

**GCSE Chemistry**

**21st Century Science**

**C7: Chemistry for a sustainable world [Part 2]**

**Name:** \_\_\_\_\_

## Reversible reactions and equilibria

There are important questions that chemists need to ask if they are going to control reactions to get the right results.

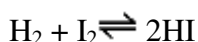
[1] We must know which way the chemical reaction will precede.

[2] We must know how much product the reaction will produce in this direction.

**Many chemical reactions are irreversible but some can be reversed, so the products can change back into the reactants. In industrial processes, it is important to be able to control these reactions to make the most product as quickly and cheaply as possible.**

### Reversible reactions

When a chemical reaction is reversible, we use the  $\rightleftharpoons$  symbol instead of an arrow. For example:



This means that while  $\text{H}_2$  and  $\text{I}_2$  can react together to form HI molecules, the HI molecules can also react with each other to make  $\text{H}_2$  and  $\text{I}_2$  again.

### Equilibrium

**Question:** Complete the following text using the words that follow:

In some reactions, the forward reaction goes at the same \_\_\_\_\_ as the backward reaction. When this happens, the \_\_\_\_\_ are replaced as fast as they are used up and the products react as fast as they are made. Even though they are reacting all the time, their total amounts do not change. This is called \_\_\_\_\_.

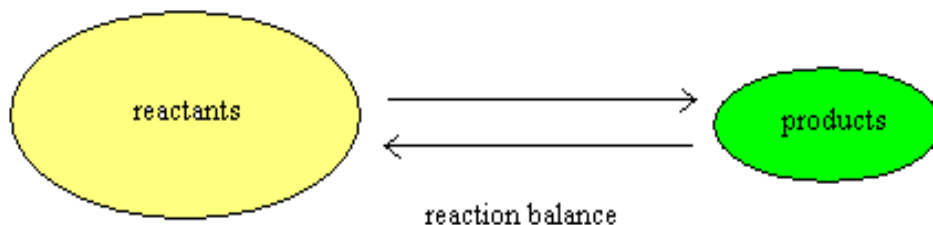
- The rate of the forward reaction equals the rate of the backward reaction.
- The concentration of reactants and \_\_\_\_\_ do not change.

**Possible words:** products, equilibrium, rate, reactants

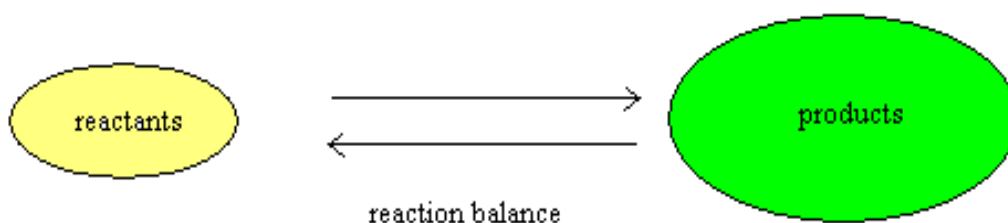
### Equilibrium position

When the backwards and forwards reaction balance, the concentrations of reactants and products are never equal. There is usually more of one than the other.

If the concentration of reactants is greater than the concentration of products, the equilibrium position is on the left.



If the concentration of the reactants is less than the concentration of the products, we say the equilibrium position is on the right.

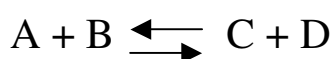


**Question:** State how you know when a reversible reaction has reached equilibrium.

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**Question:** At equilibrium, the reaction:



Has 5 moles of A, 5 moles of B, 1 mole of C and 1 mole of D.

What can you say about the equilibrium position?

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### Reaching equilibrium position

This only works if the chemicals cannot escape – if it is a closed system.\_

- Allow reactants to react
- Forward reaction creates products
- Forward reaction slows down as reactants are used up
- Backward reaction speeds up, as more and more product is available for reaction.
- Eventually the backward reaction is as fast as the forward reaction. Equilibrium has been reached.

The symbol equation showing ice turning into water is as follows:  $\text{H}_2\text{O [s]} \longrightarrow \text{H}_2\text{O [l]}$

**Question:**

[a] Write a symbol equation showing water turning into steam.

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[b] Write another equation to show steam condensing to water

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[c] Write a third equation to show the changes in parts [a] and [b] as a single reversible change.

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**Question:** Write an equation to show the reversible reaction of carbon monoxide gas with steam to form carbon dioxide and hydrogen.

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**Question:** In your equation, what happens in the forward reaction?

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**Question:** In your equation, what happens in the backward reaction?

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### **Dynamic equilibrium**

If they take place within a closed container, reversible reactions will eventually reach a state of **dynamic equilibrium**. This is when the forwards reaction and the reverse reaction are taking place at the same rate (speed).

This means that even though there will be some **products** and some **reactants** in the mixture, the amount of product in the reaction mixture remains constant over time.

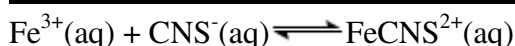
Some reactions are able to go in two directions; forward and reverse. They are known as reversible reactions.

The forward and reverse reactions occur at the same time, and never stop. As a result, they are called **dynamic reactions**.

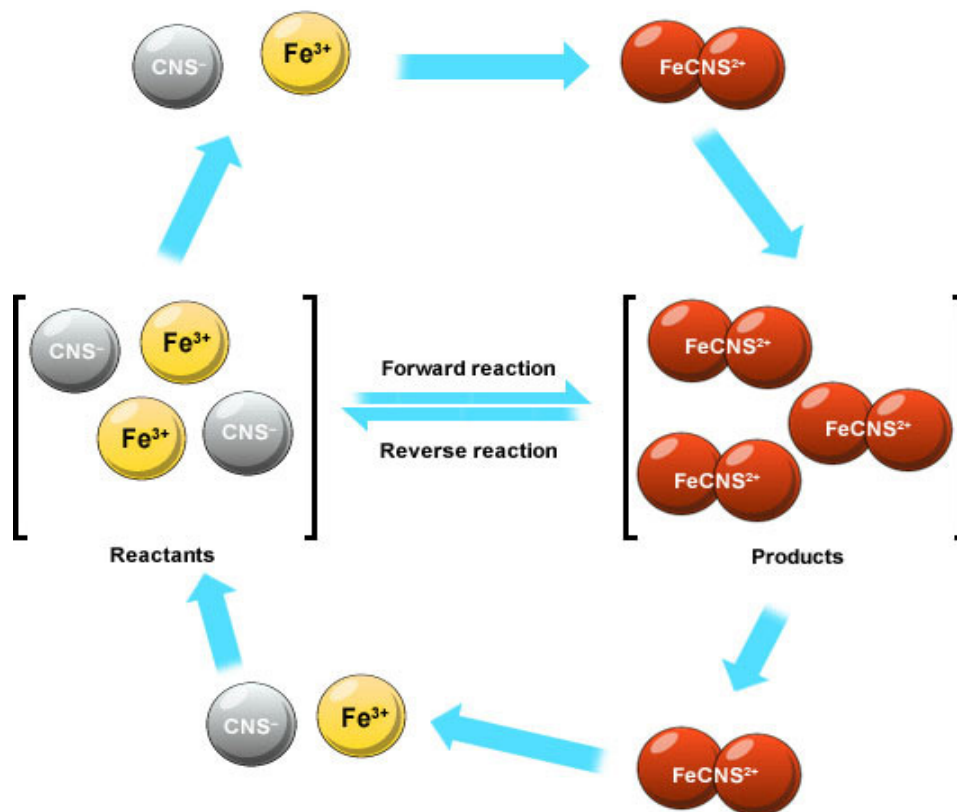
When the rate of the forward reaction is equal to the rate of the reverse reaction, the reaction is said to have reached **equilibrium**.

**At equilibrium, the concentrations of the reactants and products are constant, but are not necessarily equal.**

**Example: the reaction of iron(III) ions with thiocyanate ions.**



Pale yellow iron(III) ions react with colourless thiocyanate (CNS) to produce red iron thiocyanate.



When there are more products than reactants present, the position of equilibrium lies to the right.

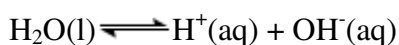
**Example: in ester formation, there are more products than reactants at equilibrium.**



In this example of ester formation, ethanoic acid reacts with methanol to produce methyl ethanoate (the ester) and water. The equilibrium lies to the right.

When there are more reactants than products present, the position of equilibrium lies to the left.

**Example: in water, only a small proportion of the molecules have split to form ions at equilibrium.**



In this example of water, the equilibrium lies to the left. Only a few molecules have split to form ions.

It doesn't matter whether the reaction starts with 100% reactants or 100% products, the reaction will always reach the same equilibrium position.

**Question:** What is meant by the expression 'The equilibrium lies to the left?'

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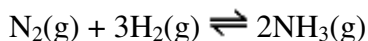
### **Making ammonia - the Haber process**

Ammonia,  $\text{NH}_3$ , is a very important chemical. It is used in the manufacture of fertilisers, explosives, dyes, medicines and a variety of other chemicals.

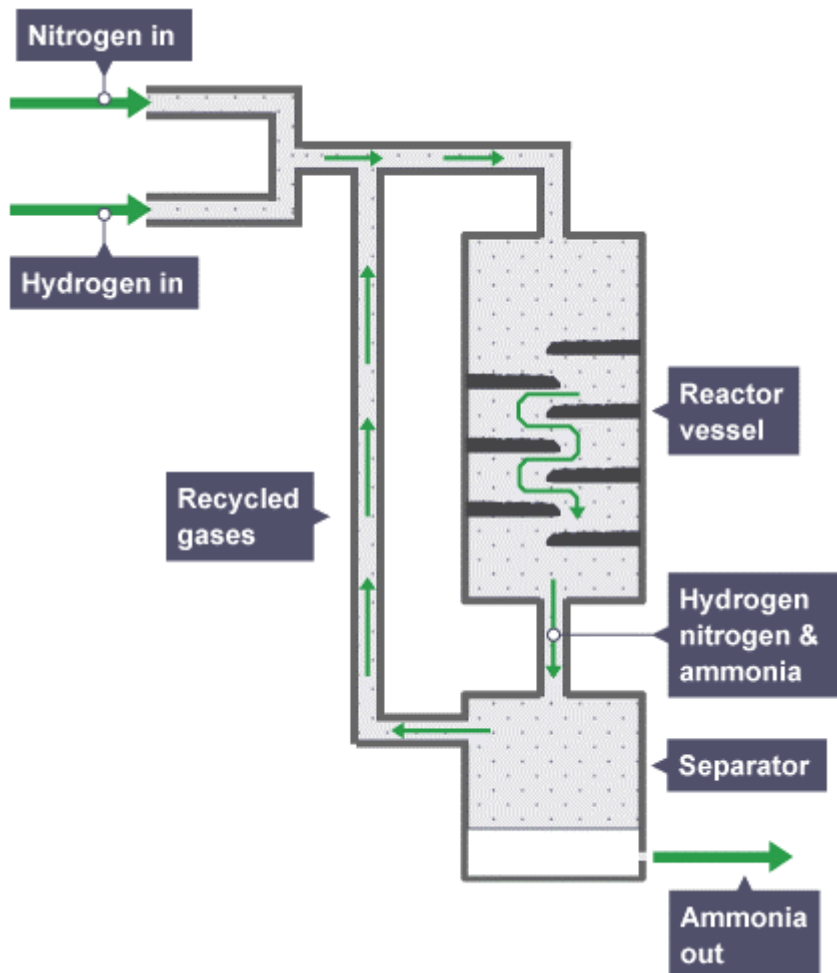
It has been estimated that one third of the world's population rely on food grown using synthetic fertilisers. Without ammonia, millions of people would starve. Some naturally-occurring bacteria use enzymes to 'fix' nitrogen from the air, but these natural processes are not sufficient to sustain the world's population.

Ammonia is produced in the **Haber process** by reacting hydrogen with nitrogen. This is difficult because nitrogen is such an unreactive gas. The chemical equations are:

nitrogen + hydrogen  $\rightleftharpoons$  ammonia



The reaction is **reversible**.



The Haber process

### **Raw materials**

The nitrogen comes from the air. The hydrogen is produced in a reaction between methane (natural gas) and steam.

### **Conditions in the Haber process**

The conditions chosen for the Haber process aim to maximise the yield of ammonia in the shortest time and at the lowest cost.

### **Pressure and temperature**

When the pressure is increased, the yield **increases**. When the temperature is increased, the yield **decreases**.

## Recycling

The three gases (nitrogen, hydrogen and ammonia) do not actually stay in the reactor for long enough to reach equilibrium.

The yield is further increased by **recycling** the unreacted hydrogen and nitrogen after they have been separated from the ammonia (when the mixture leaves the reactor).

## Using a catalyst

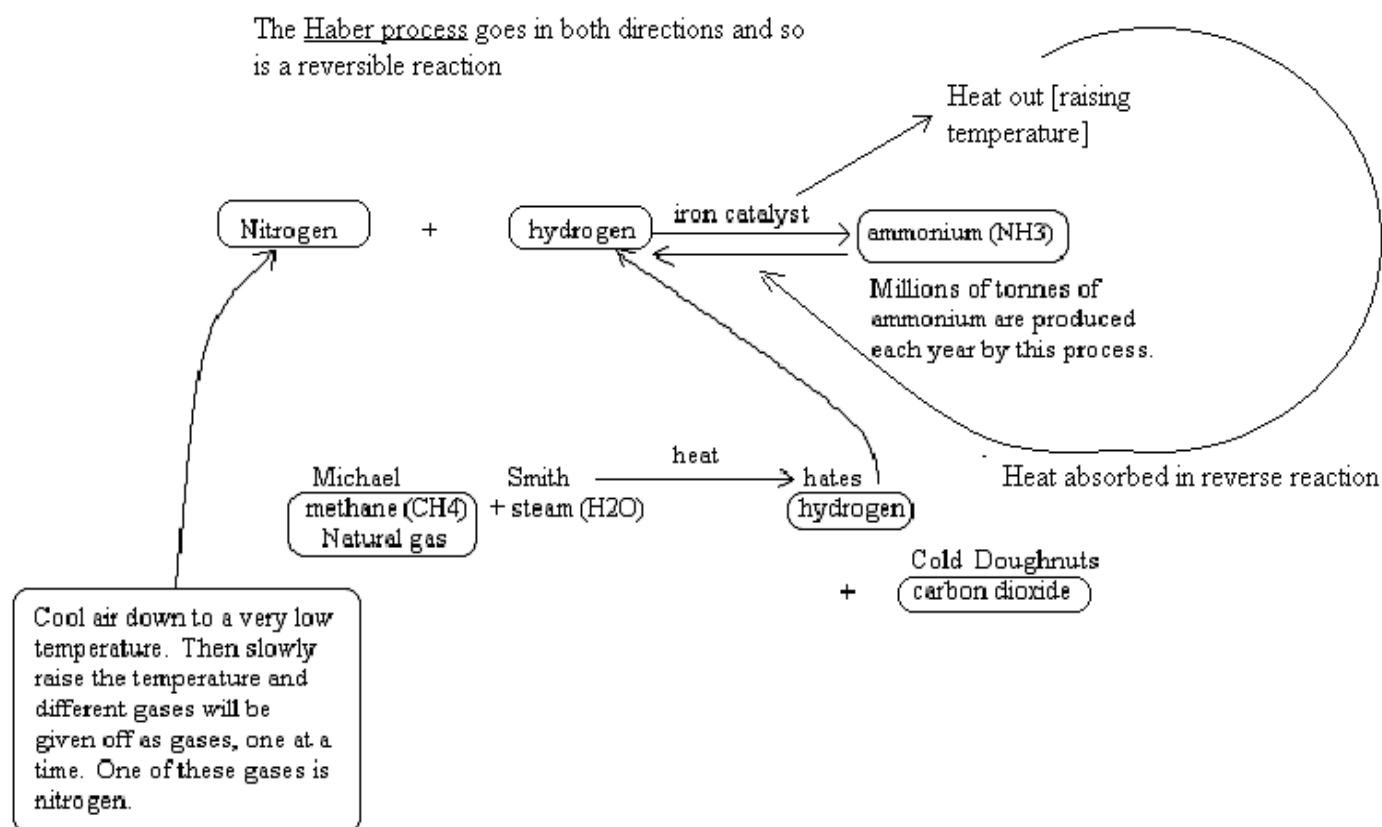
An iron catalyst is used to increase the rate of the reaction. The catalyst does not affect the yield of ammonia.

Scientists are constantly trying to find new catalysts that will **speed up the reaction** even more, and therefore make the Haber process more efficient.

## Ammonia

The formula for ammonia is  $\text{NH}_3$ . Joining nitrogen and hydrogen makes ammonia.

Nitrogen comes from air. Hydrogen is made from natural gas or by cracking oil fractions.



**Question:** Give two features of a catalyst.

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**Question:** Fritz Haber solved the problem of making nitrogen and hydrogen react. What was the problem?

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## What affects the costs of running a big chemical plant?

Modern ammonia plants make 1500 tonnes of ammonia every day. These plants are carefully designed to keep the cost of making ammonia as low as possible.

What affects the cost of making ammonia?

- The cost of building the plant in the first place.
- The labour costs – paying people's wages.
- The cost of the chemicals – the hydrogen and nitrogen.
- The energy costs.
- How fast the reaction will go – the catalyst make it go faster, so more ammonia can be made.

## What is ammonia used for?

When Fritz Haber first did his experiments, he wanted the ammonia to make explosives.

These days:

- Over 80% of all ammonia goes into fertilisers.
- Ammonia is also used to make nitric acid – most of which goes into more fertilisers.
- Very small amounts of ammonia are used to make household cleaners.

**Question:** Complete the following table:

Costs involved in making ammonia that involve buying something	Costs involved in making ammonia which do not involve buying something
Buying the plant	

**Question:** What is the main use of ammonia?

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**Question:** What would happen to the costs if ammonia could be made at a lower temperature? Explain your answer.

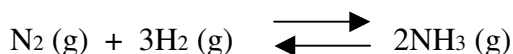
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## More on the Haber process

**Question:** Complete the following text using the words that follow:



When the temperature is \_\_\_\_\_, the reaction goes faster producing more ammonia. This forward reaction is exothermic and actually gives off more heat to raise the \_\_\_\_\_ even further.

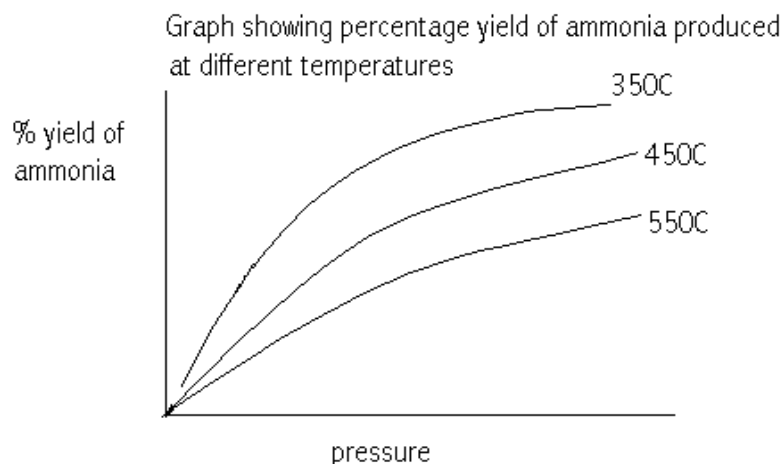
However, the reaction is \_\_\_\_\_ and ammonia can thermally decompose into \_\_\_\_\_ and \_\_\_\_\_. This reversible reaction is \_\_\_\_\_. This means that at higher temperatures a greater percentage of ammonia will \_\_\_\_\_ down.

450C is the optimum temperature – a greater yield could be made at a lower temperature but this would obviously take longer. Catalysts do not affect the \_\_\_\_\_ - they just make the reaction go faster.

**Possible words:** yield, break, reversible, nitrogen, hydrogen, temperature, endothermic, raised

### Optimum conditions

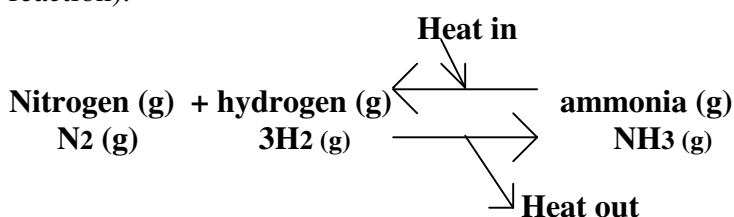
In this case the conditions that make the chemical most cheaply by producing the greatest yield / day (maximum profits) are known as the **optimum conditions**.



As can be seen from this graph, the actual yield of ammonia produced at 350C is much higher than at 550C, but it may take a very long time to produce ammonia at this temperature!

So in reality, a compromise is found! The production of ammonia takes place at 200 atmospheres in closed steel container at about 450C, (hot iron catalyst).

At high temperatures the rate of production of ammonia increases but at the same time, less remains stable and more and more breaks back down to nitrogen and hydrogen (reverse endothermic reaction).



450C is the best compromise and produces the most amount of stable ammonia each second. Overall the reaction at this temperature has a fairly low percentage yield. However, as long as the unreacted chemicals can be recycled, they can go back into the reaction vessel. This makes it profitable.

**Question:** As can be seen from the graph, increasing the pressure, increases the yield. Why don't we just use incredibly high pressures to increase the yield even more?

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## Equilibria

Some reactions are totally confused; they go in both directions, even at the same time!

### Reversible reactions

Reversible reactions can go forwards and backwards at the same time.

For example, nitrogen and hydrogen react to form ammonia. The ammonia can then break down to reform nitrogen and hydrogen in the reverse reaction.

Nitrogen + hydrogen  $\rightleftharpoons$  ammonia

Notice the symbol  $\rightleftharpoons$ , which shows that the reaction is reversible.

**Question:** Write the word equation for the forward reaction

**Question:** Write the word equation for the backward reaction.

**Question:** H<sup>+</sup> ions can join to OH<sup>-</sup> ions to form water (H<sub>2</sub>O) in a reversible reaction. Show this below:

The reaction of nitrogen with hydrogen is exothermic. The reverse reaction, the decomposition of ammonia is endothermic. Le Chatelier's principle states the following:

**'When conditions change, an equilibrium mixture of chemicals responds in a way that tends to counteract the change'.**

Le Chatelier's principle predicts that if the temperature of an equilibrium mixture of the gases rises, then the equilibrium changes in a way that takes in the energy because this would tend to lower the temperature.

**Question:** According to Le Chatelier's principle, what would happen in the Haber process if there were an increase in temperature? Explain.

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## What affects the costs of ammonia production?

**Question:** Complete the following table using the text that follows:

What affects profits when manufacturing ammonia	How savings can be made
	Expensive to reduce pollution by obeying anti pollution laws. Little savings here.
	Automate chemical plants so few people are needed to operate them.
	Difficult! At present, hydrogen is made from natural gas (methane) or cracking oil fractions. Nitrogen is made from fractional sublimation of frozen air. This has to be cleaned, dried and compressed. Hopefully in the future we will get hydrogen from the electrolysis of water using hydroelectricity or solar
	Recycle them – saves money.
	High pressure = greater yields (more collisions / second of reacting gases with each other). However, this means that more expensive machinery is needed because pipes and fittings need to be stronger. A balance / compromise is needed.
	High temperatures produce a faster reaction producing ammonia faster. However: (1) High temperatures produce a greater breakdown of ammonia by thermal decomposition. (2) It becomes more expensive to buy the fuel.
	Good efficient catalyst produces ammonia faster and increases the yield at a particular temperature. This increases profits.

Energy costs e.g. in trying to achieve high temperatures and pressures, money is spent on fuels

To achieve a high pressure, money has to be spent on compressors and pumps to move chemicals through pipes.

Cost of reactants.

Labour costs

Pollution control

Use of catalyst

Unused reactant gases. Gases flow continuously through the reactor and are only in contact with the catalyst for a short time.

**Question:** Why does the mixture never get all the way to equilibrium?

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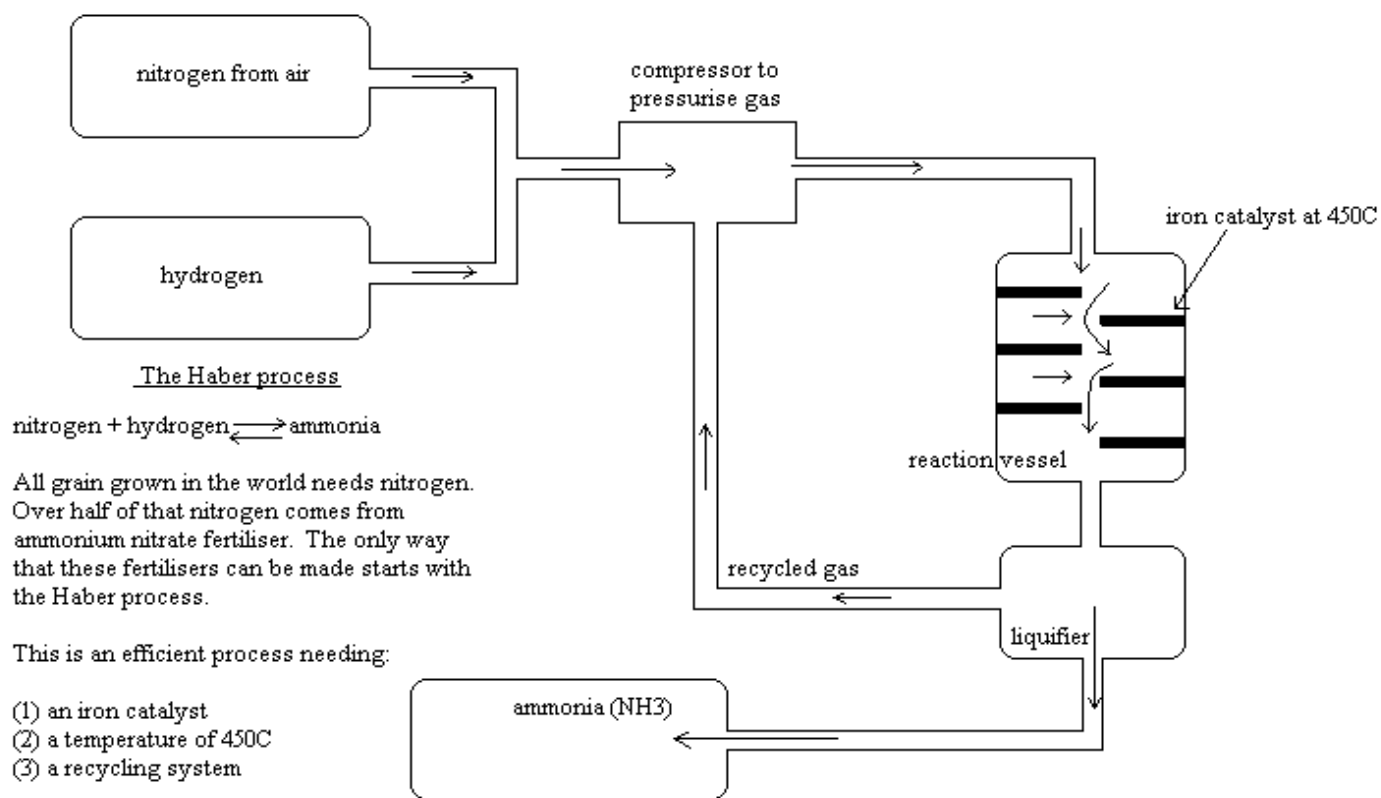


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Inside the pressure vessel the gases pass over a catalyst of finely divided iron. The gas mixture leaving the reaction vessel contains only about 15% ammonia, which is removed, by cooling and condensing – the remaining (unreacted) nitrogen and hydrogen are recycled and pass through the system again.

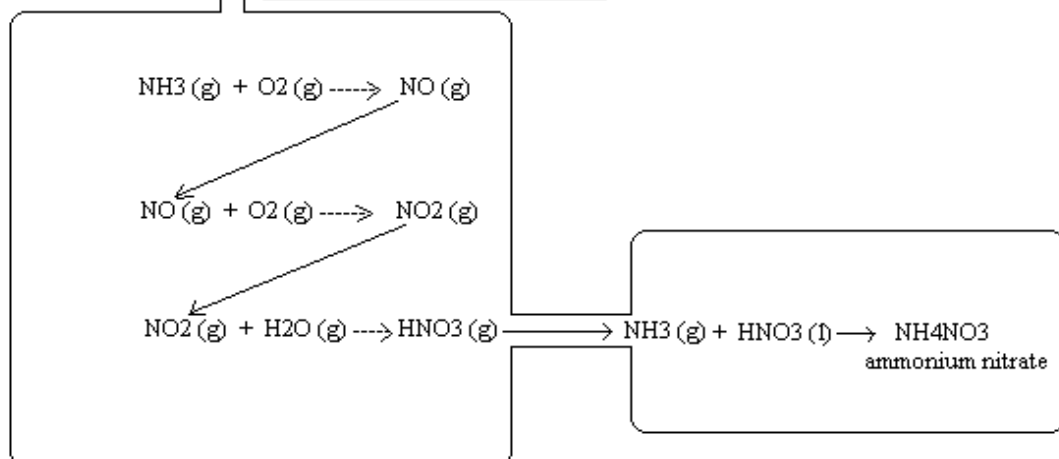
Once ammonia has been made, it can be combined with nitric acid to make ammonium nitrate fertiliser as follows:

The Haber process. Suggest how the compressor helps to make the reaction more efficient.



For the exam, you do not have to know how ammonium nitrate is made

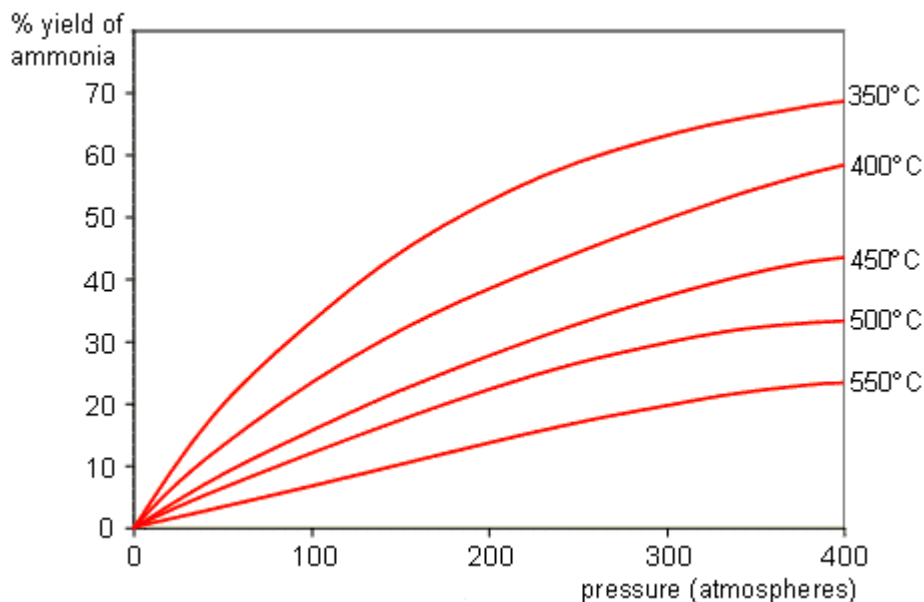
Turning ammonia into nitric acid



Not all the nitrogen and hydrogen react, so the nitrogen and hydrogen that do not react are sent back into the reaction vessel.

**Question:** Write down four important conditions used in the industrial manufacture of ammonia.

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**Question:** Consider the above graph. When the temperature increases, the reactant molecules [hydrogen and nitrogen] have more kinetic energy and so collide into each other more frequently, with sufficient activation energy to produce ammonia more frequently.

[a] How does increasing the temperature affect the rate of the reaction? [Explain]

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[b] How does increasing temperature affect the yield of ammonia produced? [Explain in terms of Le Chatelier's principle].

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[c] With reference to the graph and the last two answers, explain why a compromise temperature of 450C, is often used in the Haber process.

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In the Haber process, the conditions chosen are a **compromise**.

### **Choosing the pressure**

Increasing the **pressure** causes the **yield** to increase. However, the energy costs also increase and the equipment used for the process must be strong to withstand the high pressure, which makes it expensive.

The pressure chosen is a compromise between rate, cost, and safety.

### **Choosing the temperature**

Increasing the temperature decreases the yield but increases the rate. The temperature chosen is a compromise between rate and yield.

### **Selecting the catalyst [green chemistry]**

The **catalyst** can be expensive to develop and to buy - but it does not need to be replaced, because catalysts are not used up or chemically changed in a reaction. Care must be taken to ensure that the catalyst is not poisoned by impurities in the **reactants**.

**The catalyst does not affect the yield, but it does increase the rate of the reaction.** This allows a lower temperature to be used (increasing temperature is one way of speeding up a reaction) and using a lower temperature allows the yield to be maximised as well as saving money on energy costs. The Kellogg Advanced Ammonia Process [KAAP] ruthenium rather than iron which can produce higher yields at lower pressures.

### The environmental impact of the Haber process

The **Haber process** is an artificial way of ‘fixing’ unreactive nitrogen from the air and turning it into useful chemicals that plants can absorb and use when they build **protein** molecules in their cells.

So-called ‘**nitrogen-fixing**’ **bacteria** are able to ‘fix’ nitrogen from the air, at room temperature and pressure, using **enzymes**.

These conditions are much cheaper than the high temperature and pressure used in the Haber process. If scientists could find a way to use these enzymes on an industrial scale, this would save a lot of energy, **fossil fuels** and money.



Plants and animals need nitrogen to make **proteins**. But they cannot get nitrogen directly from the air because, as a gas, nitrogen is fairly unreactive. Plants are able to take up nitrogen **compounds** such as nitrates and ammonium salts from the soil.

**Question:** Complete the text using the words that follow

Making nitrogen compounds from nitrogen in the air is called \_\_\_\_\_ fixation.

Nitrogen \_\_\_\_\_ happens in three ways:

- The energy in lightning splits nitrogen molecules into individual nitrogen atoms. These react with oxygen to form **nitrogen** \_\_\_\_\_. Nitrogen oxides are washed to the ground by rain, where they form nitrates in the soil.
- Nitrogen-fixing bacteria found in the soil and in the root nodules of leguminous plants, such as peas, beans and clover, fix nitrogen gas into **nitrogen compounds**.
- The Haber process is used by industry to produce \_\_\_\_\_ from nitrogen and hydrogen. Ammonia is used to make nitrogen compounds that are used as fertiliser by farmers.

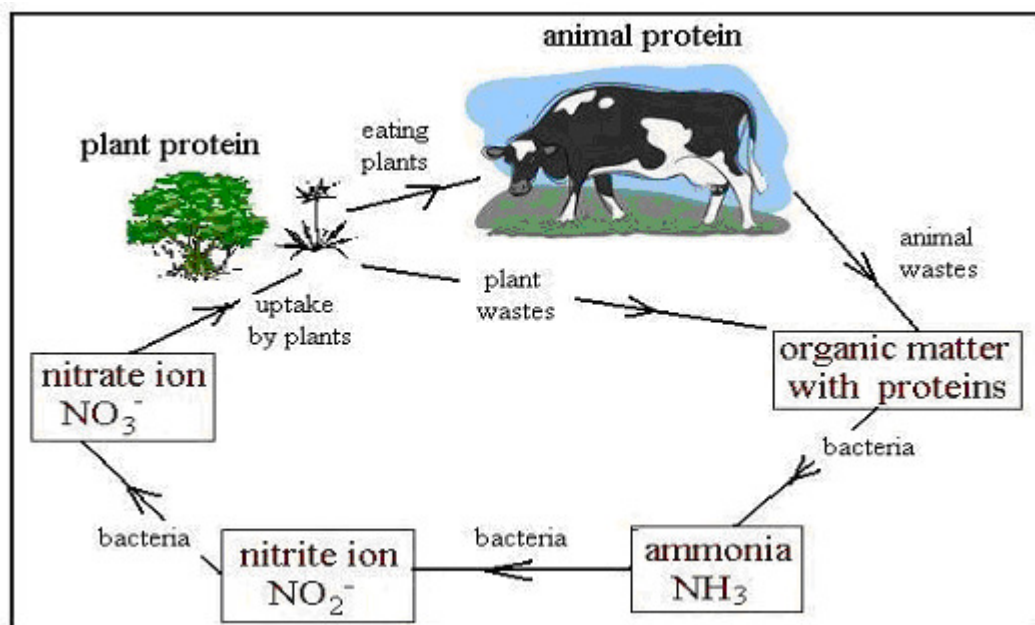
### Nitrogen compounds

Nitrogen compounds in living things are returned to the soil through:

- \_\_\_\_\_ and egestion by animals
- The decay of dead plants and animals

Denitrifying bacteria present in soil break down nitrogen \_\_\_\_\_ and release nitrogen gas into the air.

**Possible words:** nitrogen, oxides, ammonia, fixation, Excretion, compounds



As a result of these processes, nitrogen is cycled continually through the air, soil and living things. This is called 'The Nitrogen Cycle'.

Nitrogen-fixing bacteria are found in the root nodules of leguminous plants

Making ammonia using the Haber process requires a lot of energy, which usually involves burning fossil fuels. This releases carbon dioxide, which causes global warming. Oxides of nitrogen are also emitted during the process.

The widespread use of fertilisers can cause a type of pollution called eutrophication.

This is when fertilisers are washed off fields and into rivers and lakes, causing algae to grow. The algae die off when they have exhausted the nutrients. The algae are then decomposed by aerobic bacteria, which use up all the oxygen - causing the aquatic ecosystem to collapse.

**Question:** Number the following sentences in the correct order:

When this happens it encourages algae to grow on the surface of the pond.

- Fertilisers can be washed into ponds
- Fish and other forms of aquatic life suffocate.
- These pondweeds cannot carry out photosynthesis and so die
- The dead pondweed breaks up and acts as food for bacteria
- This blocks light getting to pondweed at the bottom of the pond

When water plants die and break up in this manner making the water turbid, it is called eutrophication.

The bacteria carry out aerobic respiration using these decaying plant fragments as food. They remove oxygen from the water.

**Question:** Explain why nitrogen fixation is important.

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**Question:** Explain why plants can be short of nitrogen when there is so much nitrogen in the air.

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### Explaining dynamic equilibrium

A **reversible reaction** will reach a state of dynamic equilibrium if it takes place in a sealed container and is left for long enough at constant conditions (eg temperature and **pressure**).

A dynamic equilibrium is when the amount of **products** and **reactants** in the reaction mixture remain constant over time.

When a system is at dynamic equilibrium, it does not mean that the reaction has finished. In fact, both the forward and reverse reactions are still taking place.

Because they are taking place at the same rate (speed), the reactants are turning into products at the same speed as the products are turning back into reactants.

**Note** that the fact that a system is at dynamic equilibrium does not tell us about the **yield**. The yield depends on factors like pressure and temperature.

### An analogy for dynamic equilibrium

A department store opens at 9am on the first day of the January sales. There are a lot of people on the pavement outside, so the speed at which they enter the store through the revolving door is very high.



A department store can be used as an analogy for dynamic equilibrium

A little while later, people are leaving the store. At the point when people are **entering and leaving the store at the same speed** (one in, one out, through the revolving door), there is a **dynamic equilibrium**. This is because the number of people in the store remains constant from one hour to the next, even though the people themselves in the store might be different.

The enzyme called nitrogenase, which is found in N-fixing bacteria that turns atmospheric nitrogen into ammonia, contains clusters of iron, molybdenum and sulphur.

Chemists have been successful in making similar artificial clusters that show catalytic activity. By producing and using new catalysts that mimic natural enzymes, it may be possible in the future to produce ammonia at room temperature and pressure. This would lead to low energy use during production.

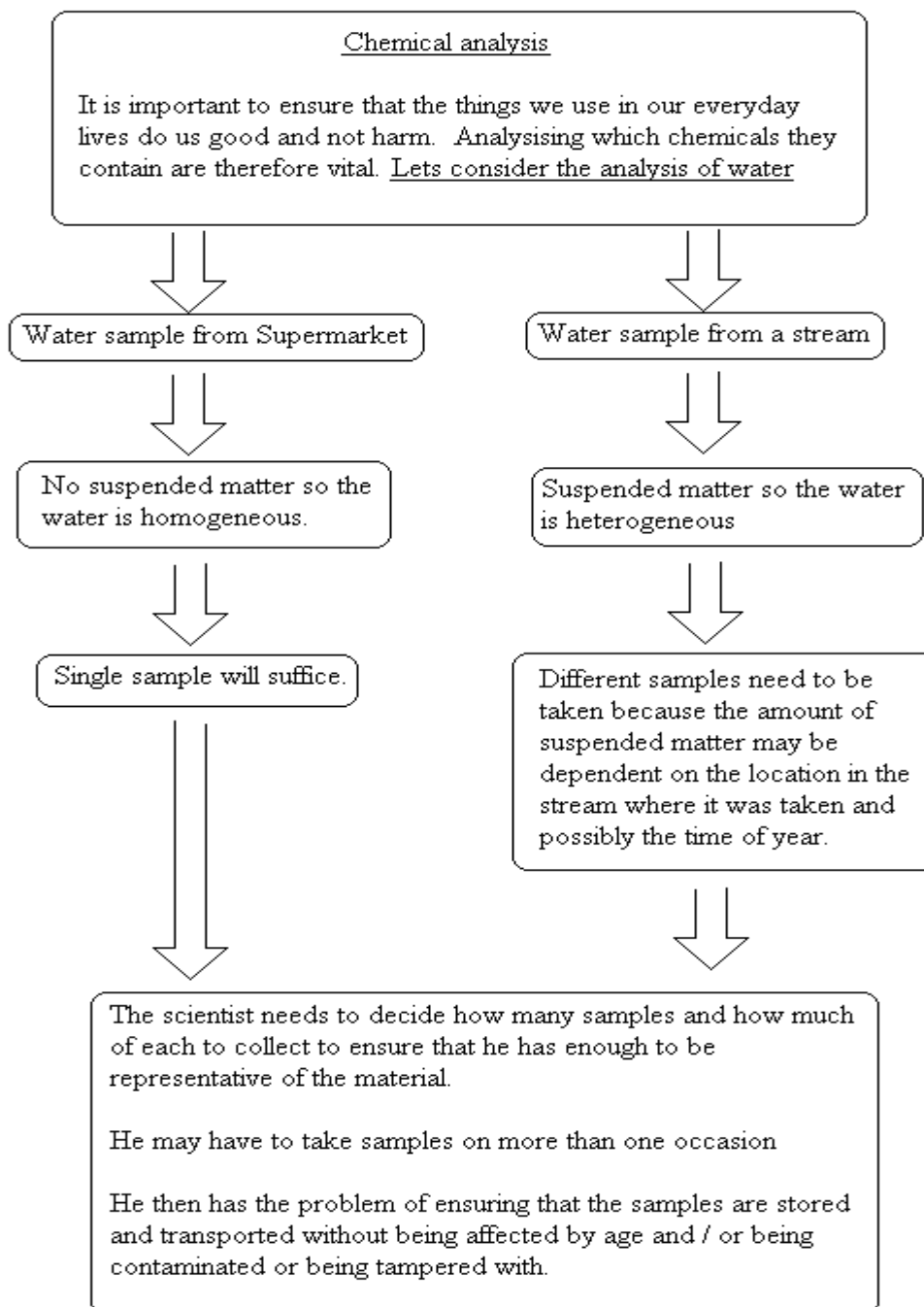
**Question:** Summarise the different ways of producing ammonia in the Haber process in a green manner.

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## Chemical analysis



**Question:** Complete the following text using the words that follow;

There are two types of analysis method. A \_\_\_\_\_ method can be used to find out the chemical composition of the specimen e.g. if it contains a particular type of salt or not [aluminium sulphate]. A \_\_\_\_\_ method can be used to find out how much of that salt is present.

Analysts take a sample and measure out accurately known masses or \_\_\_\_\_ of the material for analysis. It is common to carry out the sample with two or more samples in parallel to check on the \_\_\_\_\_ of the final result. These are \_\_\_\_\_ samples.

Many samples need to be \_\_\_\_\_ in water before they can be analysed.. This is easy with acids and \_\_\_\_\_ but more difficult with minerals, biological specimens or polymers.

Analysts then look for a particular property of the material. For example, we can calculate how much of an acid is present if we know how much alkali is needed to \_\_\_\_\_ it.

We can determine the confidence in the accuracy of the results by comparing them from two or three replicate \_\_\_\_\_.

**Possible words:** samples, neutralise, dissolved, alkalis, replicate, reliability, qualitative, quantitative, volumes

## Chromatography

Chromatography is an important analytical technique because it allows chemists to separate substances in complex mixtures. There are a variety of types of chromatography, which can be used in different contexts. They involve the following types of analysis.

**Qualitative analysis** means any method of identifying the chemicals in a sample.

**Quantitative analysis** means working out how much of a particular chemical there is in a sample.

Any analysis must be carried out on a sample of a substance that is representative of the bulk of the substance. This means that the results from the analysis can be used to draw conclusions about the rest of the substance.

It is important to mix solutions thoroughly before testing, and to take many samples at random from the solution when taking repeat readings.

It is important to follow standard procedures when analysing chemicals. This ensures that the results are reliable and valid.

Standard procedures cover the following:

- Collecting samples
- Storing samples
- Preparing samples for analysis

## Paper chromatography

In chromatography, substances are separated as they travel in a **mobile phase, which** passes through a **stationary phase**. Different substances travel at different speeds, so some move further than others in a given time.

In paper chromatography, the stationary phase is paper. The mobile phase may either be an aqueous (water-based) liquid or a non-aqueous organic (carbon-based) **solvent**. An example of an organic solvent is propanone - which is the main chemical in nail varnish remover.

For each chemical in the sample, there is a **dynamic equilibrium** between the stationary phase and the mobile phase. The overall separation depends upon how strongly attracted the chemicals are to the mobile and the stationary phases.

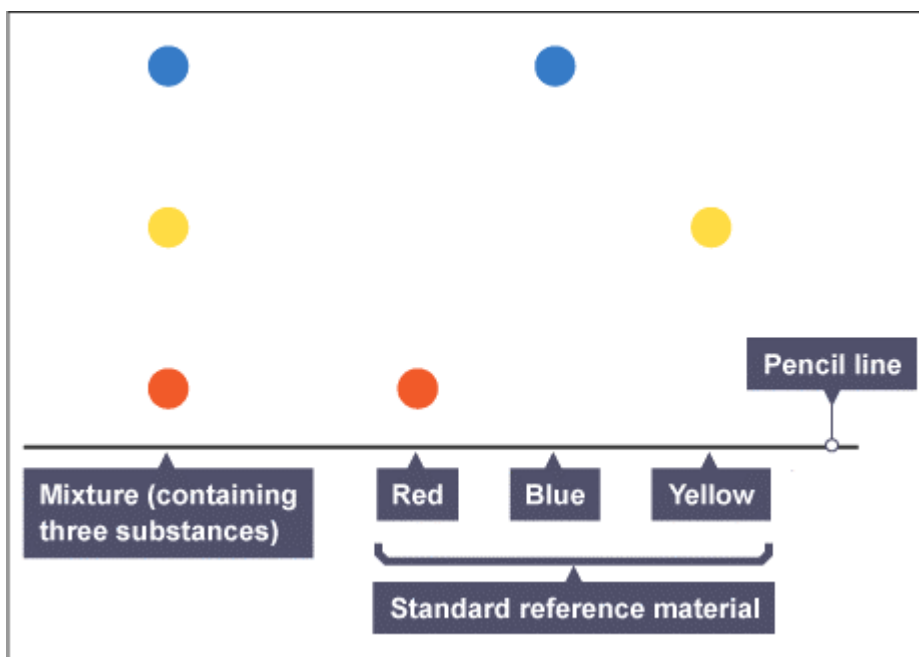
## Thin layer chromatography (TLC)

**Thin layer chromatography (TLC)** is similar to paper chromatography but instead of paper, the stationary phase is a thin layer of an **inert** substance (eg silica) supported on a flat, unreactive surface (eg a glass plate).

TLC has some advantages over paper chromatography. For example:

- The mobile phase moves more quickly through the stationary phase
- The mobile phase moves more evenly through the stationary phase
- There is a range of absorbencies for the stationary phase

TLC tends to produce more useful **chromatograms** than paper chromatography, which show greater separation of the components in the mixture - and are therefore easier to analyse.



### Locating agents

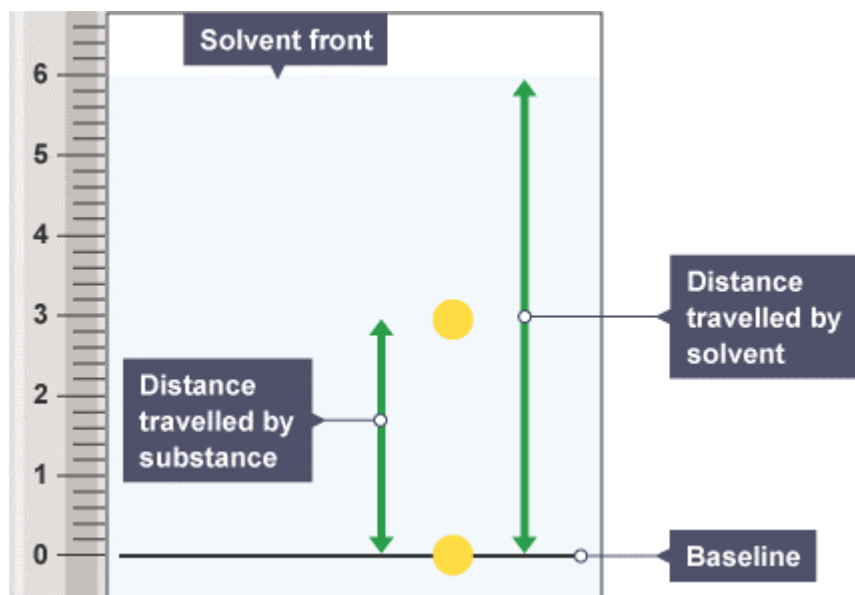
Sometimes the substances being separated are colourless. In this case, **locating agents** can be used to show where the spots are.

Locating agents bind to the chemicals in the spots. Sometimes, another chemical is then added, which reacts with the locating agent to produce a coloured spot, or the chromatogram is put under **ultraviolet** light and the locating agent glows to show where the spots are.

### R<sub>f</sub> value

The movement of a substance during chromatography, relative to the movement of the **solvent**, is measured by calculating its **retardation factor** (R<sub>f</sub>).

One way of identifying the chemicals in a sample is to add separate spots of solutions of substances suspected of being present in the unknown mixture. These are called **reference materials**.



Chromatography measurement

The R<sub>f</sub> value is worked out using this formula:

$$R_f \text{ value} = \frac{\text{distance travelled by substance}}{\text{distance travelled by solvent}}$$

Calculating the  $R_f$  value allows chemists to identify unknown substances because it can be compared with  $R_f$  values of known substances under the same conditions.

**Question:** Why is it sometimes necessary to 'develop' a chromatogram? How can this be done/

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**Question:** Why is it sometimes useful to use thin-layer chromatography plates that have been impregnated with fluorescers?

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**Question:** what are reference materials used for?

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**Question:** Using the chromatogram on this page, calculate the  $R_f$  value of the yellow spot.

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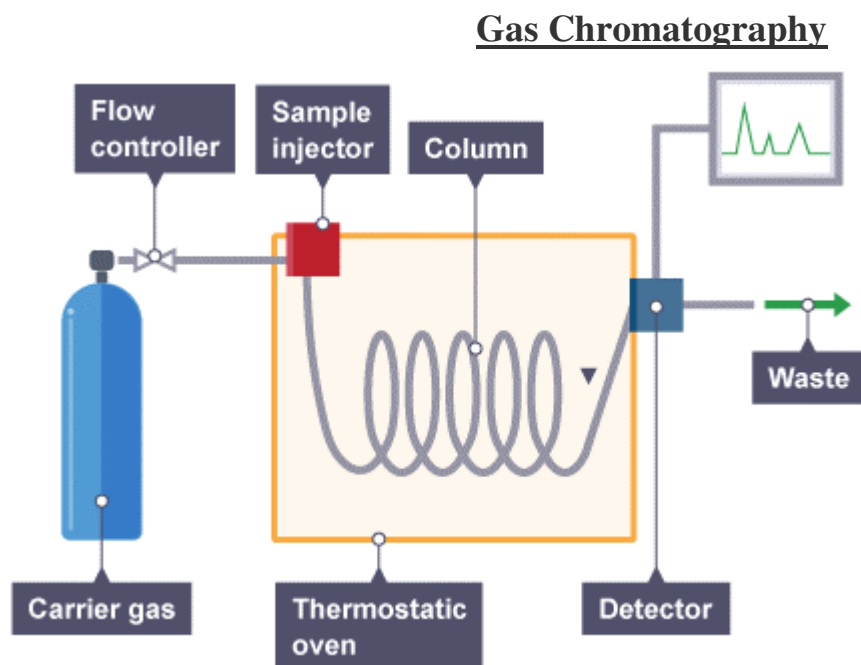
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**Question:** Draw on the chromatogram how the chromatogram separates out the different coloured solutes in an unknown mixture. It comprises of a red compound, which has a higher  $R_f$  value, and a yellow compound of the same  $R_f$  value as the first yellow spot.

**Question:** Explain exactly what  $R_f$  values show.

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Gas chromatography [GC] can be used to separate complex mixtures.

## Advantages to paper chromatography

[1] It is more sensitive than paper chromatography and Thin Layer Chromatography. It can detect smaller quantities. [2] Gas chromatography can detect the quantities of each solute present.

The principles of GC are the same for paper chromatography and TLC. A mobile phase carries a mixture of compounds through a stationary phase. Some compounds are carried through more slowly than others. This is because they have different boiling points or a greater attraction for the stationary phase. Because they travel at different speeds, the compounds can be separated and identified.

The mobile phase is a gas such as helium. This is a carrier gas.

The stationary phase is a thin film of a liquid on the surface of a powdered solid. The stationary phase is packed into a sealed tube, which is a column.

Tiny amounts of a mixture [gases or liquids] are syringed into the column. The column is coiled inside an oven, which controls the temperature of the column. This means that it is possible to analyse solids if they can be injected in solution and then turn to a vapour at the temperature of the column.

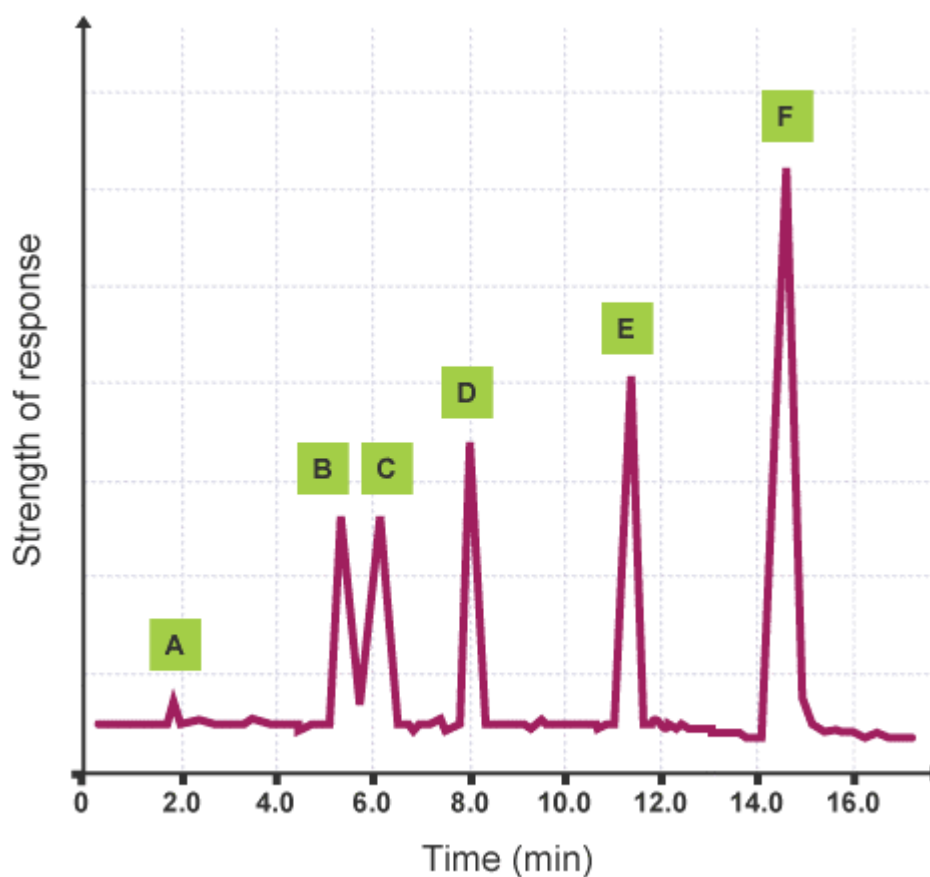
The chemicals in the mixture turn to gases and mix with the carrier gas. The gases emerge at different rates from the column and identified with a detector. A chromatogram is produced.

**Question:** It is necessary to measure the composition of different food colouring dyes in a smartie. Which method of chromatography should be used and why?

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## Gas Chromatograms



This gas chromatogram shows that:

- Substance A was present in the smallest quantity (it has the smallest peak)
- Substance A had the shortest retention time
- Substances B and C were present in equal amounts
- Substance F had the longest retention time [how long it took to pass through the column]
- Substance F was present in the greatest quantity (it has the largest peak)
- Substance F had the greatest affinity for the stationary phase

Question: Look at the chromatogram on the previous page.

[a] How many components have been separated?

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[b] Estimate the retention time of each component.

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[c] Which component was present in the largest quantity? Which one was present in the smallest quantity?

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## Titrations

It is essential for a chemist to be able to accurately measure the amount of a substance present in a sample of a solution. This is often done by titrating it against a standard solution.

### Stages in quantitative analysis

In general, quantitative analysis follows the following steps:

- Accurately measure out a specific **mass** or **volume** of the chemical to be tested.
- If the sample is a solid, **dissolve** it in a known volume of water.
- Measure a property of the solution quantitatively, eg by reacting an unknown **acid** with an **alkali** of known concentration until the solution is **neutral**.
- Analyse the results to calculate the required measurement of the unknown substance (eg concentration of the unknown acid).
- Repeat the measurement using another sample to check that the results are consistent/reliable.
- Estimate the degree of uncertainty in the results.

### Making a standard solution

A **standard solution** is one that has been made by dissolving an accurate mass of **reactant** into a known volume of water. This means that the concentration of the standard solution is accurate - so it can be used to work out the concentration of another solution that it reacts with.

Concentration is measured in  $\text{g/dm}^3$  [remember concentration ( $\text{g/dm}^3 = \text{mass (g)} / \text{volume (cm}^3)$ )]

To make  $250 \text{ cm}^3$  of a standard solution with a concentration of  $100 \text{ g/dm}^3$ , you need to:

- Accurately measure 25.0 g of the solid sample in a beaker.
- Add  $150 \text{ cm}^3$  of distilled water and stir the mixture using a glass rod until the solid has completely dissolved.
- Use a funnel to transfer the contents of the beaker into a  $250 \text{ cm}^3$  volumetric flask.
- Rinse the beaker and glass rod thoroughly with **distilled water** and pour the rinsings into the volumetric flask.
- Carefully add distilled water to the volumetric flask until the bottom of the **meniscus** is level with the horizontal line on the neck of the flask.
- Holding the stopper into the neck of the flask, carefully turn the flask upside down several times to thoroughly mix the solution.

**Question:** What is the concentration of these solutions in grams per litre [ $\text{g/dm}^3$ ]

[a] A solution of sodium carbonate made by dissolving 4.0g of the solid in water and making the volume up to  $500 \text{ cm}^3$  in a graduated flask?

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[b] A solution of citric acid made by dissolving 2.25g of the water in water and making the volume up to  $250 \text{ cm}^3$  in a graduated flask? [Ensure all solution is washed into volumetric flask with water].

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**Question:** What is the mass of solute in these samples of solution?

[a] A  $10\text{cm}^3$  sample of a solution of silver nitrate with a concentration of  $2.55\text{ g/dm}^3$

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[b] A  $25\text{cm}^3$  sample of a solution of sodium hydroxide with a concentration of  $4.40\text{ g/dm}^3$

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### Performing a titration

To work out the concentration of an acid, it can be titrated against a standard solution of an alkali.

Follow these steps:

- Fill a burette with the standard alkali solution. Run a little through the tap to make sure that no air is trapped. The zero line is at the top of the burette. Ensure that the bottom of the meniscus is level with the zero line.
- Use a pipette to measure exactly  $25.0\text{ cm}^3$  of the unknown acid and place this into a conical flask.
- Add two drops of a suitable indicator (eg phenolphthalein or methyl orange). The indicator will show its acidic colour.
- Open the tap on the burette to start adding the alkali to the conical flask. Swirl the mixture in the conical flask continuously to mix it.
- As the colour of the indicator starts to change, slow down the flow of alkali from the burette so that it is dripping into the conical flask one drop at a time.



- When the indicator changes colour permanently, the end point has been reached.
- Record the volume of alkali used to **neutralise** the acid.
- Rinse out the conical flask, top up the alkali in the burette and then repeat steps 2 to 7 to ensure that the results are reliable.

A similar procedure can be used to work out the concentration of an alkali.



This diagram shows a student using a pipette and pipette filler to transfer the acid to the conical flask. The burette is held in the clamp and contains the alkali

### Titrations

Titration reactions can be used to measure the concentration of an acid.

A neutral solution has equal numbers of hydrogen ions to hydroxide ions (OH) dissolved in water. We say that these solutions are pH 7.

An acidic solution has more hydrogen ions than hydroxide ions dissolved in water. The lower the pH number below 7 the more hydrogen ions there are relative to hydroxide ions (stronger acid) e.g. pH 1 = strong acid.

An alkaline solution has more hydroxide ions than hydrogen ions dissolved in water. The higher the pH number above 7 the more hydroxide ions there are relative to hydrogen ions (stronger alkali) e.g. pH 14 = strong alkali. The name of alkalis usually starts with a metal and ends with 'hydroxide'.

**Question:** Complete the following table:

Name	pH	Acid or alkali	Strong or weak
Sodium hydroxide	13		
Citric acid	2		
Lithium hydroxide	14		
Calcium hydroxide	10		
Hydrochloric acid	1		
Sulphuric acid	3		
Nitric acid	5		
Phosphoric acid	6		
Potassium hydroxide	12		
Phosphoric acid	1		
Acetic acid	4		

Universal Indicator can be used to determine the pH number of a solution. A few drops are added to the test solution and then the colour of the solution is compared to a standard colour chart.

**Question:** On the above table, next to each pH number, use a coloured pencil to show what colour G.U.I would turn in that solution.

### Neutralisation

When an acid (H<sup>+</sup>) is added to an alkali (OH<sup>-</sup>), the more the numbers of each become equal, the more a neutral solution is produced. We say that the acid (and the alkali) are becoming neutralised.

The H<sup>+</sup> ions rejoin with the OH<sup>-</sup> ions and reform water H<sub>2</sub>O.

Sodium hydroxide (aq) + hydrochloric acid (aq) = sodium chloride (aq) + water (l)

- Acids have low pH numbers
- Adding acid to a solution decreases its pH
- Adding alkali to a solution increases its pH

### Measuring pH

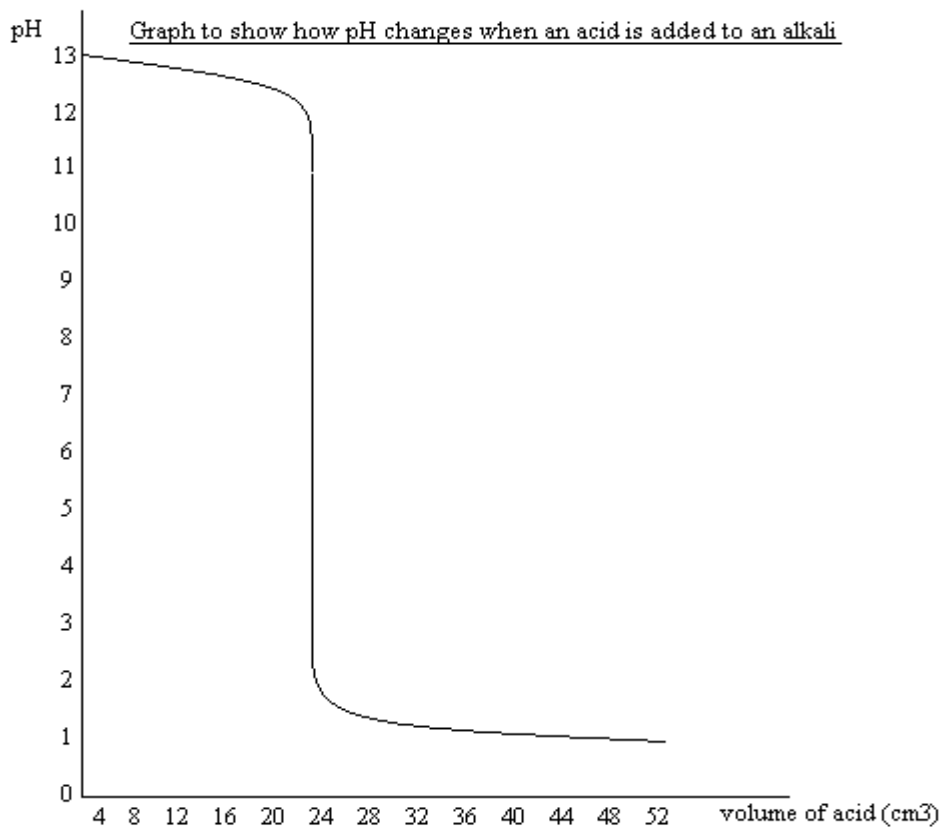
A pH meter is used to measure the pH of a solution. Universal indicator solutions and litmus paper are good for a quick estimate. Different brands of Universal indicator are slightly different, so the colour must always be compared with the reference card.

pH	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Colour	RED	ORANGE	YELLOW	GREEN	BLUE	PURPLE-VIOLET								
strength	Strong	ACIDS		Weak	Neutral	Weak	ALKALIS						Strong	

### What happens to pH when an acid reacts with an alkali?

- At the start of the experiment there is just alkali (lots of OH<sup>-</sup> / hydroxide ions compared to H<sup>+</sup> hydrogen ions). As a result the pH number is high
- As acid is added, the H<sup>+</sup> ions join with OH<sup>-</sup> ions forming water. We say that the alkali (OH<sup>-</sup>) is being neutralised. The pH number falls.
- The point where all the acid reacts with all the alkali is called the **end point**.

At the end point, pH changes very suddenly.



**Question:** Label the graph on page 18 with the following text:

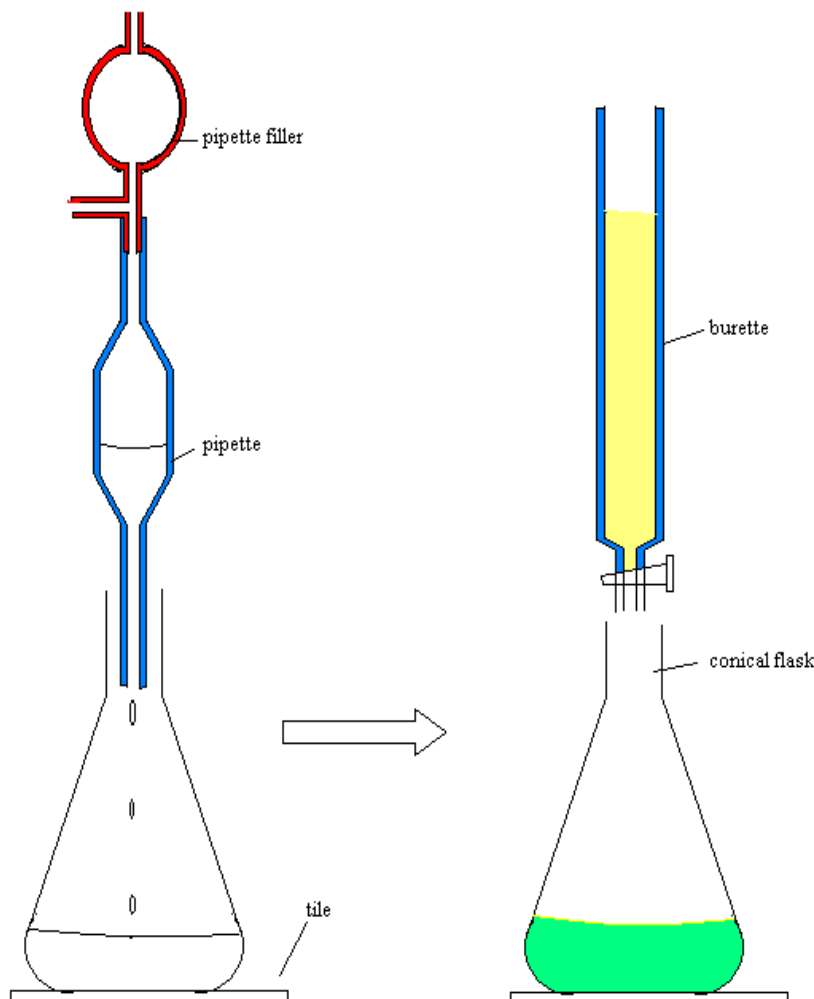
- (a) alkali, pH high
- (b) acid has reacted with some of the alkali. pH falls.
- (c) all the alkali is neutralised, pH = 7
- (d) excess acid. pH is below 7.
- (e) this volume of acid = end point (amount needed to neutralise all the acid)

**Question:** Use the graph to estimate the pH after 16cm<sup>3</sup> of acid is added.

**Question:** How much acid produces a pH of 2?

## Titration

Every time there is a new type of chemical problem to solve, a titration is the first thing scientists think of.



### Carrying out a titration

- (1) Wear safety goggles. Pipette the alkali into a conical flask using a pipette filler.
- (2) Put a few drops of indicator into the flask.
- (3) Fill a burette with acid.
- (4) Add the acid slowly to the alkali, mixing the solution in the conical flask by swirling the flask gently. This ensures that all  $H^+$  ions have a chance to come into contact with  $OH^-$  ions.
- (5) Stop adding the acid when the indicator suddenly changes colour - this is the end point.
- (6) Repeat steps 1 to 5 until the readings are the same.

### Titration results

	Volume of acid reacting with 25cm <sup>3</sup> of alkali (rough)	Volume of acid reacting with 25cm <sup>3</sup> of alkali (first)	Volume of acid reacting with 25cm <sup>3</sup> of alkali (second)	Volume of acid reacting with 25cm <sup>3</sup> of alkali (third)
At start	1.2	3.2	2.5	3.7
At end	28.2	29.6	29.0	30.1
Titre	27.0			

### What are the colours of different indicators?

Indicator	Colour in alkali	Colour in acid
Litmus	blue	red
Phenolphthalein	pink	colourless
Screened methyl orange	green	pink

## Universal indicator – a special case

Universal indicator is a mixture of indicators. It is:

- Blue in alkali
- Green at pH = 7
- Red in acid

**Question:** Look at the previous table showing titration results. Work out how much acid is needed for each of the three accurate titrations.

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**Question:** Give examples when indicators turn pink.

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### Deciding which indicator to use

(1) Indicators such as phenolphthalein, screened methyl orange and litmus give a sudden colour change. This makes the end point very easy to spot.

(2) Mixed indicators such as Universal indicator are not used in titrations. They are given a continuous colour change so it is much harder to see the end point.

### Why repeat a titration?

(1) Titration is an accurate technique but there is a chance of experimental error. If there are large differences in the readings, then it is a warning that the technique is not accurate.

(2) If the readings are close, the technique is reliable.

Small differences are due to normal experimental error, so an average reading is calculated.

**Question:** Choose the indicators from the list below that are the best for titrations.

- (1) Phenolphthalein
  - (2) Universal indicator
  - (3) Screened methyl orange
- 
- 

### Analysing titration results

A **titration** should be repeated several times to ensure that the results are reliable (consistent). These results should then be averaged.

For example, if the volume of 30 g/dm<sup>3</sup> sodium hydroxide (NaOH) that is required to neutralise 25.0 cm<sup>3</sup> of hydrochloric acid (HCl) was recorded as 24.2 cm<sup>3</sup>, 24.1 cm<sup>3</sup> and 24.3 cm<sup>3</sup>, the average can be calculated as follows:

$$\text{mean volume of NaOH} = \frac{24.2 + 24.1 + 24.3}{3}$$

$$= 24.2 \text{ cm}^3$$

This can then be substituted into a given formula in order to work out the concentration in g/dm<sup>3</sup> of the HCl:

$$\text{concentration of acid (g/dm}^3\text{)} = \frac{\text{volume of NaOH (dm}^3\text{)} \times \text{concentration of NaOH (g/dm}^3\text{)} \times 0.9125}{\text{volume of acid (dm}^3\text{)}}$$

$$\text{Therefore: Concentration of HCl (g/dm}^3\text{)} = \frac{24.2 \times 30 \times 0.9125}{25}$$

$$= 26.5 \text{ g/dm}^3 \text{ (rounded to nearest 0.1 g/dm}^3\text{)}$$

**Question:** Carry out the same technique on these results to calculate the concentration of the acid. The concentration of the alkali is again 30g/dm<sup>3</sup> sodium hydroxide (NaOH) and the alkali neutralised 25.0 cm<sup>3</sup> of hydrochloric acid (HCl).

	Volume of alkali reacting with 25cm <sup>3</sup> of acid (rough)	Volume of alkali reacting with 25cm <sup>3</sup> of acid (first)	Volume of alkali reacting with 25cm <sup>3</sup> of acid (second)	Volume of alkali reacting with 25cm <sup>3</sup> of acid (third)
At start	1.2	3.2	2.5	3.7
At end	28.2	29.6	29.0	30.1
Titre	27.0			

### Evaluating titration results

When evaluating experimental results, it is important to consider the **accuracy**, **precision** and **validity** of the measurements.

- Accuracy describes how close a result is to the true value.
- Precision is a measure of the spread of the measured values. If there is a big spread, the uncertainty is large.
- If an experiment has a good degree of accuracy **and** the results have a small uncertainty, and the experiment is not flawed in any other way (eg an unfair test), then the results are valid.

### Working out the uncertainty

We can work out the uncertainty using a series of **titration** results: 26.5 g/dm<sup>3</sup>, 25.9 g/dm<sup>3</sup>, 26.2 g/dm<sup>3</sup>, 26.8 g/dm<sup>3</sup>, and 27.1 g/dm<sup>3</sup>.

The average is calculated by adding the values together and dividing by the number of values (five in this case).

$$\text{Average} = \frac{26.5 + 25.9 + 26.2 + 26.8 + 27.1}{5}$$

$$\text{Average} = 26.5 \text{ g/dm}^3$$

The range is between 25.9 g/dm<sup>3</sup> and 27.1 g/dm<sup>3</sup>. Therefore, the uncertainty is 1.2 g/dm<sup>3</sup>.

The percentage error is calculated by dividing the uncertainty by the average Then multiply by 100:

$$\text{Percentage error} = \frac{1.2}{26.5} \times 100, \text{ therefore the Percentage error} = 4.5\%$$

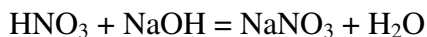
$$\text{concentration (g/dm}^3\text{)} = \frac{\text{mass (g)} \times 1000}{\text{volume (cm}^3\text{)}}$$

## Titration analysis – Higher tier

Titration results can be analysed using a balanced symbol equation and relative formula masses (RFMs).

For example, calculate the concentration of nitric acid (HNO<sub>3</sub>) if 25.0 cm<sup>3</sup> of it is exactly neutralised by 30.0 cm<sup>3</sup> of sodium hydroxide (NaOH), which has a concentration of 20 g/dm<sup>3</sup>.

First, write a balanced symbol equation:



Then work out the RFMs of the acid and alkali:



This means that 63 g of HNO<sub>3</sub> is neutralised by 40 g of NaOH.

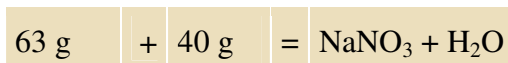
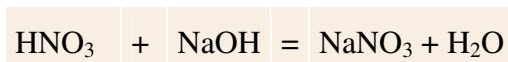
Work out the mass of NaOH in the solution:

$$\text{mass (g)} = \frac{\text{concentration (g/dm}^3\text{)} \times \text{volume (cm}^3\text{)}}{1000}$$

$$\text{Mass} = \frac{20 \times 30}{1000}$$

$$\text{Mass} = 0.6 \text{ g}$$

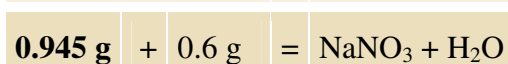
Now manipulate the ratio of the RFMs:



(divide by 40)



(multiply by 0.6)



**So, there was 0.945 g of HNO<sub>3</sub> in 25.0 cm<sup>3</sup>.**

$$\text{concentration (g/dm}^3\text{)} = \frac{\text{mass (g)} \times 1000}{\text{volume (cm}^3\text{)}}$$

$$\text{Concentration} = \frac{0.945 \times 1000}{25.0}$$

$$\text{Concentration} = \mathbf{37.8 \text{ g/dm}^3}$$

**Question:** Calculate the concentration of nitric acid (HNO<sub>3</sub>) if 20.0 cm<sup>3</sup> of it is exactly neutralised by 30.0 cm<sup>3</sup> of sodium hydroxide (NaOH), which has a concentration of 20 g/dm<sup>3</sup>.

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