – try X first
Step 3: Put distances in to equation above:

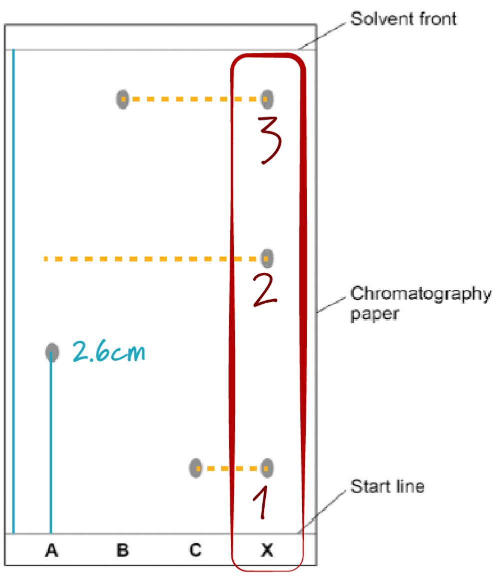
$$\frac{3.5cm}{5.0cm} = 0.7$$

(c) From the chromatogram, what conclusions can the scientist make about the colourings in sweets **S** and **P**?

Sweet S has 6 food colourings in. Sweet P has 5 food colourings in. Both contain colourings W and Y. Neither contain X and Z. The sweets cause hyperactivity.

Figure 2 shows the student's results.

Figure 2



How many dyes were in **X**?

Tick **one** box.

1 ☐	3 ☑	4 ☐	6 ☐

(c) Which dye, **A**, **B** or **C**, is **not** in **X**?

Write your answer in the box. A

The dyes that are the same will have moved up the paper by the same amount

Use **Figure 2** to complete the table below.

Calculate the value for R_f for dye **A**.

	Distance in mm
Distance moved by dye **A**	2.6cm
Distance from start line to solvent front	7.0cm

Use the equation:

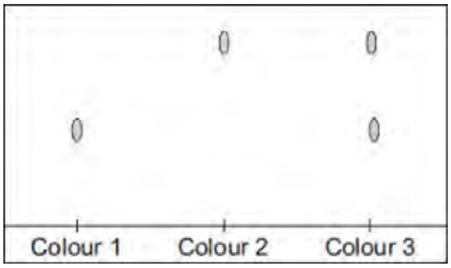
$$R_f = \frac{\text{distance moved by dye } \mathbf{A}}{\text{distance moved by solvent}}$$

Give your answer to two significant figures.

$\frac{2.6cm}{7.0cm}$

R_f value = 0.37

Chromatography was used to compare three of the colours used to coat the chocolate sweets.



What do these results tell you about these three colours?

Colour 1 and 2 are only made up of one dye.
Colour 3 is made up of two dye. These two dyes are the dyes in colour 1 and 2.

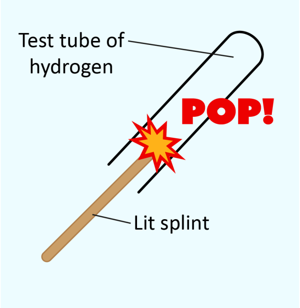
Tests for gases and ions

Learning objectives

- ❑ Know the tests for the gases; hydrogen, oxygen, carbon dioxide and chlorine.
- ❑ Know the resulting colour for the flame tests of compounds containing; lithium, sodium, potassium, calcium, copper.
- ❑ Describe the tests to identify the ions; aluminium ion (Al^{3+}), calcium on (Ca^{2+}), copper ion (Cu^{2+}), iron(II) ion (Fe^{2+}), iron(III) ion (Fe^{3+}), and zinc ion (Zn^{2+}) in either state using sodium hydroxide solution.
- ❑ Know that carbonates react with dilute acids to form carbon dioxide
- ❑ Know the test for the halide ions
- ❑ Know that sulphate ions react with barium chloride (or barium nitrate) to form barium sulphate
- ❑ Know the benefits of using instrumental analysis rather than chemical analysis
- ❑ Deduce the metals in a compound using flame emission spectroscopy

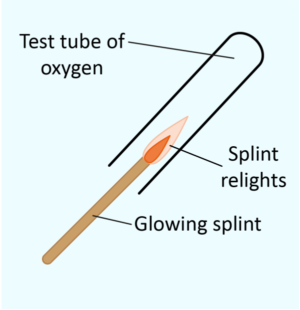
Tests for gases

Hydrogen, oxygen, carbon dioxide and chlorine can all be identified using different tests:



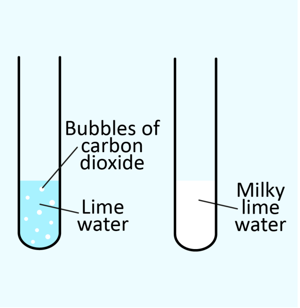
Hydrogen

To test for **hydrogen**, place **a lighted** wooden splint in a test tube of gas if the splint makes a **popping sound** there is hydrogen.



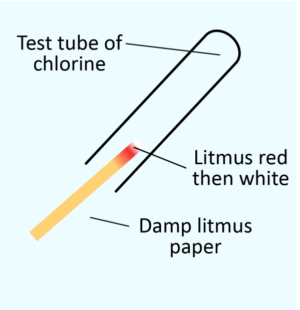
Oxygen

To test for **oxygen**, place a **glowing** splint into the test tube of gas if it **relights** it is oxygen.



Carbon dioxide

To test for **carbon dioxide bubble** the gas through **limewater**, if the limewater turns **milky**, the gas has carbon dioxide in it.



Chlorine

To test for **chlorine**, put some **damp litmus paper** in the test tube of gas if the litmus paper turns **red and then white**, the gas is chlorine. Chlorine gas also turns damp **starch-iodide paper blue-black** and has a characteristic **sharp, choking smell.**

Tests for gases and ions

Tests for cations

A **cation** is a **positive ion**. These are atoms or compounds that have **lost electrons**. As electrons are **negative**, a **loss of electrons** results in a **positive** charge.

Flame tests

When compounds containing metals are put into flames, they **change the colour of the flame.** This is known as a **flame test**. The resulting colour indicates the metal ion in the compound.

Metal	Flame	Colour
Lithium		Crimson
Sodium		Yellow
Potassium		Lilac
Calcium		Orange-red
Copper		Green

Method of the flame test:

1. Dip a **clean wire loop** into the solid sample being tested.
2. Hover the loop around the edge of a **blue flame** from a Bunsen burner.
3. Observe the **colour change.**

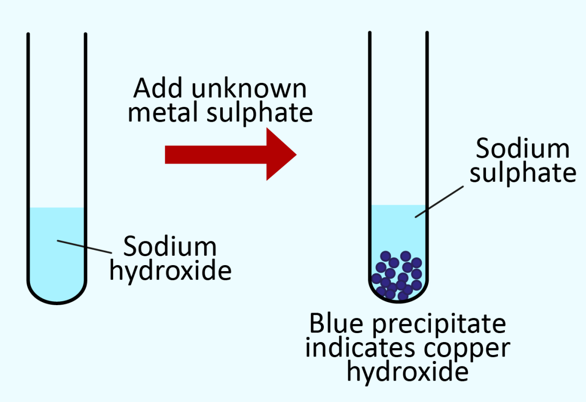
Sodium hydroxide test for metal ions

Some **metal ions** react with dilute **sodium hydroxide** forming insoluble **precipitates**. These precipitates are **metal hydroxides**, and their **colour** can identify the metal ion.

copper sulphate + sodium hydroxide → sodium sulphate + copper hydroxide

$CuSO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + Cu(OH)_2(s)$

$CU^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$



Here is a list of some ions and the colour of their resulting precipitate:

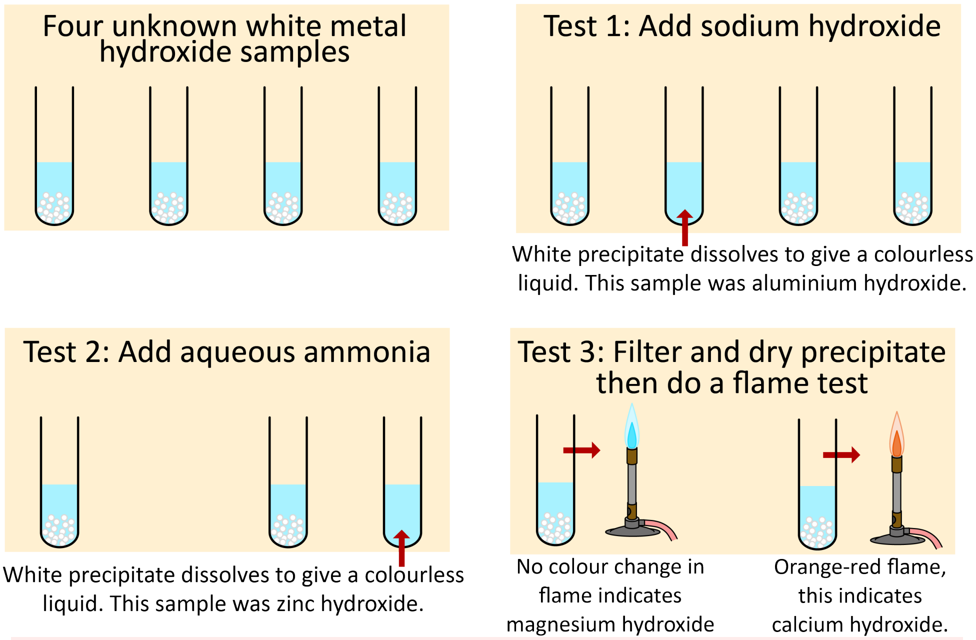
Metal ion	Formula	Precipitate colour
Aluminium	Al^{3+}	White
Calcium	Ca^{2+}	White
Magnesium	Mg^{2+}	White
Zinc	Zn^{2+}	White
Copper(II)	Cu^{2+}	Blue
Iron(II)	Fe^{2+}	Green
Iron(III)	Fe^{3+}	Brown

However, **four** of these produce a **white precipitate**. We can distinguish them from each other using **more tests:**

1. Test for **aluminium** with **sodium hydroxide**
2. Test for **zinc** with **ammonia**
3. Use the **flame test** to test for **calcium**
4. All test **negative** means **magnesium**

Tests for gases and ions

Differentiating between white metal hydroxide samples



Example equations for the formation metal hydroxides:

$$FeSO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + Fe(OH)_2(s)$$

$$Fe^{2+}(aq) + 2OH^{-}(aq) \rightarrow Cu(OH)_2(s)$$

$$AlCl_3(aq) + 3NaOH(aq) \rightarrow 3NaCl(aq) + 2Al(OH)_3(s)$$

$$Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Cu(OH)_3(s)$$

Tests for anions

Anions are **negative ions**. Negative ions have **gained electrons**. As electrons are negative, a **gain in electrons** results in a **negative charge**.

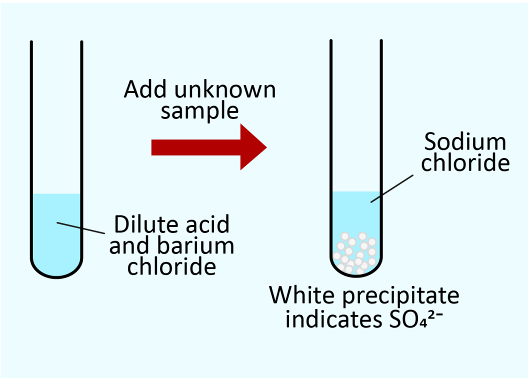
Carbonate ions, CO_3^{2-}

Metal carbonates react with **dilute acids** to form **carbon dioxide** gas. We can then capture this gas and bubble it through limewater. The limewater will turn milky from the carbon dioxide, indicating carbonate ions in the sample.

$$CaCO_3\,(aq) + HCl(aq) \rightarrow CaCl\,(aq) + H_2O(aq) + CO_2(g)$$

Sulphate ions, SO_4^{2-}

Sulphate ions can be tested for using **barium chloride** (or barium nitrate). First, we add a **dilute acid** (but not sulfuric acid) to **remove** the carbonate ions. Carbonate ions would react with barium chloride to form a **white precipitate**, giving a **false positive**. Then you add a few drops of **barium chloride or nitrate,** if sulphate ions are present a **white precipitate forms.**



$$BaCl_2\,(aq) + Na_2SO_4\,(aq) \rightarrow 2NaCl\,(aq) + BaSO_4(s)$$

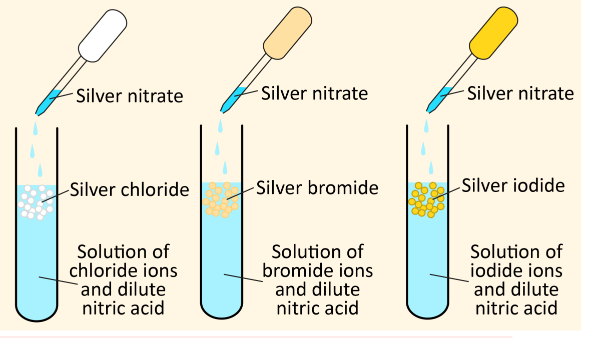
$$Ba^{2+}(aq) + SO_4^{2-}\,(aq) \rightarrow BaSO_4\,(s)$$

Tests for gases and ions

Halide ions, Cl^-, Br^- and I^-

The halogens are elements in **group 7** of the **periodic table**; this includes chlorine, bromine and iodine. Halogen ions are called **halides**, e.g. bromide, Br^-. Halides are tested for using **silver nitrate solutions**. First, the test solution is **acidified** using a little **dilute nitric acid**. This removes any **carbonate ions** – they would form a **white precipitate** and give a **false positive** for chloride ions. Next, we add the **silver nitrate**. The **halides** will react, forming **sliver-halide compound**. The colour of the silver-halide depends on which **halide is in it**.

- **Chloride** ions form silver chloride, a **white** precipitate
- **Bromide** ions form silver bromide, a **cream** precipitate
- **Iodide** ions form silver iodide, a **yellow** precipitate



Silver nitrate + sodium chloride → sodium nitrate + silver chloride

$AgNO_3 (aq) + NaCl (aq) \rightarrow NaNO_3(aq) + AgCl (s)$

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

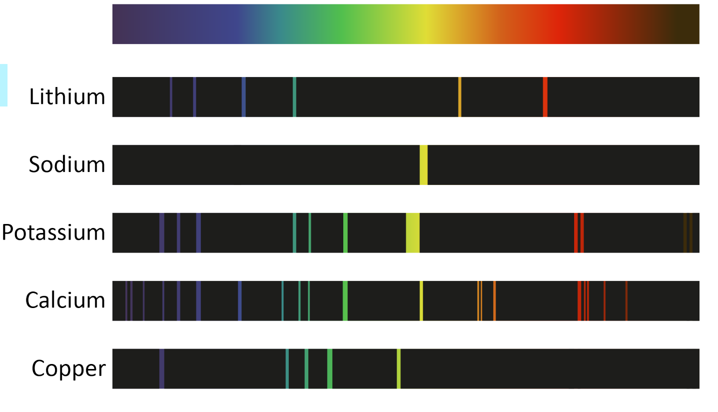
Instrumental methods

Compounds and elements can be **detected** and **identified** using a wide range of **different machines**. This is known as **instrumental analysis**. Instrumental methods is **better** than using chemical tests.

1. **Instrumental methods** are **more accurate** at identifying element and ions. Chemical test **introduces human error**, such as forgetting to add acid when testing for Sulphate or halide ions, resulting in a false-positive result.
2. **Instrumental methods** are **more sensitive**, picking up on microscopic samples. If there is **not enough sample to produce a visible change**, the chemical test will have a **false negative**. Chemical testing relies on visual results, whereas **instrumental methods** provide **quantitative data**.
3. **Instrumental methods work faster.**

Flame emission spectroscopy

Flame emission spectroscopy is a scientific technique based on **flame testing**. It works by **vaporising** a sample and then **putting it into a flame**. The **light given out** from the flame is passed through a **spectroscope**, producing an **emission spectrum**. The spectrum is **unique to each ion** and can be used to **identify the metal ions** in the sample and measure their concentrations.



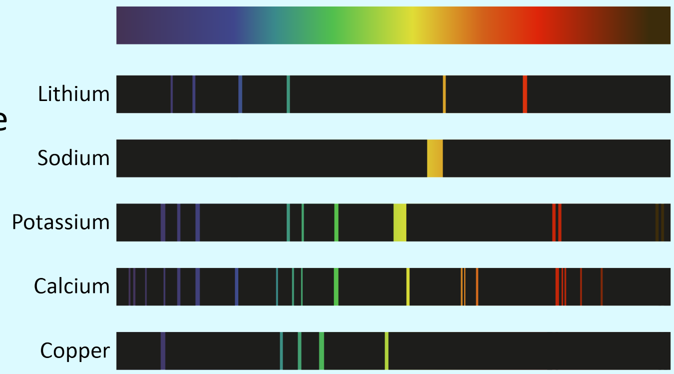
Tests for gases and ions

Example

Use the line spectra to work out what ions are in the unknown mixture. Then explain why a flame test would not identify this.

The line spectrum of the unknown mixture has the lines from **sodium** and **calcium**. This indicates that the mixture has **both** sodium and calcium in.

A flame test would **not pick up** on the sodium, as its yellow flame would be **hidden** by calcium's red flame.



Unknown mixture of two ions

Worked exam questions

Q3. Read the information in the box and then answer the questions.

> Seidlitz Powder is the name of a medicine.
>
> Seidlitz Powder comes as two powders. One powder is wrapped in white paper and contains tartaric acid ($C_4H_6O_6$). The other powder is wrapped in blue paper and contains potassium sodium tartrate ($KNaC_4H_4O_6$) and sodium hydrogencarbonate ($NaHCO_3$).
>
> The contents of the blue paper are completely dissolved in water and then the contents of the white paper are added.
>
> The equation which represents this reaction is:
>
> $$C_4H_6O_6\,(aq) + 2NaHCO_3\,(aq) \longrightarrow Na_2C_4H_4O_6\,(aq) + 2H_2O\,(l) + 2CO_2\,(g)$$

(a) Describe and give the result of a test to identify the gas produced in this reaction.

The gas is CO_2. The test for CO_2 is to bubble it through lime-water. If the lime-water turns milky the gas is CO_2

Chlorine is produced at the positive electrode. Describe and give the result of a chemical test to prove that the gas is chlorine.

Chlorine gas will turn damp litmus paper red, then it will bleach it white.

Q4. Alums are salts. They have been used since ancient times in dyeing and medicine and still have many uses today.

Three alums are shown in the table:

Name	Ions present
Ammonium alum	NH_4^+ Al^{3+} SO_4^{2-}
Potassium alum	K^+ Al^{3+} SO_4^{2-}
Sodium alum	Na^+ Al^{3+} SO_4^{2-}

(a) These alums contain sulfate ions (SO_4^{2-}).

Describe and give the result of a chemical test to show this.

Test Sulphate ions can be tested for using barium chloride (or barium nitrate).

Result The test results in the formation of a white precipitate (barium sulphate).

(b) These alums contain aluminium ions (Al^{3+}).

Describe how sodium hydroxide solution can be used to show this.

When aluminium is put in sodium hydroxide, it forms a white precipitate. However, it is not the only metal that does. We must do a further test by adding excess sodium hydroxide. This turns makes the white precipitate dissolves.

(c) Aluminium ions do not give a colour in flame tests. However, flame tests can be used to distinguish between these three alums.

Explain how these three alums could be identified from the results of flame test:

Potassium alum will turn flame lilac.

Sodium alum will turn flame yellow.

Ammonium alum not change the colour of the flame

Q5.(a) The colours of fireworks are produced by chemicals.

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Three of these chemicals are lithium sulfate, potassium chloride and sodium nitrate.

(i) A student wants to carry out flame tests on these three chemicals.

Describe how to carry out a flame test.

Dip a clean wire loop into samples.
Then hold the loop in a blue flame.

....

....

(iii) Dilute nitric acid and silver nitrate solution are added to solutions of the three chemicals.

A white precipitate forms in one of the solutions.

Which chemical produces the white precipitate?

Potassium Chloride – remember that silver nitrate is a test for the halide ions!

(b) The student tests a fourth chemical, **X**.

(i) The student adds sodium hydroxide solution to a solution of chemical **X**.

A blue precipitate is formed.

Which metal ion is in chemical **X**?

Copper – sodium hydroxide tests for metal ions