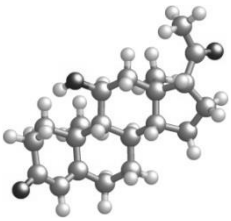

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Separation Techniques: Chromatography, Distillation, Filtration, Separating Funnel and Sublimation

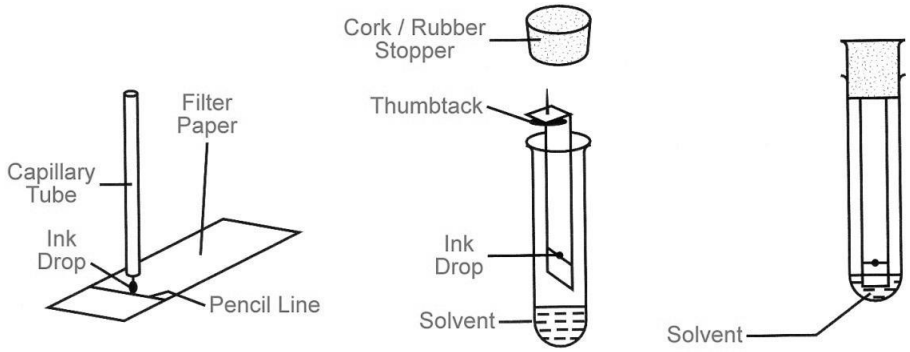
Answers

• **Method One – Chromatography:**

a) What type of mixture(s) can be separated by chromatography?

Chemicals that are soluble in the same solvent. **Note:** The chemicals do *not* need to be *coloured*.

b) One possible way to separate a mixture of chemicals that are often, but not necessarily, coloured is to use chromatography. The essential steps to perform chromatography are given below:



Give a clear and concise explanation of how chromatography works in order to separate a mixture of chemicals:

The solvent (known as the *mobile phase*) moves up the filter paper (known as the *stationary phase*). The ink drop (mixture of chemicals to be separated) dissolves into the solvent and travels up the filter paper. The *more soluble* it is in the solvent and the *less it adsorbs* to the filter paper, the *further* the chemical travels. The *less soluble* it is in the solvent and the *more it adsorbs* to the filter paper, the *shorter* the distance that the chemical travels. This causes the chemicals in the mixture to travel different distances and therefore become separated.

c) Why must the starting line on the filter paper be drawn in *pencil*?

Pencil lead is a mixture of graphite and clay, both of which are insoluble in most solvents. Pencil lead will not dissolve in the solvent and will not interfere with the results. If the start line were drawn in ink, then the ink may dissolve in the solvent, travel up the paper, and interfere with the results.

d) Why is it essential for the ink drop be *above* the level of the solvent?

The ink drop must be *above* the level of the solvent in order to dissolve in the solvent and travel up the filter paper. If the ink drop were *below* the level of the solvent, then it would dissolve in the solvent and remain at the bottom of the test tube without any separation taking place.

e) How are the R_f values of the different pigments in the ink drop calculated?

Rf = distance travelled by spot ÷ distance travelled by solvent.

All distances are measured from the start line, and maybe measured in mm of cm. The R_f value itself is a ratio and therefore does not have any units.

f) What additional step(s) must be taken when performing chromatography on a mixture of *colourless* chemicals?

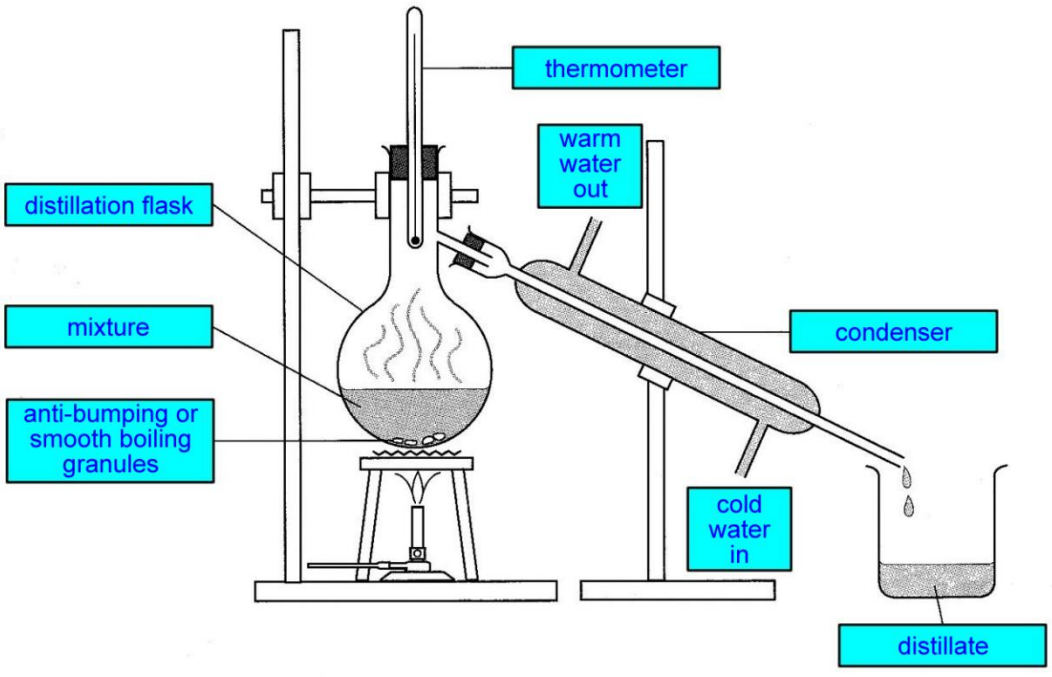
The colourless spots must be sprayed with a *locating agent*. The locating agent reacts with the colourless spots to form coloured products which can be seen with the naked eye.

• Method Two – Distillation:

a) What type of mixture(s) can be separated by distillation?

A solute and a solvent. *Simple distillation* is normally used to separate two miscible liquids that have significantly different boiling points, *e.g.* ethanol (b.p. 78 °C) and water (b.p. 100 °C). *Fractional distillation* is used to separate a complex mixture of miscible liquids which have similar boiling points.

b) A mixture of ethanol (boiling point = 78°C) and water (boiling point = 100°C) can be separated by distillation. Label the diagram of the distillation apparatus given below:



c) Give a clear and concise explanation of how the distillation apparatus works in order to separate two chemicals:

The mixture to be separated is placed in a distillation flask and heated. The chemical with the lower boiling point (*i.e.* more *volatile*) will be the first to boil. Vapours of this first fraction rise, and a small amount condenses on the bulb of the thermometer which will indicate the chemical's boiling point. The rest of the vapour enters the condenser where it is cooled and condenses from a gas to a liquid. The liquid flows out the bottom-end of the condenser and is collected as the *distillate*. The chemical with the higher boiling point will remain in the distillation flask as the *residue*.

Note: A *fractionating column* may be added between the distillation flask and condenser. The fractionating column offers a large surface area over which vapours with different boiling points can condense and return to the distillation flask. This ensures that vapours from a *complex mixture* only enter the condenser – and are separated – one-at-a-time.

d) What is the role of the *anti-bumping granules* in the distillation apparatus?

The anti-bumping or smooth boiling granules present a large surface area over which *many small* bubbles can form once the mixture starts to boil. When the small bubbles burst, they do *not* cause the apparatus to *shake* or *bump*. By contrast, if anti-bumping granules were *not* used, then *large* bubbles would form when the mixture boils. These larger bubbles *would* cause the apparatus to shake when they burst.

e) Pay careful attention to where the *thermometer* is located in the distillation apparatus. Why is the bulb of the thermometer placed at this exact location?

Some vapours will condense on the bulb of the thermometer just before entering the condenser. The thermometer will therefore show the boiling point of the chemical that is entering the condenser and being collected as the distillate.

f) In which direction does the water flow through the *condenser*? Why is the direction in which the water flows through the condenser important?

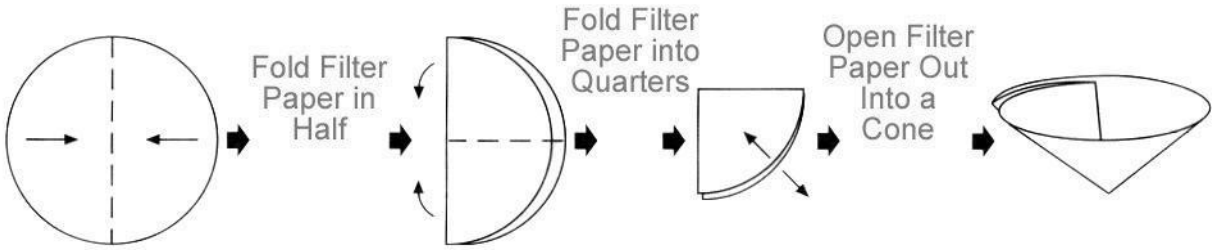
Water enters at the bottom of the condenser and exits from the top of the condenser. As a consequence, the cold water flows in the *opposite direction* to the hot vapour (a process called counter current flow) which is the most efficient way of cooling and condensing the vapour.

• **Method Three – Filtration and Crystallisation:**

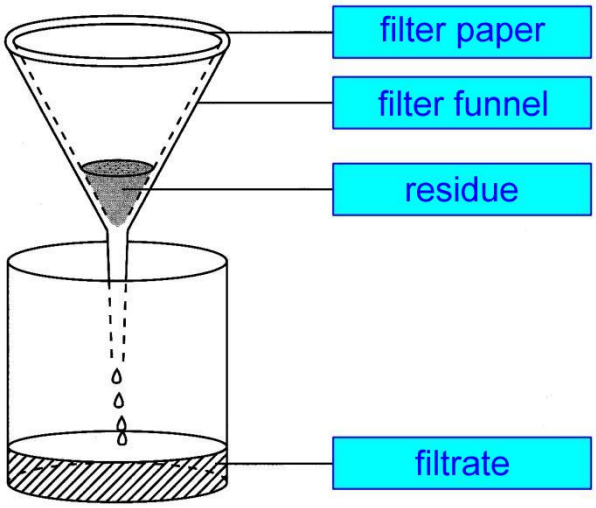
a) What type of mixture(s) can be separated by filtration?

An insoluble solid can be separated from a liquid or a solution. For example, sand is insoluble in water, so filtration can be used to separate sand from water, or separate sand from a solution of salt water.

b) A mixture of copper(II) sulfate and sand can be separated by filtration and crystallisation. A brief summary of the procedure is given in the diagram below.



Label the diagram of the filtration apparatus given below:



c) Give a clear and concise explanation of how the filtration apparatus works in order to separate the mixture of copper(II) sulfate and sand:

Firstly, distilled water must be added to the mixture of copper(II) sulfate and sand. Copper(II) sulfate is soluble in water and will dissolve to form a blue solution, while sand is insoluble in water and will settle to the bottom of the beaker. Filter paper is folded and placed inside the filter funnel. The mixture of copper(II) sulfate solution and sand is then poured into the filer paper. The particles that make-up the copper(II) sulfate solution are small enough to pass through the tiny holes in the filter paper and are collected as the *filtrate*. The grains of sand are too large to fit through the tiny hole in the filter paper – the sand remains in the filter paper as the *residue*.

d) After the filtration is complete, what additional steps need to be taken in order to obtain a sample of *pure*, *dry* sand?

The sand should be rinsed with distilled water to wash away any remaining copper(II) sulfate solution. The sand can then be dried by pressing it between layers of filter paper. Any remaining distilled water can be allowed to evaporate at room temperature, leaving pure, dry sand.

e) What additional steps need to be taken in order to obtain *crystals* of copper(II) sulfate from the *solution* of copper(II) sulfate?

The solution of copper(II) sulfate should be heated until it is *saturated*. The saturated solution should then be left at room temperature for copper(II) sulfate to crystallise. **Note:** Solutions are *not* normally heated to complete dryness just in case the solute decompose at a high temperature.

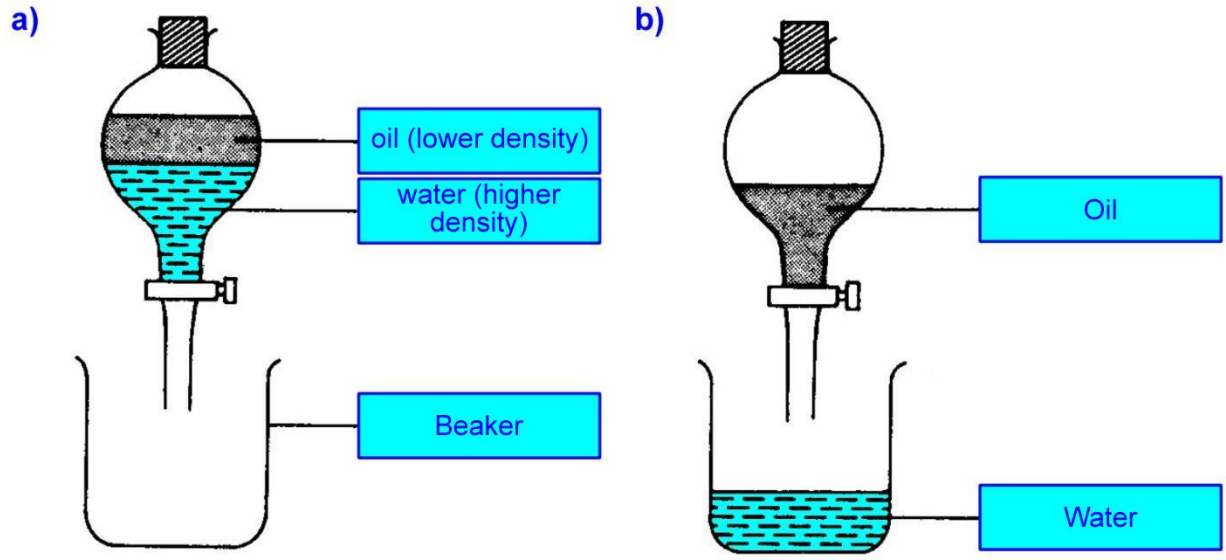
• Method Four – Separation Funnel:

a) What type of mixture(s) can be separated using a separating funnel?

Two immiscible liquids, for example, oil and water.

b) A mixture of oil and water can be separated by using a separating funnel. A brief description of the procedure is given in the diagram below:

Label the diagram below of the separating funnel experiment:



d) Give a clear and concise explanation of how the separating funnel works in order to separate the mixture of oil and water:

Ensure that the tap of the separating funnel (also known as a tap funnel) is closed. Pour the mixture of oil and water into the separating funnel. Allow time for the mixture to settle so that two distinct layers can be seen in the separating funnel. The top layer is the lower density oil while the bottom layer is the higher density water. Place a beaker under the separating funnel. Carefully open the tap allowing the lower layer – liquid water – to flow out of the separating funnel. Close the tap when the interface between the oil and water reaches the tap. The more dense of the two immiscible liquids is collected in the beaker. The less dense of the two immiscible liquids remains in the separating funnel.

• **Method Five – Sublimation:**

a) What type of mixture(s) can be separated by sublimation?

A mixture of two solids, one which sublimes on heating (changes directly from a solid into a gas) and one which does not sublime. Chemicals that sublime on heating include ammonium chloride (NH_4Cl), solid carbon dioxide (CO_2 – dry ice) naphthalene ($C_{10}H_8$ – moth balls) and iodine (I_2).

b) Give a clear and concise explanation of how sublimation works in order to separate a mixture of ammonium chloride and sodium chloride:

When the mixture of two solids is heated, the more volatile solid sublimes (changes directly from a solid into a gas) and its vapour rises. This vapour may be collected by deposition (changes directly from a gas into a solid) on a cold surface, *e.g.* a test tube that contains some crushed ice. The solid that does not sublime will remain as the residue.