54. Separation and purification of compounds

HKDSE syllabus

Separation and purification methods

Students should learn

- crystallisation
- distillation / fractional distillation
- liquid-liquid extraction
- paper, column or thin layer chromatography

Students should be able to

- describe various separation and purification methods
- separate and purify substances by the following methods:

i. crystallisation

ii. distillation / fractional distillation

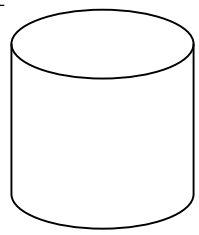
iii. liquid-liquid extraction

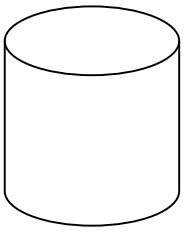
iv. chromatographic methods

- determine the Rf values of substances in a chromatogram
- determine the melting point or boiling point of a substance
- examine the purity of a substance by measuring its melting or boiling point
- justify the choice of an appropriate method used for the separation of substances in a mixture

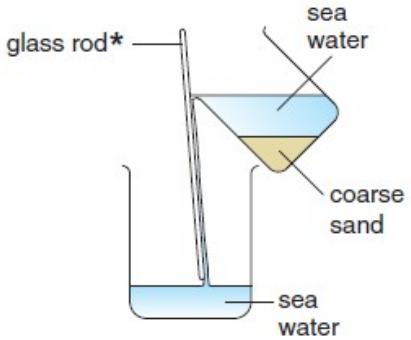
A. Separation and Purification

How to separate and purify the salt solid (NaCl) from the following mixture?



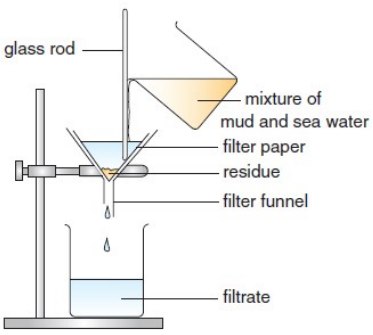


Process 1:_____



To separate a mixture of a liquid from a much denser insoluble solid.

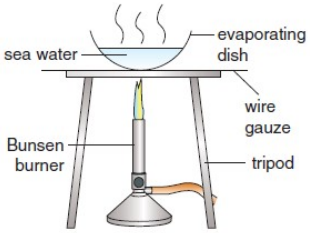
Process 2:_____-____



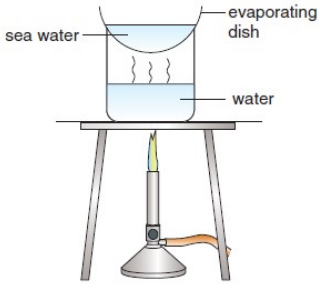
To separate a mixture of a liquid and an insoluble solid suspending in the liquid.

Process 3:_____

To obtain a soluble solid from its solution.
When do we use this method?
1. Obtaining salt from sea water.
2. To concentrate a solution quickly.



(a) Direct heating



(b) Heating with a steam bath

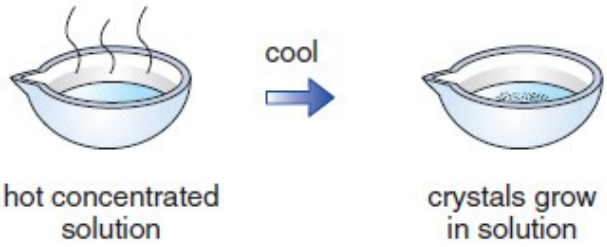
Is the salt pure?_____ , because impurities such as _____ is present.

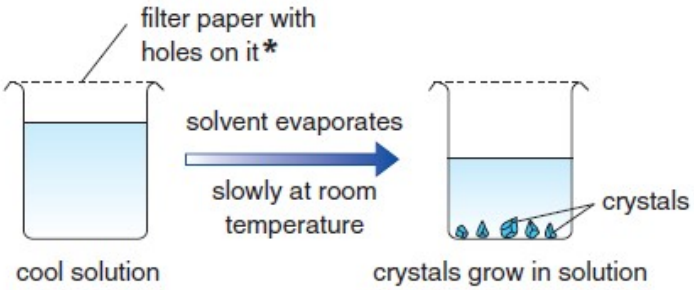
Purification

Process 4:_____

Method 1: Cooling a hot concentrated solution
1. Some solvent is boiled away in order to concentrate it.
2. Test for saturation: cold dry glass rod
3. Stop heating and start cooling

Method 2: Evaporating a solution slowly at room temp
1. As the solvent in a solution evaporates away, the solution becomes more and more concentrated until it becomes **saturated**.
2. Further evaporation causes crystallization to occur.





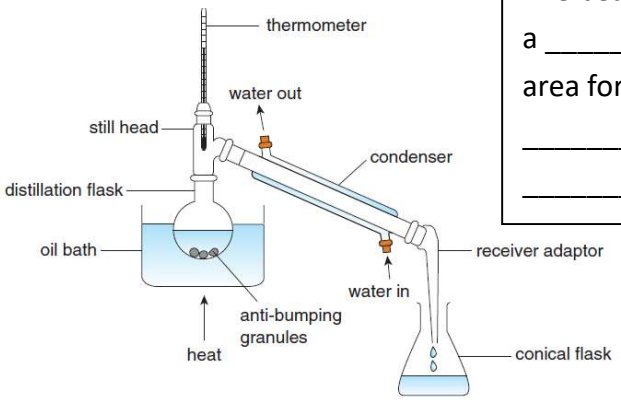
Principle of crystallization
1. As temperature **increases**, solubility of salt **increases**.
2. The solution cannot hold **All** of its solute
3. **Excess** Salt are separated out as crystal.

Washing of crystals
1. After crystallization, the crystals are separated from the remaining solution by **Filtration**
2. After filtration, the crystals are washed with **cold distilled water** as this removes any soluble impurities on the surface of the crystals.
3. The crystals are taken out with a pair of forceps and then dried by blotting on filter paper.

How to separate each chemical species from the following mixture?

Process 5:_____

Difference in b.p. _____



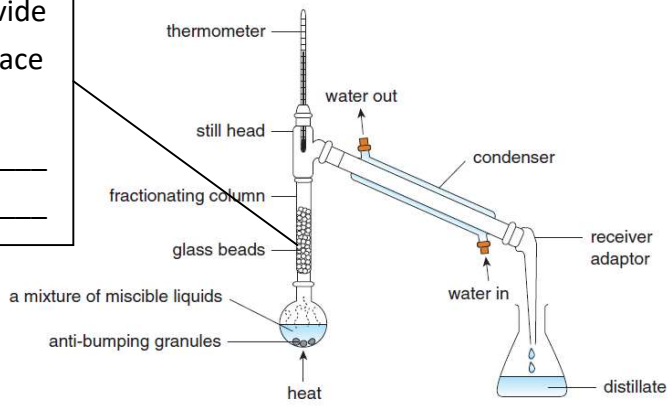
The beads provide a _____ surface area for

_____

_____

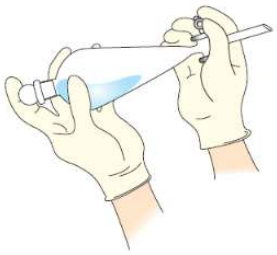
Process 6:_____

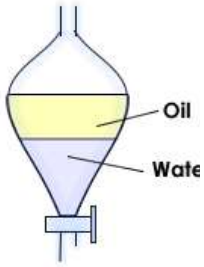
Difference in b.p. _____

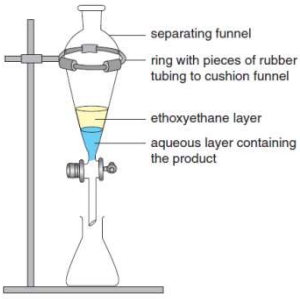


Process 7:_____

You are provided with a mixture of two liquids, **heptanoic acid** and **cylcohexanone**. Outline an experimental procedure, based on liquid-liquid extraction, t**o isolate pure heptanoic acid from the mixture.**





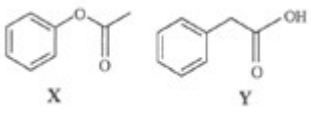


1. Add _____ and dilute _____ solution to the mixture in a separating funnel and **shake**. The heptanoic acid reacts with sodium hydroxide to give sodium heptanoate.
2. Allow the organic layer and the aqueous layer to _____ after shaking.
3. The organic layer contains _____ while the aqueous layer contains _____. Run off and collect the aqueous layer.
4. Regenerate the heptanoic acid by adding dilute hydrochloric acid to the aqueous layer.
5. Then extract the heptanoic acid with ethoxyethane, remove final traces of water using a drying agent, and filter off the drying agent.
6. Finally distill off the ethoxyethane to obtain pure heptanoic acid.

Like dissolves like

Water:_____

Oil:_____



(1) You are provided with dilute $Na_2CO_3(aq)$ and dilute $H_2SO_4(aq)$. Outline an experimental procedure, based on solvent extraction, to separate solid **Y** from a solution of **X** and **Y** in dichloromethane. 2016 DSE PII Q3 C)ii)1

_____

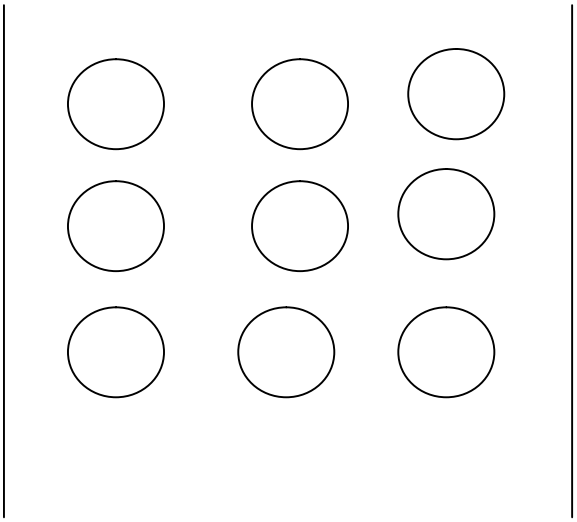
_____

_____

_____

B. Chromatography (色層法)

Principle of chromatography



The movement of each dye depends on 2 factors:

1. _____ of dye in the solvent
2. _____ of the dye on the stationary phase.

There are _____ types of chromatography in the syllabus.

R_f (Retention factor) value of substance

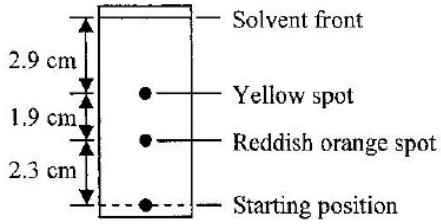
R_f = ----------

Different substance will have different R_f value in different solvent.

(c) The main pigments in a certain brand of tomato paste are lycopene (reddish orange) and β-carotene (yellow). In order to isolate lycopene from the tomato paste, an experiment involving solvent extraction, thin-layer chromatography (TLC) and column chromatography was performed.

2015 DSE P2 Q3 3c)i)

(i) The result of TLC is shown below:



Calculate the R_f value for the lycopene spot.

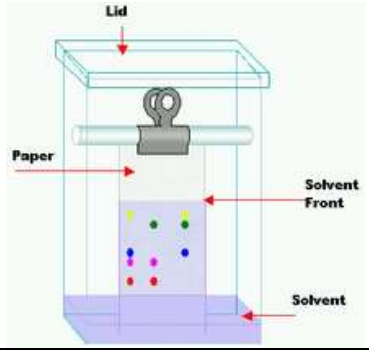
(1 mark)

3c)ii)

(ii) With reference to the result of TLC, explain whether the first-collected coloured fraction in the column chromatography is lycopene or β-carotene, if the same stationary phase and mobile phase are used.

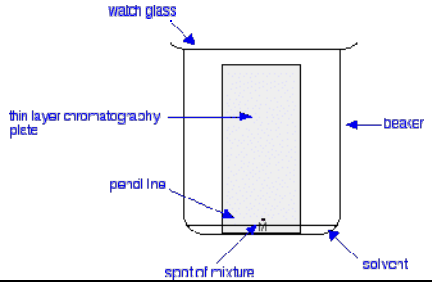
(1 mark)

_____ Chromatography

Diagram	Procedures
	1. Draw a _____ using a pencil. 2. Apply a _____ of coloring on the baseline and allowed to _____. 3. Put the paper in the solvent with the baseline above/below the liquid level. 4. Allow the solvent to move up (diffusion)

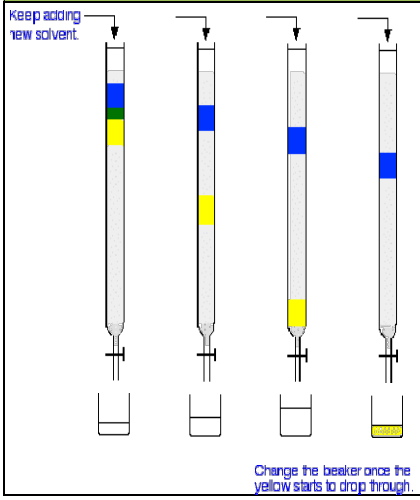
Mobile phase	
Stationary phase	_____ in the Paper
Functions	1. _____ 2. _____
Remarks	If the component is colourless: 1. If it is amino acid, use _____ to make it visible. 2. Or place the chromatogram in the atmosphere of I_2 vapour. 3. If it is fluorescent, use _____ to make it visible.

_____ Chromatography (TLC)

Diagram	Procedures
	TLC is the similar to that of paper chromatography EXCEPT that the adsorbent is a _____

Mobile phase	
Stationary phase	Fine layer of _____ or _____ _____ coated onto a glass plate.
Functions	1. _____ 2. _____
Remarks	Adv: 1. _____ Disadv: 2. _____

_____ Chromatography

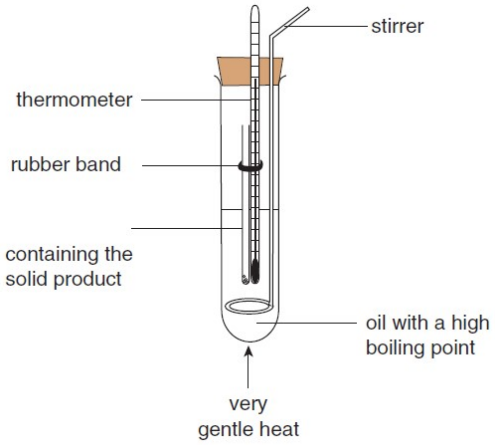
Diagram	Procedures
	1. Mixture to be separated is applied to the ____ of the column. 2. The liquid solvent (_____) is passed through the column by _____ or by the application of_____. 3. Because the different components in the mixture have different _____ with the **stationary** and **mobile** phases, they will be carried along with the mobile phase to varying degrees and a separation will be achieved. 4. The individual components (_____) are _____ as the solvent drips the bottom of the column.

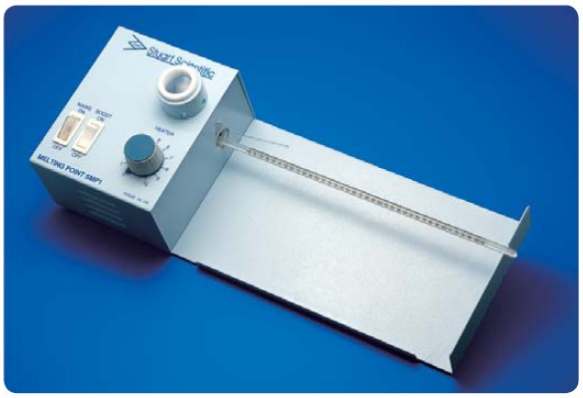
Mobile phase	
Stationary phase	_____ in vertical glass column. E.g.: _____/ _____
Functions	1. _____ 2. _____

C. Test for purity

Determination of melting point

Presence of even ___% impurity can lower the melting point and _____ the range to several degrees





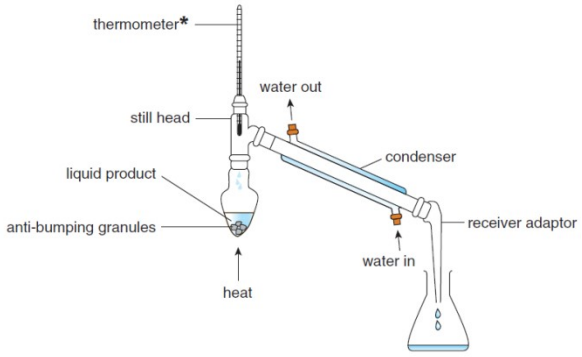
A pure solid should have a <u>sharp/ wide range</u> of melting point because melting point depends mainly on the _____ of structure.

The presence of impurity <u>lowers / increases</u> the melting point of a solid.

Precaution

1. Make sure the level of the solids in the tube is the _____ as the bulb of the thermometer.
2. Avoid heating the liquid too _____.

Determination of boiling point



A pure liquid should have a <u>sharp/ wide range of</u> boiling point

3. (a) Outline how hex-1-ene can be obtained from a mixture of hex-1-ene, octane **and water** by physical methods.
2013 P2 Q3 (Boiling points: hex-1-ene = 63°C, octane = 125°C, water = 100°C)
(4 marks)

2014 DSE P2 Q3)a) (ii) Which of the following chemicals is most suitable for drying ethyl ethanoate ?
anhydrous magnesium sulphate , concentrated sulphuric acid, solid sodium hydroxide
(1 mark)

2014 DSE P2 Q3)a) (iii) Suggest how copper powder can be obtained from a mixture of copper powder and iron(III) oxide by chemical method.
(2 marks)

1. Directions: Questions 1 and 2 refer to the following information. A student obtained the following chromatogram in the identification of the colourings in four fruit drinks, P, Q, S and T.

Which of the drinks contains the green colouring?

A Drink P B Drink Q C Drink S D Drink T

2. Why should the student make further checks on drink T?

A Its colour is too dark.
B Its colour is different from the other drinks.
C It contains too many food colourings.
D It contains a colour that is not identified in this test.

3. A student used paper chromatography to separate two components, X and Y, in a solution. A spot of the solution was initially placed at the origin.
When the spot corresponding to compound X (R_f = 0.60) had advanced 4.5 cm, the spot corresponding to component Y was 1.0 cm before X.
The R_f value of component Y is

A 0.13. B 0.22. C 0.47. D 0.73.

4. Directions: Questions 4 and 5 refer to the following experiment.
Four red substances, W, X, Y and Z, were tested by paper chromatography. The test was done using two different solvents. The chromatograms obtained are shown below.

What is the R_f value of substance Y in solvent 1?

A 0.28 B 0.52 C 0.63 D 1.57

5. An unknown red substance was tested. The chromatogram obtained using solvent 1 is shown below.Its R_f value in solvent 2 is 0.82. The unknown red substance could be

A W.
B X.
C Y.
D Z.

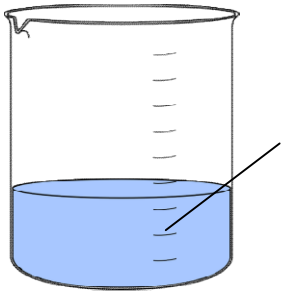
2018 DSE

(iii) What is meant by the 'R_f value' of a substance in a paper chromatogram ?

(2 marks)

BDDCB

Liquid – Liquid Extraction



Example: To Extract the pure $I_{2(S)}$ from $I_{2(aq)}$



If you want to extract more Iodine from the aqueous layer, you can **repeat** the extraction.

Liquid – Liquid Extraction Scheme

Iodine in water

Steps:

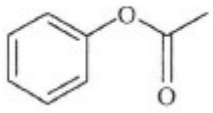
1. Add _____ and _____ _____ into a _____ _____
2. _____ it and allows two layers to separate.
3. _____ the aqueous layer and _____ the upper non-aqueous layer.
4. The solvent is _____ and pure I_2 is collected.

More Example:

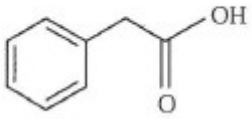
Mixture containing heptanoic acid and benzene

2016 DSE

(c) **X** and **Y** are isomeric compounds with their structures shown below :



X



Y

(ii) The melting point of **X** is 50 °C while that of **Y** is 77 °C. Both of them are insoluble in water but soluble in dichloromethane. When treated with dilute $Na_2CO_3(aq)$, no reaction occurs for **X** but reaction occurs for **Y** to form a soluble salt.

(1) You are provided with dilute $Na_2CO_3(aq)$ and dilute $H_2SO_4(aq)$. Outline an experimental procedure, based on solvent extraction, to separate solid **Y** from a solution of **X** and **Y** in dichloromethane.

(2) Suggest how you can identify that the solid obtained in (1) is pure compound **Y**.

(5 marks)

2017 DSE

3. (c) Many plants contain useful organic compounds which can be obtained by extraction using suitable solvents.

(i) The leaf of a certain plant contains a useful organic compound **S**. **S** can dissolve gradually in a warm organic solvent, and can be extracted from the leaves by using this solvent.

(1) 'Heating under reflux' is a method commonly used to carry out this kind of extraction. State the advantage of this method.

(2) After extraction, the solvent can be removed from the extract by simple distillation. Draw a labelled diagram for the set-up required for this simple distillation.

(3) **S** obtained from the extraction may contain other organic impurities. Suggest a method for separating **S** from these impurities.

(4 marks)

2018 DSE

(iii) What is meant by the 'R_f value' of a substance in a paper chromatogram ?

(2 marks)

2016 DSE Marking

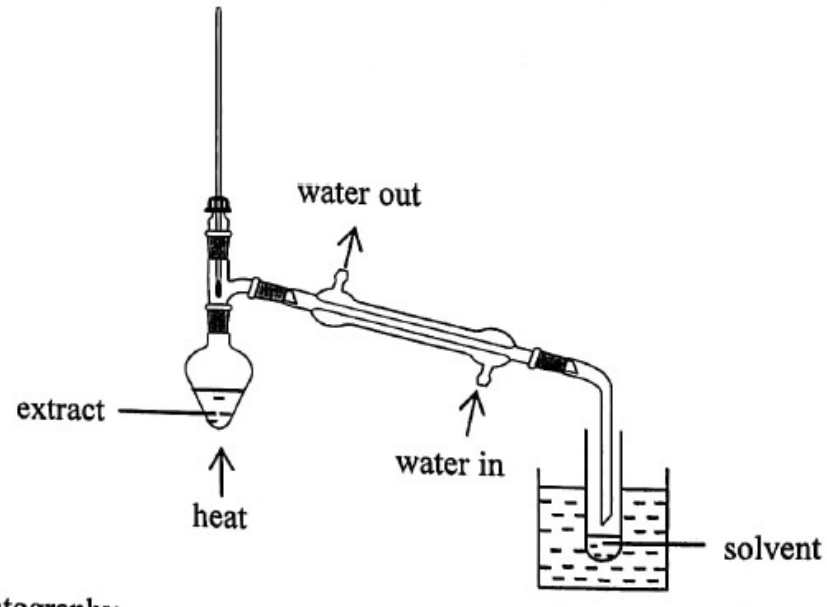
(ii) (1)
- $Na_2CO_3(aq)$ is added to the solution of **X** and **Y** in dichloromethane. 1
- The mixture is shaken in a separating funnel. 1
  The mixture in the separating funnel is allowed to settle, and the aqueous layer is then separated from the organic layer.
- Dilute $H_2SO_4(aq)$ is added to the aqueous layer until no more precipitate is formed. 1
- Solid **Y** can be obtained by filtration. 1

(2) Measure the melting point of the solid obtained.
If the melting point of the solid is 77°C, it may be pure compound **Y**. 1

2017 DSE marking

(c) (i) (1) The solvent will not lose during heating. 1

(2) 2



(3) column chromatography 1

2018 DSE marking

R_f = Distance travelled by the _____ / Distance travelled by the _____

The value of R_f depends on the _____ with mobile phase and _____ with stationary phase.

