1 Experimental chemistry

Content

1.1 Experimental design

1.2 Methods of purification and analysis

1.3 Identification of ions and gases

It is expected that any course in chemistry will be based on experimental work. Teachers are encouraged to develop appropriate practical work for candidates to facilitate a greater understanding of the subject. Candidates should be aware of the hazards and appropriate safety precautions to follow when handling the reagents mentioned in this section.

Learning outcomes

Candidates should be able to:

1.1 Experimental design

(a) name appropriate apparatus for the measurement of time, temperature, mass and volume, including burettes, pipettes, measuring cylinders and gas syringes

(b) suggest suitable apparatus, given relevant information, for a variety of simple experiments, including collection of gases and measurement of rates of reaction

1.2 Methods of purification and analysis

(a) describe methods of purification by the use of a suitable solvent, filtration and crystallisation, distillation and fractional distillation, with particular references to the fractional distillation of crude oil, liquid air and fermented liquor

(b) suggest suitable methods of purification, given information about the substances involved

(c) describe paper chromatography and interpret chromatograms including comparison with 'known' samples and the use of R_f values

(d) explain the need to use locating agents in the chromatography of colourless compounds

(e) deduce from the given melting point and boiling point the identities of substances and their purity

(f) explain that the measurement of purity in substances used in everyday life, e.g. foodstuffs and drugs, is important

1.3 Identification of ions and gases

(a) describe the use of aqueous sodium hydroxide and aqueous ammonia to identify the following aqueous cations: aluminium, ammonium, calcium, chromium(III), copper(II), iron(II), iron(III) and zinc (formulae of complex ions are **not** required)

(b) describe tests to identify the following anions: carbonate (by the addition of dilute acid and subsequent use of limewater); chloride (by reaction of an aqueous solution with nitric acid and aqueous silver nitrate); iodide (by reaction of an aqueous solution with nitric acid and aqueous silver nitrate); nitrate (by reduction with aluminium and aqueous sodium hydroxide to ammonia and subsequent use of litmus paper), sulfate (by reaction of an aqueous solution with nitric acid and aqueous barium nitrate), and sulfite (by warming with dilute acid)

(c) describe tests to identify the following gases: ammonia (using damp red litmus paper); carbon dioxide (using limewater); chlorine (using damp litmus paper); hydrogen (using a burning splint); oxygen (using a glowing splint) and sulfur dioxide (using acidified potassium manganate(VII))

(d) describe a chemical test for water

Chemistry O level

WITH REVISED SYLLABUS FROM 2017 ONWARD & ALONG WITH SOLUTION OF IMPORTANT QUESTIONS FROM PAST PAPERS

By : Rashid Majeed (M.Sc. Chemistry)

0300-4815012

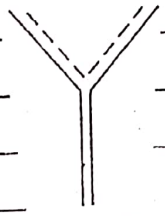
Experimental Design:

Chemical apparatus, their use and accuracy

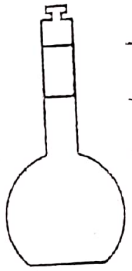
Beaker
used for measuring fixed volume of liquids.

Evaporating dish
To concentrate a dilute solution

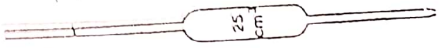
Funnel
To transfer liquid smoothly



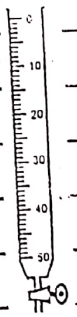
Volumetric flask
To prepare solution of exact concentration



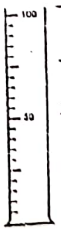
Pipette:
To measure fixed volume of liquid
e.g. $25cm^3$



Burette:
To measure volume of liquid to an accuracy of $0.1cm^3$

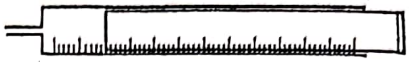


Measuring cylinder
To measure volume of liquid to an accuracy of $1cm^3$.



Syringe

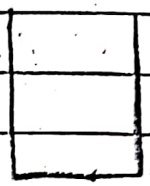
Measures volume of liquid or gas to an accuracy of $0.1 cm^3$ or $1 cm^3$.



Gas Jar

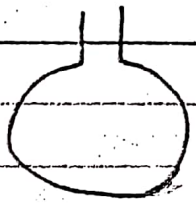
To collect gases

Gas Jar →



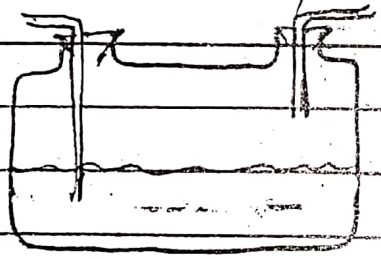
Round bottom flask

To bring about those reactions which produce gases.



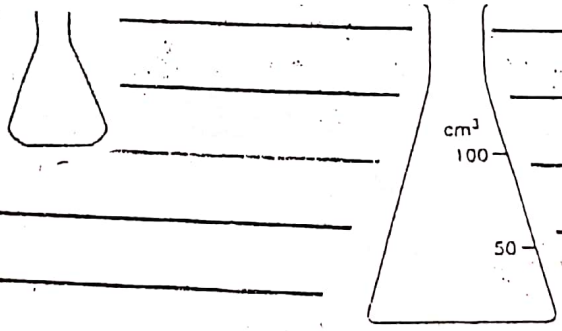
Wolf's bottle

To dry moist gases by using concentrated sulphuric acid.



Titrating flask or conical flask

To bring about acid-alkali reaction, it is useful because solution does not splash due to its narrow neck on shaking.



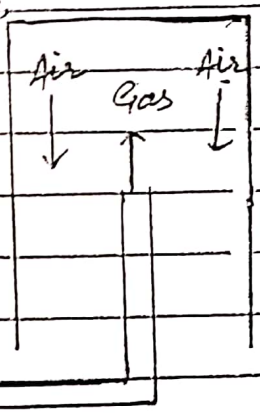
Some common gases and their properties

Gas	Mass(H_2)	Density as compared to air	Solubility in water
Hydrogen (H_2)	2	Lighter	Insoluble
Methane (CH_4)	16	"	"
Oxygen (O_2)	32	Equal to air	Slightly soluble
Ammonia (NH_3)	17	Lighter	Soluble
Carbon dioxide (CO_2)	44	Heavier	"
Hydrogen chloride (HCl)	36.5	"	"
Chlorine (Cl_2)	71	"	"
Sulphur dioxide (SO_2)	64	"	"

Methods of Collection of gases

Collection of lighter, water soluble gases

Lighter substances always settle themselves above heavier substances, as water is always above honey, so lighter gases are collected by upward delivery of gas and downward displacement of air.

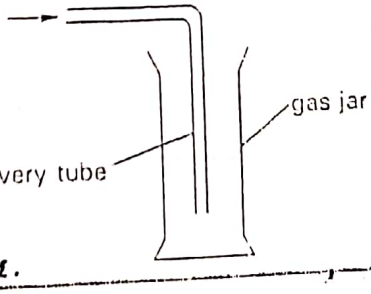


NH_3 or O_2,

Collection of Heavier gas

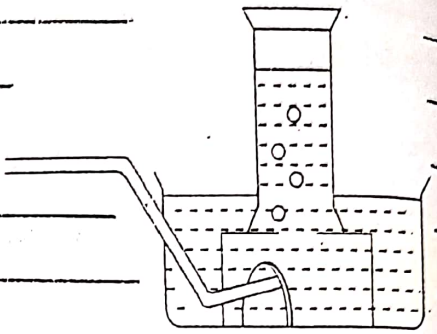
$Cl_2, HCl, CO_2 \rightarrow$

Heavier gases are collected by downward delivery of gas and upward displacement of air as honey always settles at the bottom of water.



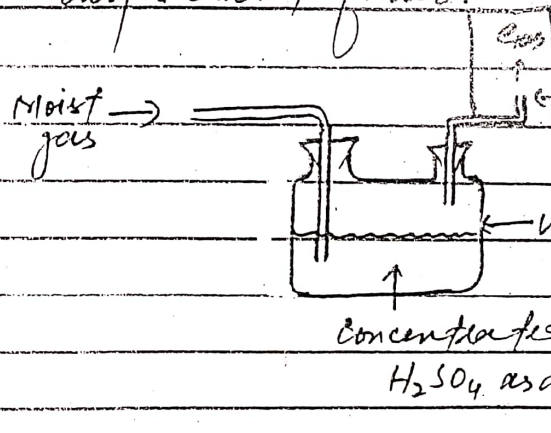
Collection of lighter and water insoluble gases

Water insoluble gases e.g; H_2 and CH_4 are collected by the upward delivery of gas and downward displacement of water.



Collection of dry gases (water insoluble)

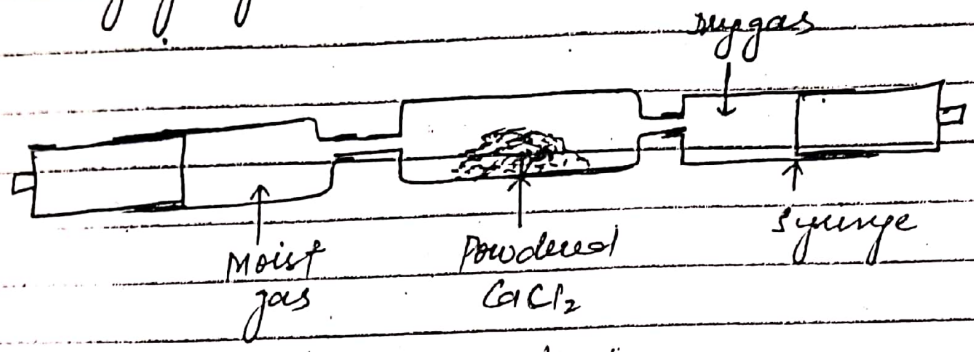
Gases which are insoluble in water and contains water vapours are dried by passing moist gas through concentrated sulphuric acid and then is collected by upward delivery of gas and downward displacement of air.



NH_3 and other water soluble gases e.g; SO_2, CO_2, Cl_2 and HCl cannot be dried by this by this method as they in the solution of concentrated sulphuric acid (H_2SO_4)

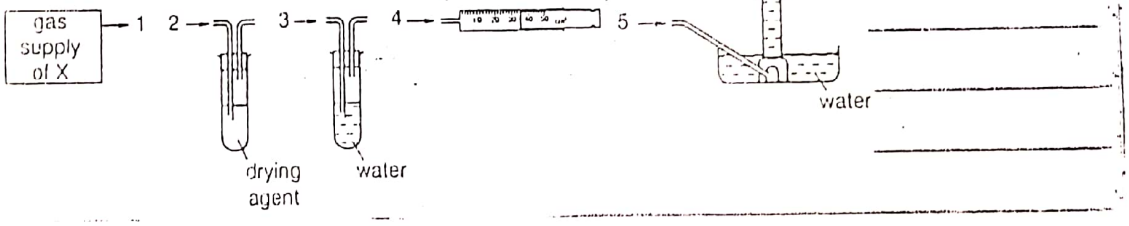
Drying of water soluble gases

water soluble gases e.g; NH_3, Cl_2, HCl, SO_2 etc are dried by using Calcium Chloride $CaCl_2$ as drying agent.



Past paper question

Q:- A gas X is insoluble in water and less dense than air. An impure supply of X contains water vapours and a water soluble impurity.



In which order should the pieces of apparatus be joined together to collect a pure dry sample of X?

(A) 1, 2, 3, 4 (B) 1, 2, 3, 5 (C) 1, 3, 2, 5 (D) 1, 3, 2, 4

Answer "D"

Passing 1 through 3 removes any water soluble impurities. Step 2 is required to dry X. Finally Step 4 is used to collect the dry gas.
Step 5 will make the dry gas to moist again.

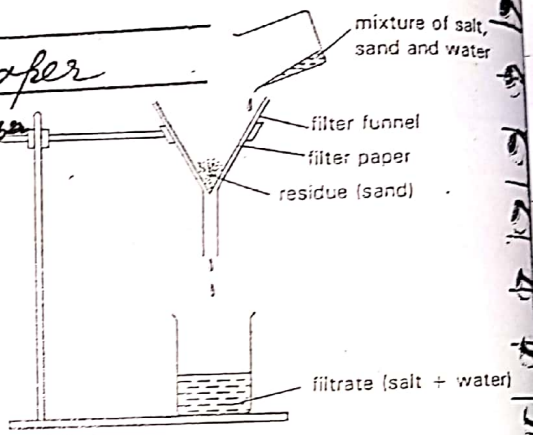
Methods of purification or separation techniques

Filteration

This technique is used to separate an insoluble solid from a liquid.

e.g. Sand + water

Experimental procedure



⇒ Fold a piece of filter paper and place it into a filter funnel

⇒ Pour the mixture into the filter funnel

⇒ Allow the liquid to pass through a filterate.

⇒ The solid (sand) remains on the filter paper as residue.

Uses of filteration

This technique can separate a mixture of two solids in which one is soluble and other is insoluble in a liquid e.g mixture of sand and salt.

Crystallisation

Crystallisation is used to separate pure solids in the form of crystals from the impurities suspend in the solution.

Procedure

⇒ The impure solid is dissolved in the solvent.

The salt solution is filtered leaving the solid impurities behind
The salt solution is heated until a saturated solution is formed.
The saturated solution is cooled to form crystals that can be dried on a filter paper.

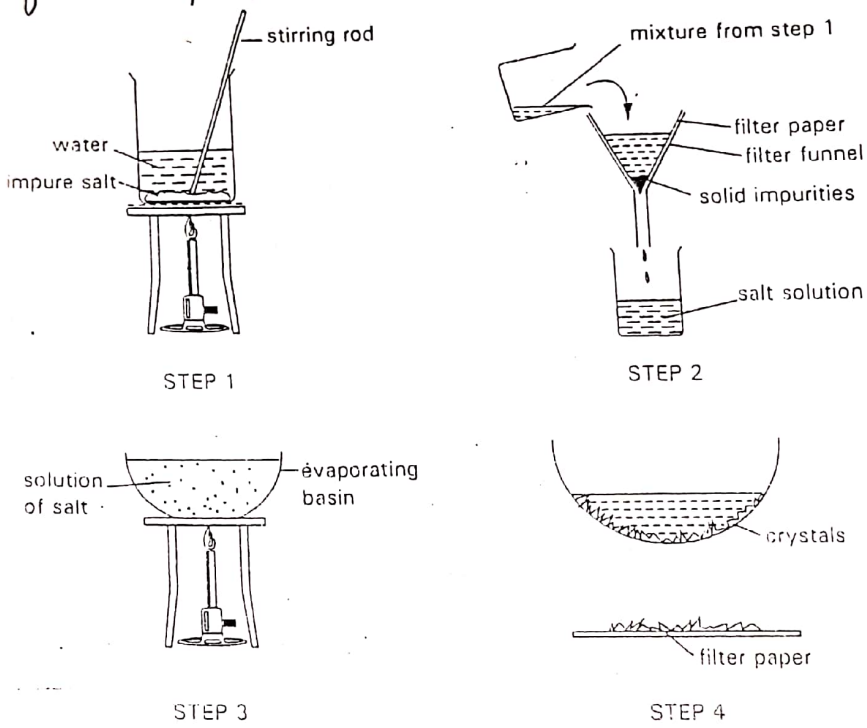
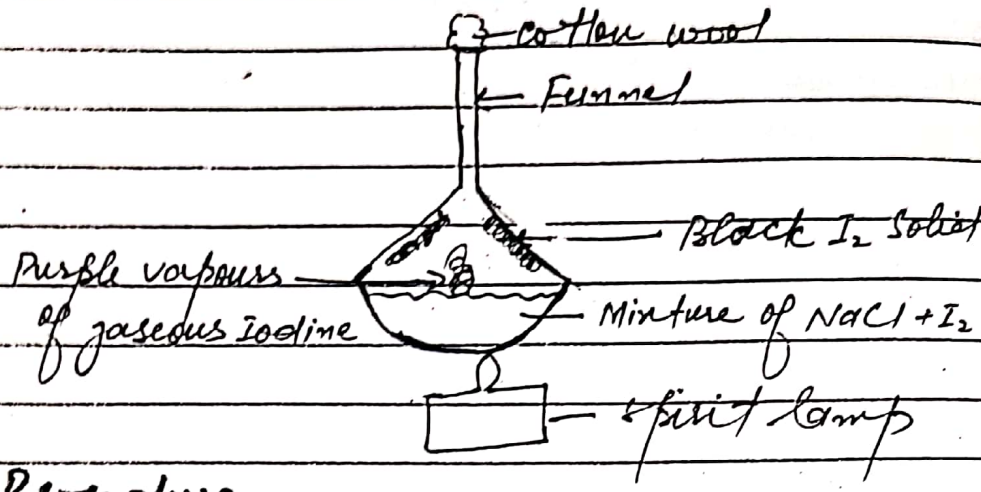


Fig 1.5 Crystallisation

Sublimation

This technique is used to separate a mixture of two solids in which one can sublime, e.g. mixture of NaCl and Iodine, mixture of NaCl and NH_4Cl etc

Definition:- Sublimation is a process in which a solid on heating is converted into gas and a gas on cooling is converted into solid without being converted into liquid.



Procedure

⇒ Mixture of NaCl and Iodine is heated in an evaporating dish.

⇒ Iodine is converted to gas and is condensed on the funnel which is inverted over evaporating dish

⇒ NaCl cannot sublime, remains in the evaporating dish.

Distillation

This technique is used to separate a pure liquid (solvent) from a solution of a solid and liquid.

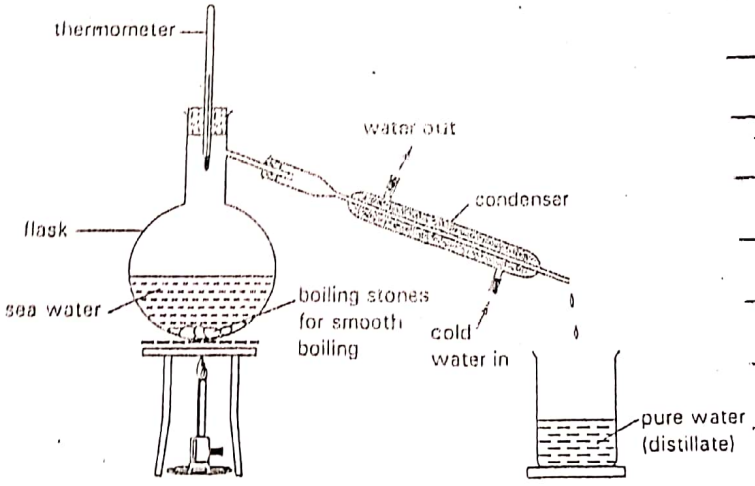


Fig 1.6 Distillation

Experimental procedure

⇒ Solution is heated in a flask
⇒ On boiling thermometer reads a constant temperature and droplets of liquid are collected in the beaker
⇒ Discard the first few drops of liquid collected as they have air molecules dissolved in it.
⇒ water in should be from the lower side and out should be from the upper side of the condenser so that cold water should remain in the condenser for a longer time and will be more effective in condensing.
⇒ Mouth of the receiving flask should remain open otherwise vapour pressure will be built up which will slow down the flow of liquid in the flask or may cause the receiving flask to burst.

use of distillation

To obtain pure water from sea water.

Fractional distillation

⇒ This technique is used to separate a mixture of two or more miscible liquids with different boiling points.
⇒ Separation occurs due to the difference in the boiling points of liquids.
⇒ Liquids with close boiling points cannot be separated by using this technique.

Experimental set up

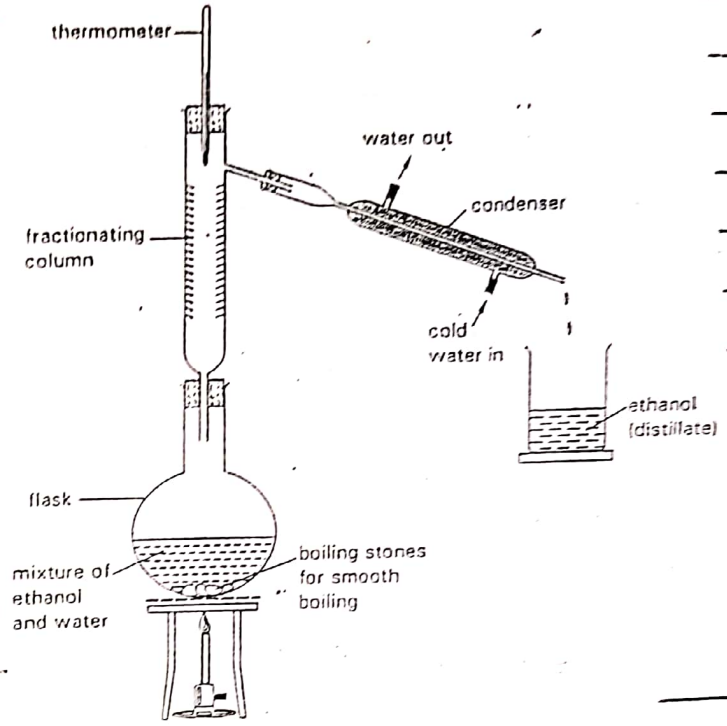


Fig 1.7 Fractional distillation

Experimental procedure

⇒ The miscible liquids are heated in a flask

⇒ The liquid with lower boiling point boils first and thermometer shows a constant temperature

⇒ Droplets of liquid are collected in the receiving flask.

⇒ Replace receiving flask with an empty receiving flask when temperature starts rising after remaining constant to collect the 2nd liquid.

Use of Fractionating column

It is used to separate mixture of liquids by providing them large surface area for condensation.

Uses of fractional distillation

⇒ For the separation of O_2, N_2 and Ar from Air.
⇒ For the refining of crude oil
⇒ For the separation of fermented liquor into ethanol and water.

Chromatography

Khromatos — Colour
Graphy — writing

In Ancient time this technique was used to separate mixture of coloured substances but now this technique can be used to
⇒ separate mixture of coloured and colourless substances both
⇒ For the identification of substances
⇒ For the determination of purity of substances.

Principle of Chromatography

⇒ Chromatography separates substances due to the difference in their solubility in a given solvent.
⇒ The more soluble the substance is, the faster it will dissolve in the solvent.
⇒ If the solvent is flowing on a medium, the more soluble substance will get carried further by the solvent ahead

compared to the less soluble ones.

Paper Chromatography

There are two types of paper chromatography
Ascending Chromatography
Descending Chromatography

Comparison of Ascending & Descending Chromatography

Ascending Chromatography	Descending Chromatography
⇒ Solvent level is below the sample or starting line	⇒ solvent level is above the sample or starting line.
⇒ Solvent moves slower due to gravitational pull	⇒ solvent move faster as it moves towards gravity.
⇒ separate is complete and clear	⇒ Separation is not very clear
⇒ shorter chromatography paper is used	⇒ Longer paper is used.

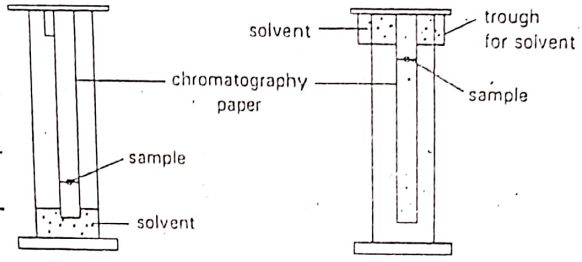


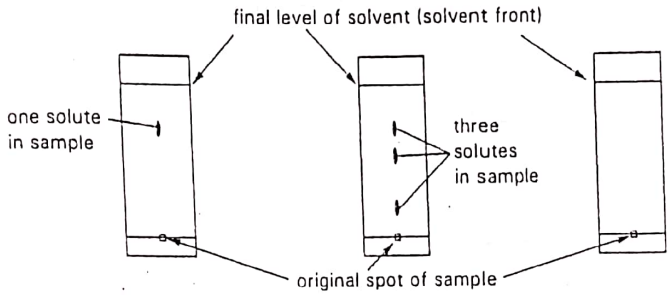
Fig 1.10 Ascending and descending chromatography

Chromatogram

After experiment, the chromatography paper with result is called chromatogram.

Types of Chromatograms

There are three types of chromatograms that can be developed, which are given below



1. Chromatogram of a pure substance
2. Chromatogram of a mixture
3. Chromatogram of no soluble substance in solvent

Solvent front

The distance travelled by the solvent from the starting line is called solvent front.

Rf value (Retention factor)

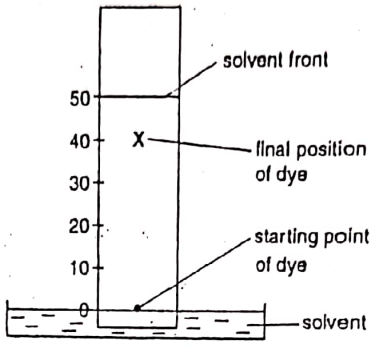
Rf value helps in the identification of substances.

It is measured by using following formula.

$$Rf = \frac{\text{Distance travelled by the substance}}{\text{Distance moved by the solvent}}$$

$$Rf \text{ of } P = \frac{8}{46} = 0.17$$

The diagram shows the chromatogram for a dye



Which fraction shows the R_f value for the dye?

(A) $\frac{10}{50}$ (B) $\frac{50}{100}$ (C) $\frac{50}{40}$ (D) $\frac{40}{50}$

Ans is D

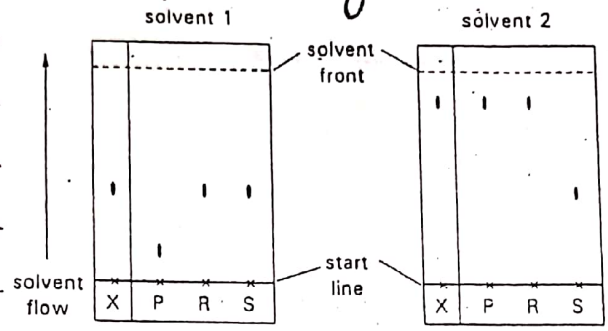
Distance travelled by the dye is 40 and by the solvent is 50

Identification of colourless substances (use of locating agent)

For converting colourless substances to coloured compounds locating agent is sprayed onto the chromatogram, this helps to locate the position of colourless substances. For example a locating agent "ninhydrin" is used in detecting the presence of amino acids.

Past paper question

Substance X contains one of the three substances P, R or S. Two chromatograms of the four substances were obtained using different solvent. The diagram shows the results.



What does X contain

(A) P only (B) R only (C) Either P or R
(D) Either R or S

Answer B

The first chromatogram confirms the absence of P while the 2nd confirms the absence of S. Hence by combining the conclusions from both chromatograms, S and P are not present in X.

Uses of Chromatography

⇒ used by the forensic scientists in the investigation of crimes.
⇒ used to test the purity of food substances.