- Sodium chloride shows only a very small increase in solubility as the temperature is increased.
- The increase in solubility with temperature is usually a smooth curve or an almost straight line.

Calculations using solubility curves

The solubility curve for potassium nitrate is shown in Figure 2.4.2. We can use information from this curve to deduce:

- the temperature at which crystals start to form when a saturated solution is cooled
- the mass of solid deposited when a saturated solution is cooled.

1 At what temperature does a saturated solution containing 140 g potassium nitrate first form crystals as it is cooled from 80 °C?

- Draw a line XY from 140 g on the vertical axis to join the curve.
- The line YZ gives the temperature at which potassium nitrate is just soluble. So 70 °C is the maximum temperature at which crystals will start to form.

2 Calculate the mass of potassium nitrate that would come out of solution when a saturated solution of potassium nitrate is cooled from 80 °C to 40 °C.

- At 80 °C a mass of 170 g of potassium nitrate dissolves (lines ABC).
- At 40 °C a mass of 65 g of potassium nitrate dissolves (lines DEF).
- On cooling from 80 °C to 40 °C the mass of potassium nitrate that comes out of solution is (170 g – 65 g) = 105 g.

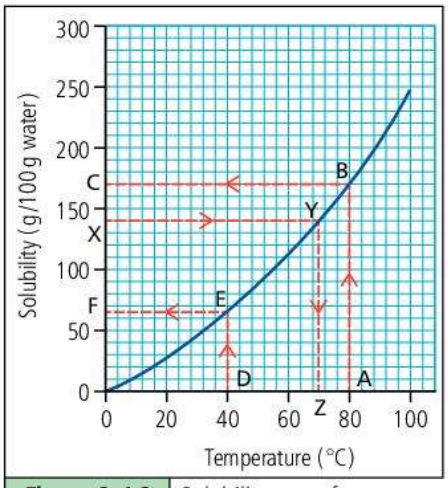


Figure 2.4.2 Solubility curve for potassium nitrate

Fractional crystallisation

Fractional crystallisation can be used to separate two dissolved substances which have different solubilities at different temperatures. A warm concentrated solution containing the two solutes is cooled. A larger proportion of the solute with the lower solubility crystallises out. The solute with the higher solubility remains in solution. The crystals still contain a small amount of the solute with higher solubility. So the procedure is repeated several times to improve the purity. Filtration is used to separate the crystals from the solution.

EXAM TIP

If you are asked to draw a solubility curve or similar curves from data provided, make sure that you draw the curve of best fit and do not draw straight lines from one point to another.

KEY POINTS

1. The solubility of a solute in a solvent is the number of grams of solute needed to form a saturated solution per 100 grams of solvent used.
2. The solubility of most substances increases with temperature
3. The information from solubility curves can be used to calculate the mass of solid deposited when a saturated solution is cooled.

2.5 Filtration, crystallisation and distillation

LEARNING OUTCOMES

At the end of this topic you should be able to:

- describe filtration, crystallisation and distillation
- explain how filtration and distillation work
- explain fractional distillation in terms of differences of particle size and boiling points of the components.

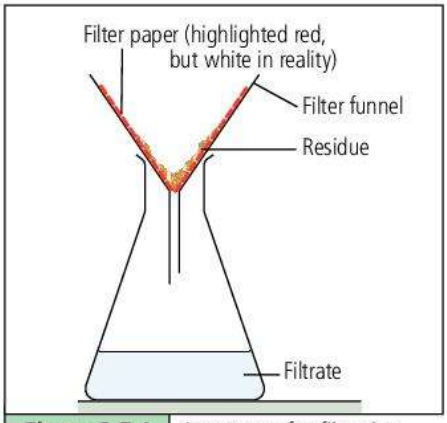


Figure 2.5.1 Apparatus for filtration

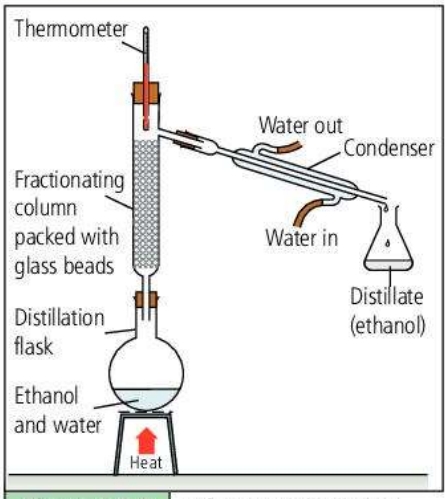


Figure 2.5.3 In fractional distillation the components move up the column at different rates, which depend on their boiling point and the temperature of the column.

Filtration

Filtration separates undissolved solids from a solution or liquid (see Figure 2.5.1). The spaces in the filter paper are very small, so the tiny particles in solution can flow through but the particles in the solid are too large to flow through. They get trapped on the filter paper.

- The solution passing through the filter paper is called the **filtrate**.
- The solid remaining on the filter paper is called the **residue**.
- The solid is washed with a suitable solvent to remove traces of solution.

Crystallisation

We obtain crystals from a solution by the following method (see also Figure 2.5.2):

1. Gently heat the solution in an evaporating basin to concentrate it.
2. Evaporate the solvent until a saturated solution (crystallisation point) is reached. This is reached when a drop of the solution forms crystals when placed on a cold tile.
3. Leave the saturated solution to cool and form crystals.
4. Filter off the crystals and dry them between filter papers.

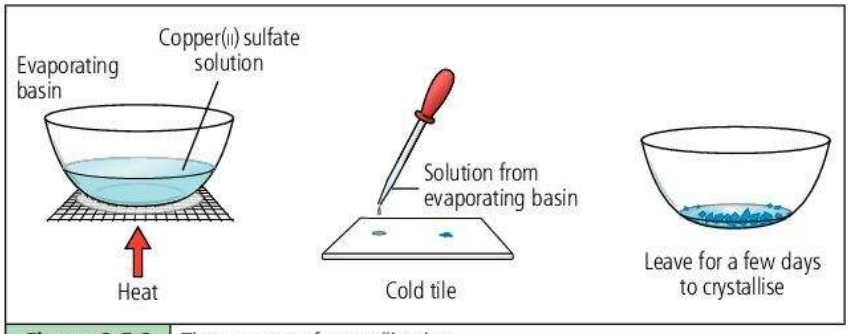


Figure 2.5.2 The process of crystallisation

Simple distillation

Simple distillation is used to separate a liquid from a solid, e.g. to separate salt and water from a solution of sodium chloride. Distillation involves the processes of boiling and condensation. It works because the components to be separated have very different boiling points.

The procedure for separating water from salt by simple distillation is as follows (see also Figure 2.5.3):

1. Heat the solution of salt in water in a distillation flask.
2. The water boils first because it has a much lower boiling point than salt.

3 The steam goes into the condenser.

4 The condenser is at a lower temperature than the boiling point of water, so the water condenses here, and is collected in a flask.

5 The salt remains in the distillation flask because it has a much higher boiling point than water.

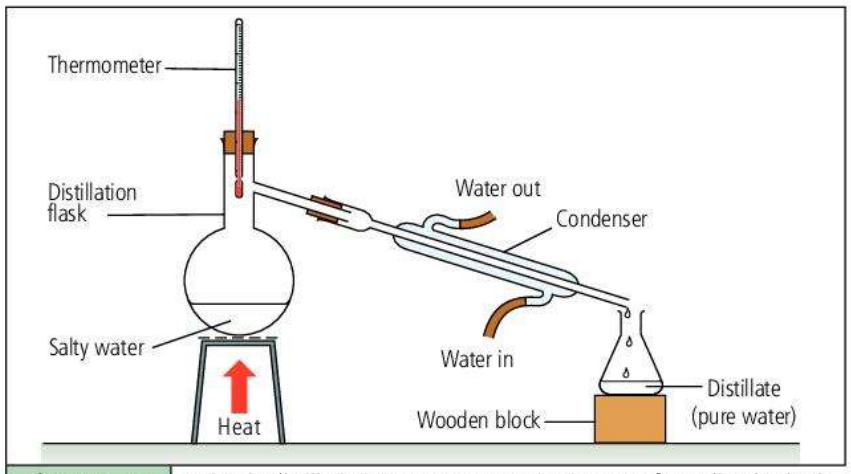


Figure 2.5.4 A simple distillation apparatus to separate water from dissolved salt

Fractional distillation

Fractional distillation is used to separate two or more liquids with different boiling points from each other (Figure 2.5.5). This method is used to separate petroleum fractions (see 12.4) and to purify alcohol from a mixture of water and alcohol.

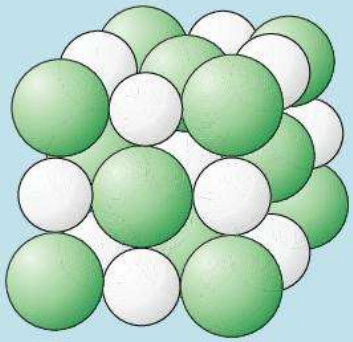
- The more volatile components in the liquid mixture (the ones with the lower boiling points), boil first, so as they move up the column, the vapour contains more of the more volatile component.
- There is a gradient of temperature in the column, which is hotter at the bottom than at the top.
- More of the less volatile components (the ones with higher boiling points) condense lower down because they have higher boiling points, so the more volatile components move further up the column.
- As the mixture is heated more and more, the vapours move up the column. The ones with lower boiling points move ahead of those with higher boiling points.
- The components of the mixture reach the condenser in turn. They change from vapour to liquid in the condenser and the fractions containing particular components of the mixture are collected one at a time.

The boiling point and therefore the distance moved up the column depends on:

- the size of the molecules – smaller molecules of similar types tend to have lower boiling points than larger molecules and so move further up the column than larger ones
- the mass of the molecules – lighter molecules of similar types tend to have lower boiling points and so move further up the column than heavier ones.

DID YOU KNOW?

In crystals, the atoms or ions are arranged regularly in a lattice structure. In some crystals, water molecules form a part of the structure. This is called water of crystallisation.



A lattice

KEY POINTS

1 In filtration, the filtrate passes through the filter paper and the residue remains on the filter paper.

2 Crystallisation is carried out by heating a saturated solution and then leaving the solvent to evaporate.

3 Distillation is a process involving boiling and condensing.

4 The separation of substances by distillation depends on the differences in boiling points of the components in the mixture.

2.6 Extracting sucrose

LEARNING OUTCOMES

At the end of this topic you should be able to:

- describe the extraction of sucrose from sugar cane
- explain the importance of crushing, filtration, precipitation, treatment under vacuum crystallisation and centrifugation in the extraction of sucrose.

Laboratory versus industrial extraction

The apparatus used in the laboratory for extracting compounds or making new compounds is often different from that used when the compound is extracted or made on an industrial scale. The principles, however, are the same. It is difficult to extract sugar from sugar cane in the laboratory by grinding, filtration and evaporation for the following reasons:

- Considerable force is needed to extract the juice from the fibrous sugar cane.
- Ordinary laboratory filters get blocked and it would take too long to filter the sticky filtrate.
- Simple evaporation leaves a treacle-like substance with few crystals and this easily chars on overheating.

Sugar from sugar cane



Figure 2.6.1 Sugar cane plants

Extraction of sugar cane juice

1. Cane sugar stalks are harvested and sent to the factory.
2. The stalks are cleaned by jets of water to remove soil, and the leaves are stripped off.
3. A shredding machine cuts up the stalks into small chips.
4. The chips are crushed and rolled flat under a spray of hot water.
5. The sugar cane juice extracted from the crushing and rolling is a slightly acidic green suspension. The fibrous material (bagasse) is removed.
6. Calcium hydroxide is added to neutralise the acidity and remove some of the impurities. The impurities form a sediment. This process is called clarification.

Filtration

Non-sugar impurities are removed by a continuous filtration process in special industrial filters. This leaves a syrup containing about 85% water.

Evaporation (boiling)

The syrupy sugar extract is passed successively into three or four boilers (evaporators) in order to concentrate it. In each successive boiler the pressure is decreased. This is to prevent the sugar from getting charred. The concentrated juice then contains about 35% water.

DID YOU KNOW?

Sugar cane originally came from south-east Asia. It was first brought to the Caribbean by Christopher Columbus in the 1490s, but it did not become important in the Caribbean economy until the Dutch introduced it from Brazil in about 1625. In the following century, sugar cane rapidly replaced cotton and tobacco as the major crop produced in the Caribbean.

Crystallisation

1 This takes place under vacuum in a crystallisation pan, where the syrup is evaporated until it is saturated with sugar.

2 A few crystals of sugar are added to help more sugar crystals to form.

3 The mixture then contains a thick syrup called molasses together with sugar crystals.

Centrifugation and drying

A centrifuge is a machine that spins round and round at very high speeds. The force of the spinning pulls heavier particles downwards and separates them from lighter particles. This process is called **centrifugation**.

1 A basket centrifuge (Figure 2.6.2) is used to separate the molasses from the sugar crystals. The mixture is placed in the perforated basket (a basket with tiny holes in it). This is spun round at a high speed.

2 The sugar remains in the basket. The molasses are forced out through the holes in the basket.

3 The sugar crystals are dried in air using a type of tumble-drier.

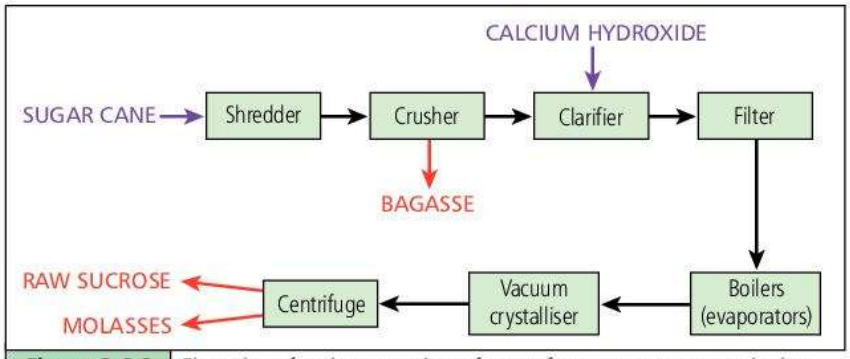


Figure 2.6.3 Flow chart for the extraction of sugar from sugar cane – only the main stages are shown.

KEY POINTS

1 The main stages in sucrose extraction are filtration, precipitation, treatment under vacuum, crystallisation and centrifugation.

2 Calcium hydroxide is used to neutralise the juice extracted from the sugar cane and to precipitate impurities.

3 The sugar solution is concentrated by evaporation under reduced pressure.

4 Crystallisation is done under vacuum.

5 Sugar crystals are separated from molasses by centrifugation.

DID YOU KNOW?

A single worker can harvest as much as 5 tonnes of sugar cane in a day but machines developed to harvest sugar cane can crop 300 tonnes a day.

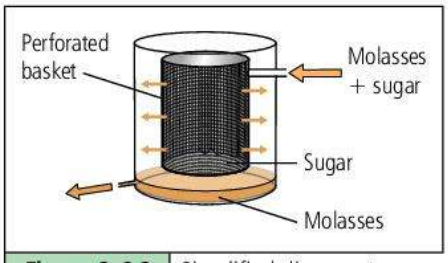


Figure 2.6.2 Simplified diagram to show the idea behind a basket centrifuge

EXAM TIP

When revising sugar extraction concentrate on the most important points which are relevant to separation techniques: filtration, precipitation, treatment under vacuum, crystallisation and centrifugation.

2.7 Chromatography and use of a separating funnel

LEARNING OUTCOMES

At the end of this topic you should be able to:

- describe chromatography
- describe the use of a separating funnel
- explain how chromatography and the use of a separating funnel depend on the differences in solubility of the components in the solvents used.

Paper chromatography

Paper chromatography is used to separate a mixture of different dissolved substances. The method is often used to separate the different pigments (coloured compounds) present in food colourings and inks. Figure 2.7.1 shows how to carry out paper chromatography of a mixture of coloured dyes.

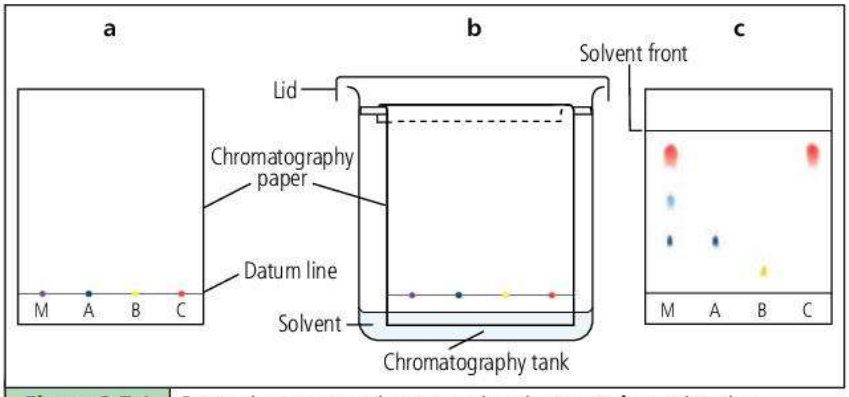


Figure 2.7.1 Paper chromatography: **a** spotting the paper, **b** starting the chromatography, **c** the finished chromatogram

EXAM TIPS

- You must draw the datum line in pencil because the graphite in the lead does not dissolve in the solvent. If you use ink, the different colours in the ink would move up the paper!
- You must make sure that the solvent level is below the datum line. Otherwise the components would wash off into the solvent and would not separate.

1. Draw a pencil line (the datum line) on a piece of chromatography paper (a type of fine-grained filter paper).
2. Put a spot of concentrated dye mixture, M, on the datum line using a very fine glass tube (capillary tube).
3. Small spots of pure substances, e.g. A, B and C, that you think are in the dye mixture, can also be placed on the datum line for comparison.
4. A chromatography tank is set up with the solvent level below the datum line.
5. As the solvent moves up the paper, the dyes separate.
6. The paper is removed when the solvent front is near the top.

The spots on the chromatogram can be compared with those of known dyes. For example, in Figure 2.7.1(c), the mixture, M, contains dyes A and C as well as a third dye.

We can identify the components in the mixture because, for a particular solvent, they travel a certain distance compared with the solvent front.

How chromatography works

The separation of the components in the mixture depends on the solvent or mixture of solvents used.

- With a single solvent, the separation of the components in a mixture depends on the solubility of the component in the solvent and the attraction of the component to the paper. Components that are attracted more to the paper move more slowly up the paper.
- In a mixture of two solvents, one solvent is attracted to the paper more strongly than the other. If a component in the dye is more soluble in the solvent that is more strongly attracted to the paper, it will not move as fast during chromatography. If a component in the dye is more soluble in the solvent that is less strongly attracted to the paper, it will move faster during chromatography.

Using a separating funnel

A **separating funnel** is used to separate immiscible liquids that have different densities. Immiscible liquids are liquids that do not mix. Figure 2.7.2 shows how we use a separating funnel.

We can use a separating funnel to separate two solutes dissolved in a solvent. An example is a solution of iodine and potassium iodide in water (see Figure 2.7.3). Iodine is more soluble in hexane than in water, but potassium iodide is more soluble in water than in hexane. We can separate the iodine from the potassium iodide in the following way:

1. Put the solution of iodine and potassium iodide in a separating funnel.
2. Add hexane to the separating funnel (hexane is immiscible with water).
3. Shake the contents of the funnel to mix the solutions.
4. Most of the iodine moves to the hexane layer and the potassium iodide remains in the water.
5. The layer of potassium iodide in water is run off, leaving the iodine in the hexane layer.

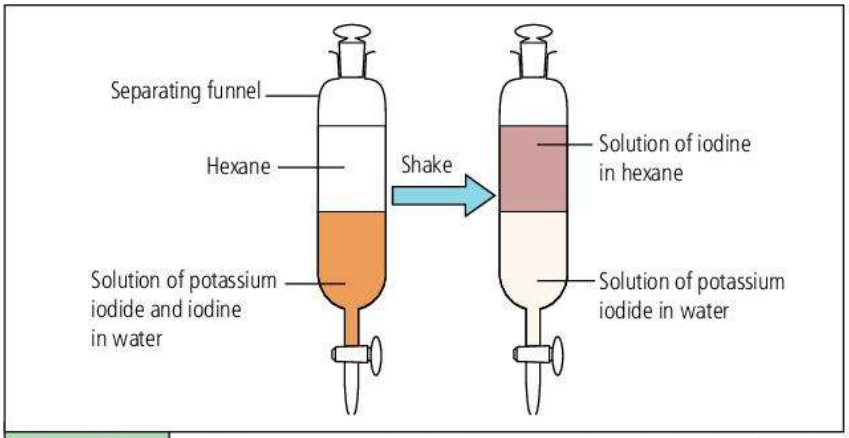


Figure 2.7.3 When a solution of iodine and potassium iodide in water is shaken with hexane, the iodine moves to the layer of hexane.

DID YOU KNOW?

Substances that are not coloured can still be separated by chromatography. After chromatography, the paper is dried, then sprayed with a compound that reacts with the components of the mixture to form coloured compounds. The compound we use as a spray is called a locating agent.

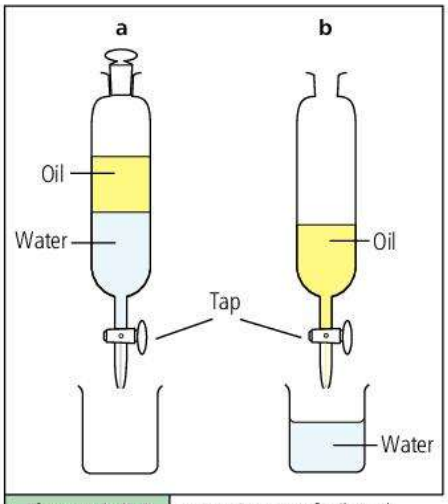


Figure 2.7.2 **a** A mixture of oil and water is allowed to settle into two layers. **b** The lower, denser layer is run off into the beaker.

KEY POINTS

1. Paper chromatography is used to separate a mixture of two or more different solutes in a solution.
2. In paper chromatography, a solvent carries the components of the mixture up the paper at different rates.
3. Paper chromatography can be used to identify the components in a mixture.
4. A separating funnel is used to separate two immiscible liquids.