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Chem Lectures

## Purification and Separation



Viagra is a drug to increase potency and treatment of erectile dysfunction. It should also be noted that Viagra has an effect on the natural mechanism of erection, in other words, the action of Viagra appears only when adequate sexual stimulation, and you should not expect a spontaneous erection after taking Viagra.

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The Food and Drug Administration (FDA) warns consumers about the possible dangers of buying medicines online. Some websites sell prescription and over-the-counter medicines that may not be safe to use.

Buying prescription drugs and over-the-counter medicines on the Internet from an unreliable company means that you do not know what you are getting exactly. There are many 'rogue' websites that offer to sell potentially dangerous drugs that have not been checked for safety or effectiveness, or manufactured by dubious factories. The site may look professional and legitimate, but it could in fact be an illegal operation selling knock-offs. These 'rogue' sites often sell unapproved drugs that contain the wrong active component, no active component, or in the worst cases, other dangerous components such as arsenic and mercury.<sup>#</sup>

**Why do medicines need to contain the right component(s)?**

**How do we know what are the component(s) present?**

**How can we separate out the individual components?**

Excerpt extracted from:

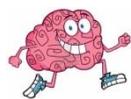
#FDA website at [www.fda.gov/forconsumers/consumerupdates/ucm048396.htm](http://www.fda.gov/forconsumers/consumerupdates/ucm048396.htm)

### Learning Outcomes

1. Explain, with examples, why pure substances are needed.
2. Suggest methods of separation and purification given information about components of mixtures.
3. Describe methods of separation and purification for the components of the following types of mixtures: solid-solid, solid-liquid, liquid-liquid (miscible and immiscible).
4. Techniques to be covered for separations and purification includes: (i) use of a suitable solvent, filtration and crystallisation or evaporation, (ii) sublimation, (iii) distillation and fractional distillation, (iv) use of a separating funnel, (v) paper chromatography.
5. Interpret chromatograms including comparison with known sample and the use of  $R_f$  values.
6. Explain the need to use locating agents in the chromatography of colourless components.
7. Deduce from the given melting point and boiling point, the identities of substances and their purity.
8. Explain that the measurement of purity in substances used in everyday life is important.

## 1. What is a Pure Substance?

- **A pure substance is** \_\_\_\_\_, e.g., pure gold, pure silver, distilled water.



### Rack-your-brains

**Q.** Table salt can be bought from the supermarkets or provision shops in a packet as follows. Do you think that such a packet will contain pure salt (or pure sodium chloride)?

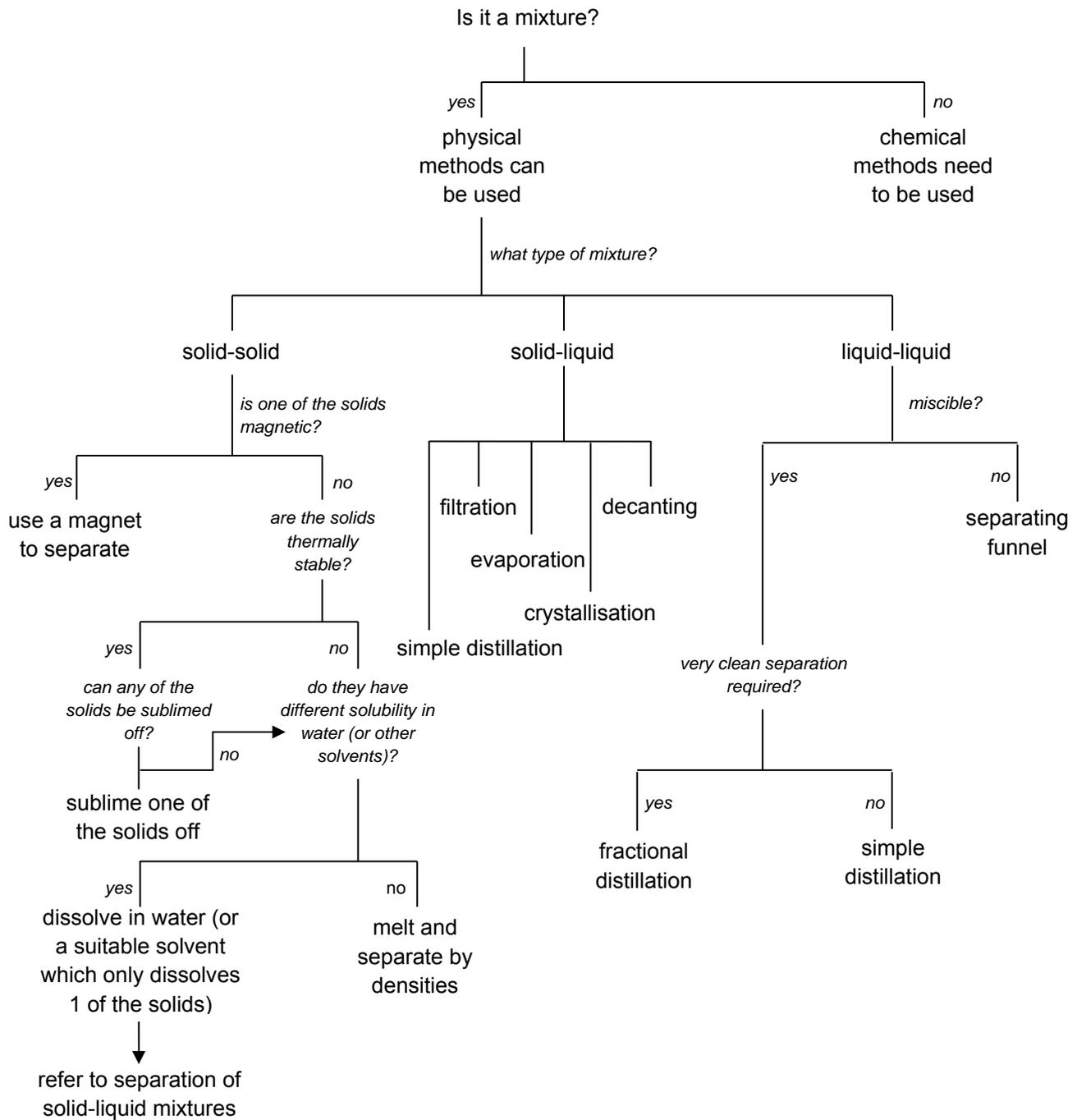


**Figure 1.1** Packet of table salt and the labelling on the side

**A.** No. Table salt contains other substances such as magnesium and iron compounds, but in very small quantities. Sometimes, table salt may also be iodised, i.e., potassium iodide added. Iodine is required for thyroid functions, and adding it into salt will allow us to have a steady intake of iodine to prevent iodine deficiency.

- **A mixture consists of** \_\_\_\_\_, e.g., milk, Milo, Ribena, orange juice, sea water.
- An impure substance can be a mixture of more than two different substances.
- Purity is an important indicator in industry of:
  - extent of a reaction
  - efficiency of a reaction
  - efficacy of a chemical
  - price of a chemical, e.g., analytical grade (99.999% pure) ethanol costs about 100/L while laboratory grade (99.5% pure) ethanol costs about 20/L

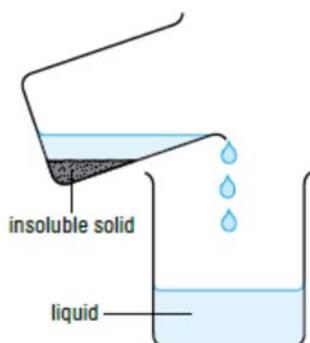
## 2. How can we Obtain Pure Substances from Mixtures?



- Mixtures can be easily separated using physical methods.

### **(i) Solid-liquid mixtures**

#### ***(1) Decanting***



**Figure 2.1** Decanting

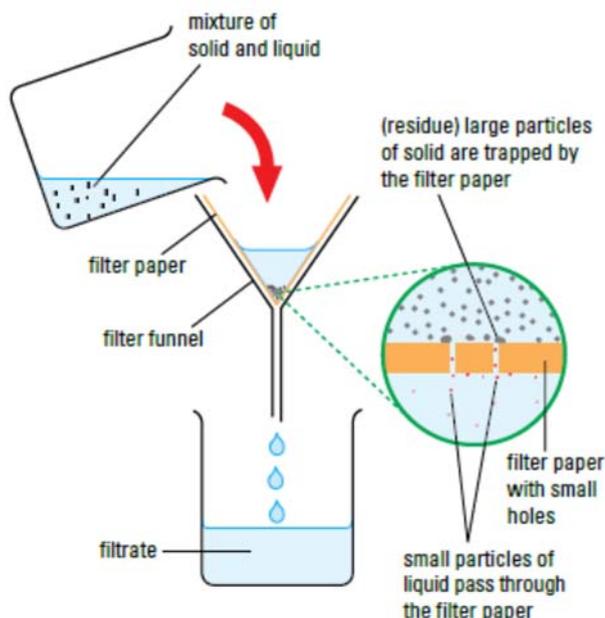
- Decanting separates*** \_\_\_\_\_ from the container carefully.
- This method is seldom used in the laboratory.
- This method is often used at homes to pour soup out of a pot full of other ingredients.



**Figure 2.2** When you pour chicken soup out from the bowl carefully into another bowl, you are practicing the process of decantation.

#### ***(2) Filtration***

- Filtration is used to*** \_\_\_\_\_ - but this does the separation more thoroughly.



**Figure 2.3** Filtration (gravity-assisted)

- The process is as follows:
  1. Fold filter paper
  2. Insert into filter funnel
  3. Place a clean beaker below the filter funnel
  4. Slowly pour the mixture into the filter paper in the funnel
  5. The liquid passes through the small holes in the filter paper while the insoluble solid remains trapped by the filter paper
  6. The **solid is collected in the filter paper** ⇒ \_\_\_\_\_
  7. The **liquid that passes through the filter paper** and collected in the beaker ⇒ \_\_\_\_\_

## Reality Check



In Singapore, we are blessed with readily available clean water. The water collected in our reservoirs are not only filtered to remove particulate impurities, but also treated with chemicals to further remove harmful substances.

**Figure 2.4** Women collecting drinking water in Africa

Would you want to be drinking the water that was collected from the pool of mud in Figure 2.4? Some Collectives have been promoting the use of simple cloth filters in the poorer African states, where clean water is not readily available. A simple filter made from several layers of cloth can effectively remove most of the larger particulate impurities from water. The filtered water is then

boiled to kill any harmful micro-organisms before it is drunk.



**Figure 2.5** Filtering dirty water with a simple cloth filter can yield remarkable results

### (3) *Evaporation*

- *Evaporation is used to separate*



**Figure 2.6** Salt pans by the sea in Gozo, where seawater is evaporated by the heat from the Sun and the salt left behind harvested

- However, evaporation has two main disadvantages:
  1. Some solids are not thermally stable and will decompose when heated, e.g., sugar.



**Figure 2.7** Sugar caramelises on gentle heating, before it decomposes into black carbon

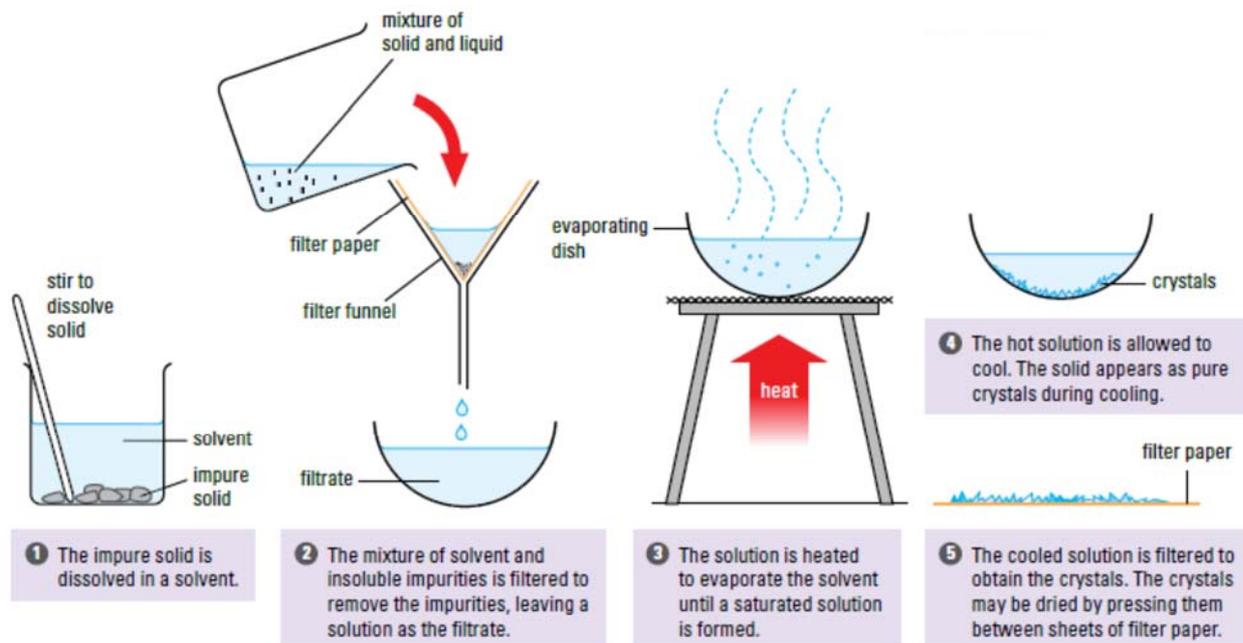
2. Even after evaporation, other soluble impurities will still be present in the solid residue.

**Checkpoint 1**

Describe and illustrate with diagrams how a mixture of iron filings, salt and sand can be separated.

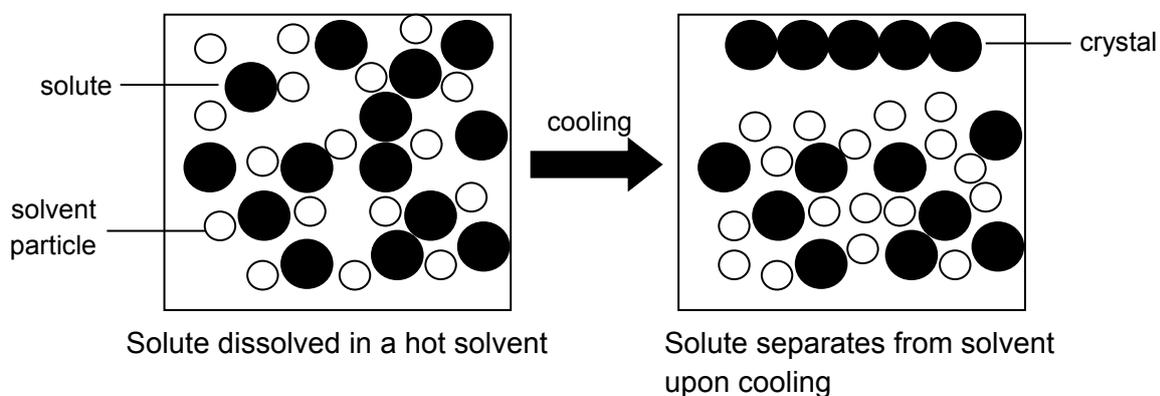
**(ii) Solid-solid mixtures****(4) Crystallisation**

- **Crystallisation separates a**



**Figure 2.8** Process of crystallisation

- A saturated solution is one where no more solute will dissolve in the solvent.
- The slower the rate of cooling, the larger the crystals that will be formed.
- Crystallisation occurs because the solubility of the solute decreases as the temperature decreases. When the solution cools, the extra solute that cannot remain dissolved is separated as pure crystals.



**Figure 2.9** Crystallisation at the atomic level

## Crystallisation on the Global Scale



Large crystals of quartz can be found in the Earth's crust. They are formed by the cooling of molten rock. Quartz crystals are widely used in making watches, where they are used in an electronic oscillator circuit to keep time.

Figure 2.10 Quartz crystals

### (5) Sublimation

- Sublimation is used to

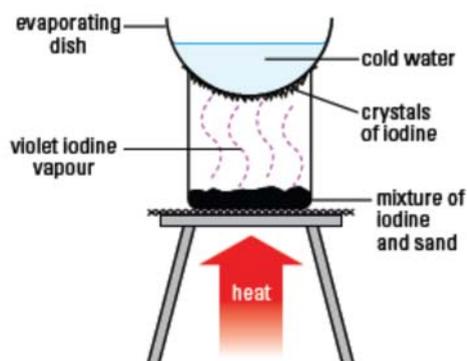


Figure 2.11 Purification of a mixture of iodine and sand by sublimation

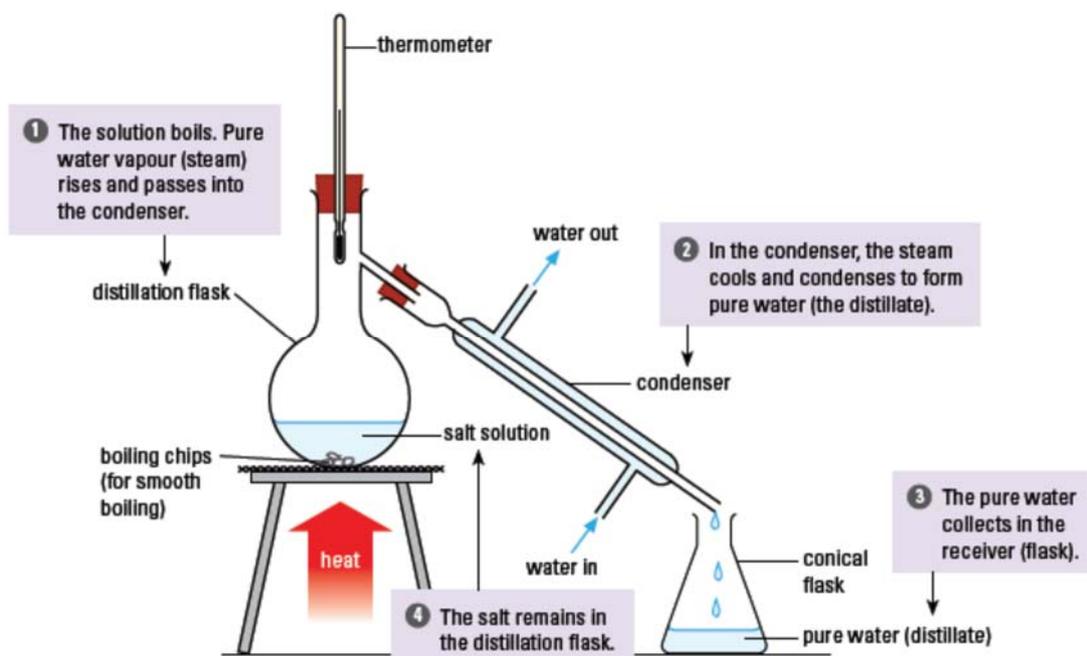
- Sublimation is the process of changing from a solid to vapour on heating without going through the liquid state.



Figure 2.12 Dry ice (solid carbon dioxide) sublimates

**(6) Simple Distillation**

- Simple distillation separates**



**Figure 2.13** Simple distillation set-up

- The thermometer is placed near the exit of the distillation flask. This allows it to measure the temperature of the vapour which will enter the condenser and be condensed and collected as distillate.
- Distillation can be used to obtain a pure solvent from a solution of a solute, e.g., distillation of seawater, also known as desalination.

**Singapore's Water Supply**

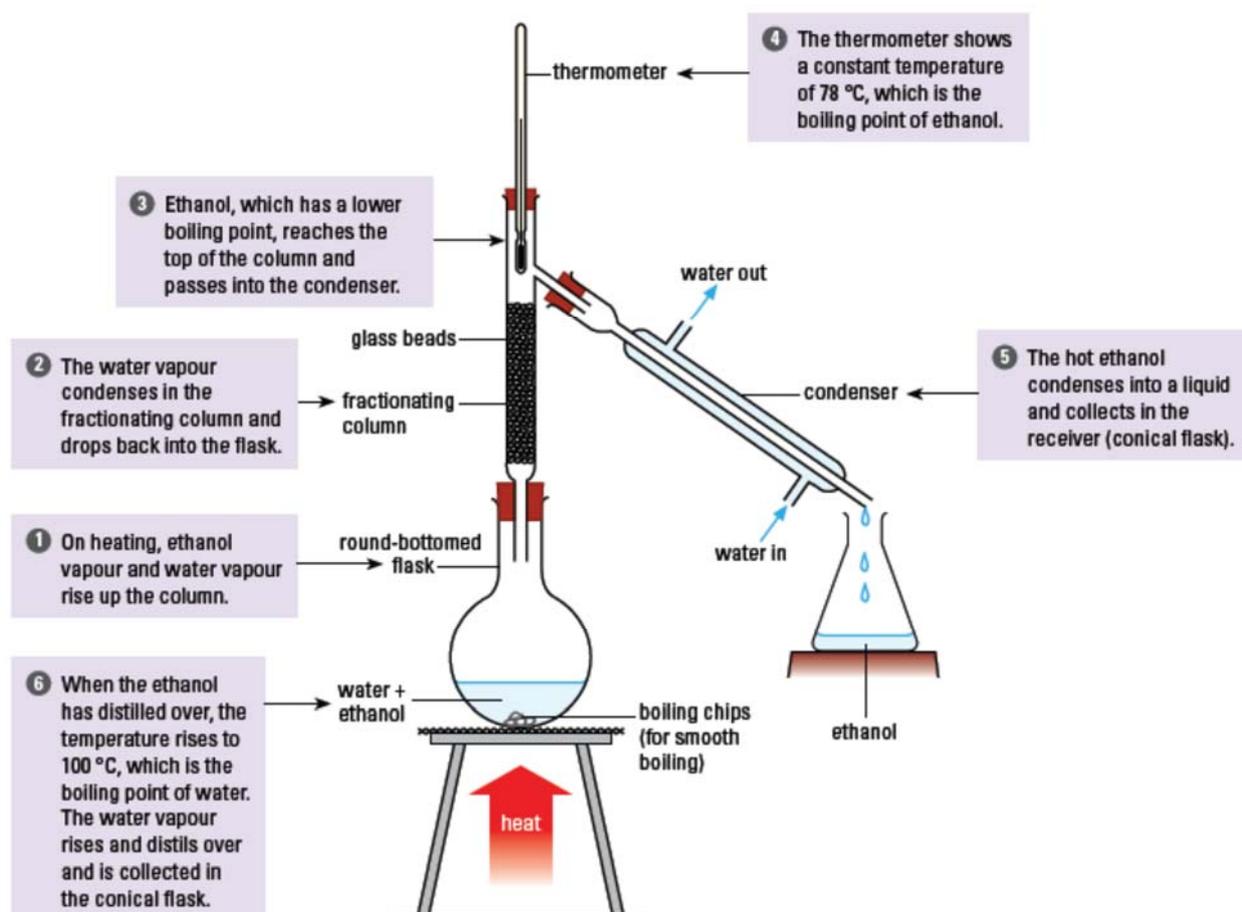
### Mini Case Study: Singapore's Water Supply

Desalinated water plays a big role in Singapore's water strategy. Currently, about two-thirds of Singapore's water supply comes from our reservoirs and Malaysia. The rest is either recycled water, or desalinated water. However, this is not enough for the long term, and with water demand forecasted to double in the next 50 years, the government intends for water recycling and desalination to play a bigger role. In a news report on 28 June 2010, it was cited that the Singapore government intends to raise our desalination capacity to meet at least 30% of long-term demand. This is up from the current 3%.

**Figure 2.14** Singapore's Water Supply Strategy

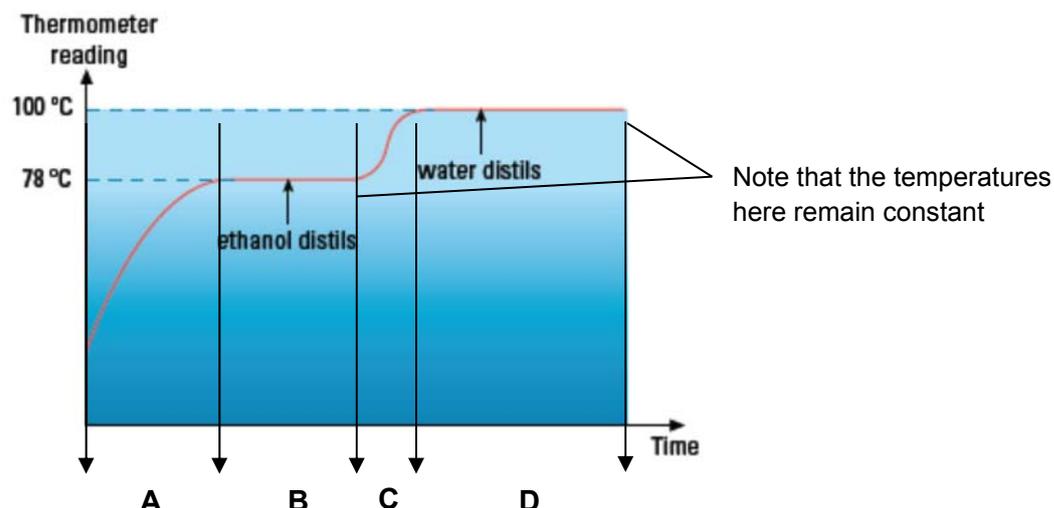
**(iii) Liquid-Liquid Mixtures****(7) Fractional Distillation**

- **Fractional distillation separates mixtures of**
- Miscible liquids are completely soluble in each other to form one liquid, e.g., ethanol and water.



**Figure 2.15** Fractional distillation of a mixture of water and ethanol

- The glass beads in the fractionating column provides a large surface area to volume ratio for cooling of the vapours. This helps to ensure that only one liquid of the lower boiling point distils over. This allows better separation of the two or more miscible liquids in the round-bottomed flask.
- The thermometer is placed near the exit of the fractionating column. This allows it to measure the temperature of the vapour which will enter the condenser and be condensed and collected as distillate.



**Figure 2.16** Thermometer readings during the fractional distillation of the ethanol-water mixture

- Description of the various segments A-D in Figure 2.16:
  - A. The temperature rises as the ethanol-water mixture is heated.
  - B. The temperature of the mixture reaches the boiling point of ethanol. The temperature remains at the boiling point of ethanol as ethanol boils (distils) off. Any water which evaporates off at this point is cooled and condensed by the condenser.
  - C. All the ethanol has distilled off. The temperature of the liquid rises.
  - D. The temperature of the liquid reaches the boiling point of water. The temperature remains at the boiling point of water as water distils off.
- Fractional distillation has some of the following uses:
  - To separate air into its various components, e.g., pure oxygen, pure nitrogen.
  - To separate petroleum into its various important fractions, e.g., petrol, diesel.
  - To produce ethanol which is used in alcoholic drinks.



**Figure 2.17** Aerial shot of Pulau Bukom, one of the major refining centres in Singapore

**Checkpoint 2**

1. Filtering seawater will not give us potable water. Give a reason for this, and illustrate with a diagram how we can obtain potable water from seawater.

2. When liquid air is fractionally distilled, the various components which make up air boil off at different temperatures.

Component	Boiling point/ °C
Nitrogen	-195.8
Oxygen	-183
Argon	-186
Neon	-246
Krypton	-152
Carbon dioxide	-57

List the order in which the gases will be fractionally distilled off.

3. A liquid mixture containing methanol (boiling point of 65 °C), ethanol (boiling point of 78 °C), and

water (boiling point of 100 °C) was fractionally distilled. Draw a graph to show the temperature changes as recorded by the thermometer in the fractional distillation column for the entire distillation process.

### (8) Chromatography

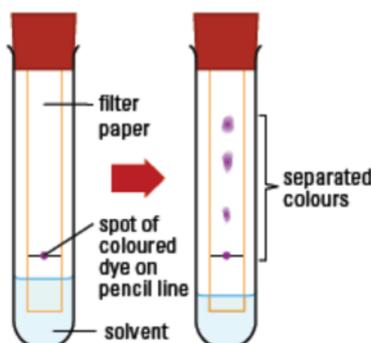
- Chromatography is a method of



**Figure 2.18** Chromatogram of common food dyes

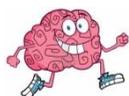
- There are various types of chromatography techniques available:
  - Paper chromatography (as shown in Figure 2.18)
  - Thin layer chromatography
  - High pressure liquid chromatography
- Chromatography has the following uses:
  - Separate and identify the components present in coloured substances and food dyes
  - Separate and identify substances in drugs
  - Separate and identify substances in blood and urine (to determine if athletes have been using banned performance-enhancement drugs)

- Only a very small initial sample is required to generate the chromatogram.



**Figure 2.19** Paper chromatography

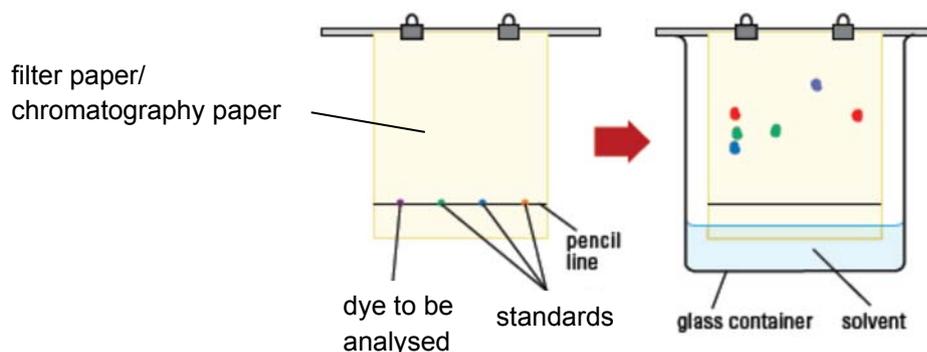
- The following steps are involved in generating the paper chromatogram in Figure 2.19
  - (1) The dye to be chromatographed is dissolved in a suitable solvent.
  - (2) A drop of the dissolved dye is placed on the pencil line near the bottom of a strip of filter paper (or chromatography paper can be used). The pencil line shows the starting position of the drop of dissolved dye. This is important in determining the  $R_f$  value.
  - (3) The filter paper is dipped into a tube containing a suitable solvent, making sure the solvent level is below the spot.
  - (4) The solvent travels up the filter paper and the dye in the spot dissolves and travel up the paper at different speeds. This separates the dye into its various components.
  - (5) When the solvent front reaches nearly the top of the filter paper, the filter paper is removed.
  - (6) The result is called a chromatogram.



## Rack-your-brains

- Q. What is the purpose of the pencil line?
- Q. Why must we ensure that the solvent level in the tube is below the spot on the pencil line?

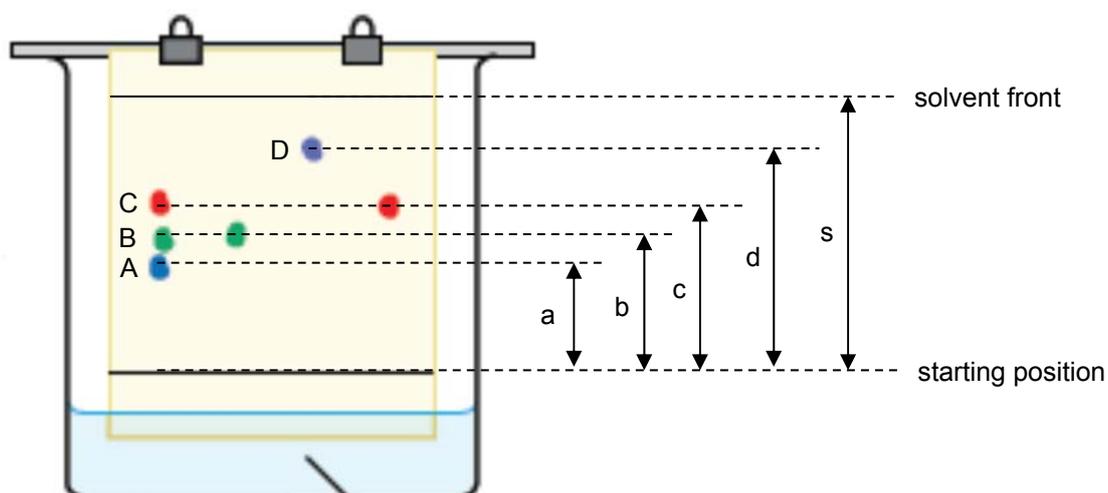
- To use paper chromatography to identify the components in the dye, we will need to run the dye together with some known components (also known as standards).



**Figure 2.20** Identifying the individual components in a dye

- Interpretation of Figure 2.20
  - Identical substances travel \_\_\_\_\_.
  - We can compare the components in the dye with the standards used.
  - The dye to be analysed is made up of \_\_\_\_\_ different components.
  - Two of the components (red and green) are the same as two of the standards because they are at the same distance from the pencil line.
  - The dye contains a third component which is not the same as any of the standards used, and cannot be identified.
- **$R_f$  value is the distance moved by a substance relative to that moved by the solvent.**

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

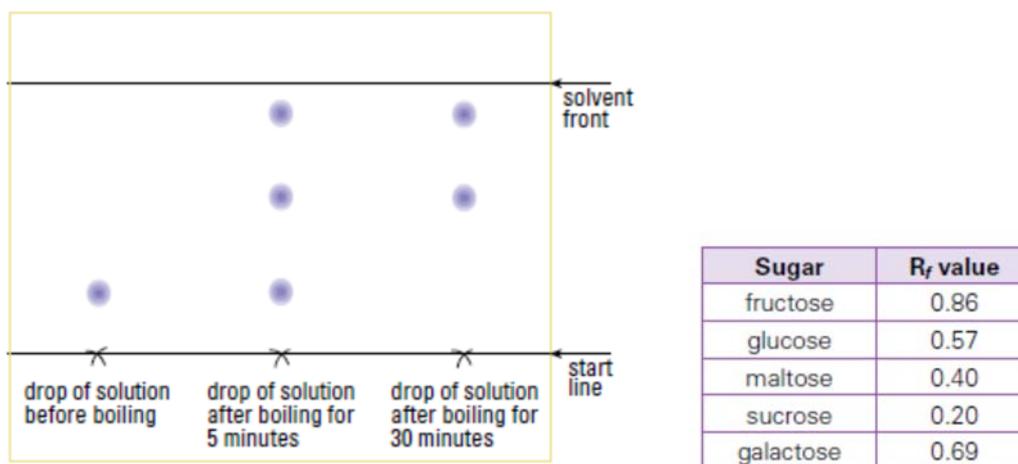


**Figure 2.21** Sample chromatogram

Substance	Distance travelled by substance	Distance travelled by solvent	R <sub>f</sub>
A			
B			
C			
D			

- We can determine the identity of the three components in the dye by comparing their R<sub>f</sub> values with the standards. We can see that components B and C in the dye are the same as the standards used. Component A is unknown.
- R<sub>f</sub> values from the chromatography of different substances in various solvents and temperatures are known. We can use these R<sub>f</sub> values to compare with that on our own chromatogram to identify the components.
- When a chromatogram is run for a colourless substance, a locating agent is used. **A locating agent is a substance that reacts with the substances on the chromatography paper to produce a coloured product.** This allows measurements to be taken to determine the R<sub>f</sub> value of its components.

### Checkpoint 3



**Figure 2.22** Sample chromatogram and R<sub>f</sub> values of some sugars

When sucrose is boiled with acids, a reaction takes place.

Some sucrose was added to dilute hydrochloric acid and boiled for 30 minutes. At 5 minutes, a drop of the mixture was applied to a chromatography paper. At 30 minutes, another drop of the mixture was applied.

A chromatogram was produced by running the paper in a suitable solvent. A locating agent was sprayed on the chromatogram at the end of the experiment.

1. Why was a locating agent used?
2. How much sucrose remained in the mixture after boiling for 30 minutes?
3. Use the  $R_f$  values in the table to explain what happened to the sucrose in the mixture.

### 3. How Do We Know if Something is Pure?

- ***A pure substance has a*** \_\_\_\_\_, e.g., pure water melts at 0 °C and boils at 100 °C.

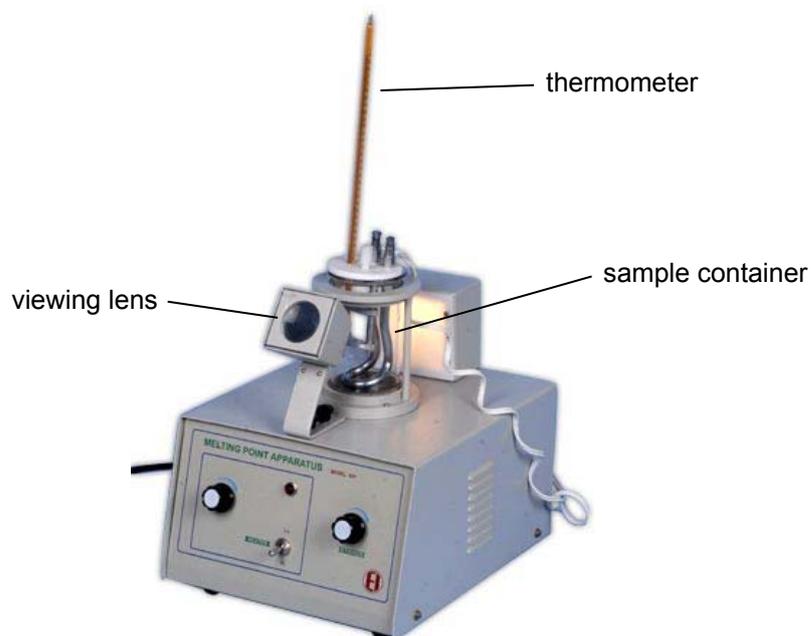


Figure 3.1 Melting point apparatus

- Impurities affect the melting point in two ways:
    - (1) **Lowers the melting point**. The greater the amount of impurity, the lower the melting point.
    - (2) Substance **melts over a range of temperatures**, i.e., no sharp melting point.

The melting point apparatus in Figure 3.1 allows us to determine the temperature at which a sample melts. If it melts sharply, the sample is pure. Since different substances have different melting points, we can use the temperature reading from the melting point apparatus to identify the sample (if its identity is unknown).
  - Impurities affect the boiling point in two ways:
    - (1) **Raises the boiling point**. The greater the amount of impurity, the higher the boiling point.
    - (2) Liquid **boils over a range of temperatures**, i.e., no sharp boiling point.
  - Mixtures melt/ boil over a range of temperatures (as they are not pure).
  - When chromatographed, **a pure substance only**
- 

**Checkpoint 4**

1. An impure substance X melts at 113 °C. Underline one of the substances below that could be substance X? Give your reasons.

Resorcinol (111 °C), catechol (105 °C), nitrophenol (114 °C)

2. A sample of a pure dye was dissolved in ethanol, applied onto a piece of filter paper and chromatographed with a 70/30 ethanol-water mixture as the solvent. Draw a probable chromatogram below.

**Why must drugs be pure?**

1. Why do medicines need to contain the right component(s)?
2. How do we know what are the component(s) present?
3. How can we separate out the individual components?

Medicines are taken into our bodies to cause a certain effect. The wrong type of component may be harmful or even deadly.

We can use various chromatographic techniques such as HPLC and GC-MS.

Due to the very small amount of substances present in a sample of drug, we usually use chromatographic techniques to separate out the individual components, e.g., HPLC, GC.