Chapter No. 2

EXPERIMENTAL TECHNIQUES IN CHEMISTRY

TEXT BOOK EXERCISE

q.1. Multiple choice questions.

i. Filtration process could be very time consuming if it were not aided by a gentle suction which is developed:

(a) if the paper covers the funnel upto its circumference.
(b) if the paper has got small sized pores in it.
(c) if the stem of the funnel is large so that it dips into the filtrate.
(d) if the paper fits tightly.

ii. During the process of crystallization, the hot saturated solution:

(a) is cooled very slowly to get large sized crystals
(b) is cooled at a moderate rate to get medium sized crystals
(c) is evaporated to get the crystals of the product.
(d) is mixed with an immiscible liquid to get the pure crystals of the product.

iii. Solvent extraction is an equilibrium process and it is controlled by.

(a) law of mass action
(b) the amount of solvent used
(c) distribution law
(d) the amount of solute
iv. Solvent extraction method is a particularly useful technique for separation when the product to be separated is:

(a) non-volatile or thermally unstable
(b) volatile or thermally stable
(c) non-volatile or thermally stable
(d) volatile or thermally unstable

v. The comparative rates at which the solutes move in paper chromatography depend on:

(a) the size of paper
(b) Rf-value of solutes
(c) temperature of the experiment
(d) size of the chromatographic tank used

Ans: (i) d  (ii) b  (iii) c  (iv) d  (v) b

Q.2. Fill in the blanks.

i. A complete chemical characterization of a compound must include __________.

ii. During filtration the tip of the stem of the funnel should touch the side of the beaker to avoid __________.

iii. A fluted fuller paper is used to _________ the process of filtration.

iv. A solvent used for crystallization is required to dissolve __________ of the substance at its boiling point and __________ at the room temperature.

v. Repeated solvent extractions using small portions of solvent are __________.

Ans: (i) qualitative & quantitative analysis (ii) splashing (iii) increase (iv) large amount, small amount (v) more efficient

Q.3. Tick the correct sentences. If the sentence is incorrect, write the correct statements.
i. A qualitative analysis involves the identification of elements present in a compound.

ii. If the process of filtration is to run smoothly, the stem of the funnel should remain empty.

iii. If none of the solvents is found suitable for crystallization, a combination of two or more immiscible solvents may be used.

iv. A solute distributes itself between two immiscible liquids in a constant ratio of concentrations depending upon the amount of solvent added.

v. Paper chromatography is a technique of partition chromatography.

Ans: (i) true (ii) false (iii) true (iv) false (v) true

Q.4. Why is there a need to crystallize the crude product?
Ans: When a compound is prepared in laboratory, it may contain impurities. This impure and un-refined compound is called crude product. It is necessary to purify the crude product. So there is a need to crystallize the crude product.

Q.5. A water insoluble organic compound aspirin is prepared by the reaction of salicylic acid with a mixture of acetic acid and acetic anhydride. How will you separate the product from the reaction mixture?
Ans: Aspirin is separated from reaction mixture is added to cold water. The aspirin forms crystals and other products remain in solution. Finally aspirin is filtered from water by sintered glass crucible.

Q.6. A solid organic compound is soluble in water as well as in chloroform. During its preparation, it remains in aqueous layer. Describe a method to obtain it from this layer.
Ans: The organic compound can be extracted by solvent extraction. The aqueous solution of compound is mixed with carbon tetrachloride (CCl₄). The mixture is put into the separating funnel. Here two layers are formed. The water layer is separated and evaporated to get the compound.
Q.7. The following figure shows a developed chromatogram on paper with five spots. (i) Unknown mixture X  (ii) Sample A (iii) Sample B  (iv) Sample C  (v) Sample D
Find out (i) the composition of mixture X  (ii) which sample is impure & what is its composition.

Ans: The unknown mixture X contains the components B and C. The sample D is impure. It contains components A and C. We should know that a pure sample give only one spot. The samples A and C are pure.

Q.8. In solvent extraction technique, why repeated extraction using small portions of solvent are more efficient than using a single extraction but larger volume of solvent?

Ans: The solvent extraction technique is based upon distribution coefficient. The ratio of concentration of a solute dissolved in two immiscible solvents is a constant. It is called distribution coefficient.

In repeated extractions, we can get maximum amount of solute from the other solvent. It is the reason that repeated extractions using small portions of solvent are more efficient than using a single extraction but larger volume of solvent.

Q.9. Write down the main characteristics of a solvent selected for crystallization of a compound.

Ans: The process of crystals formation is called crystallization. When a hot saturated solution of a substance is cooled, then crystals of solid substance separate out. It is called crystallization. This process is used for purification of solid compounds. E.g. Purification of Naphthalene. The process of crystallization involves the following steps.

i. **Choice of Solvent:**
A suitable solvent is choosen by hit and trial method. An ideal solvent has following properties

i. It should dissolve maximum amount of solute.
ii. It should not react with solute.

iii. It should not dissolve the impurities.

iv. On cooling, it should form pure crystals

v. It should be cheap (in-expensive)

vi. It should be easily removable.

vii. Its use should be safe

viii. An inflammable solvent should be heated on water bath. The most common solvents are water, rectified spirit, absolute alcohol, ether, chloroform, acetone, acetic acid, carbon tetra chloride.

ii. **Preparation of Saturated Solution:**
The substance is dissolved in minimum amount of solvent. To dissolve more and more solute, the solution should be heated directly or on water bath. The stirring of solution is necessary.

iii. **Filtration:**
The hot saturated solution is filtered to remove insoluble impurities. Sometimes premature crystals can form. It can be prevented by using hot water funnel.

iv. **Cooling:**
The hot filtered solution is cooled. The moderate cooling gives the medium sized crystals and slow cooling gives bigger crystals. Sometimes a few crystals of pure solid are added in solution to start the crystallization process.

v. **Collecting the Crystals:**
When the crystallization is complete, then mixture of crystals and mother liquor is filtered through Gooch crucible using a vacuum pump. The mother liquor is removed completely by full suction. Then the filter cake is pressed to remove the rest mother liquor. Finally the crystals are washed with small amount of cold solvent. This process is repeated many times till pure crystals are formed. By evaporation of the mother liquor we can get a fresh crop of crystals.

vi. **Drying of Crystals:**
There are three methods for drying the crystals.
a) Press the crystals between the folds of filter paper. Repeat this process many times method crystals are crushed and fibers of filter paper mix with them.

b) Dry the crystals at 100°C in an oven. Here solid should not melt or decompose at 100°C.

c) Spread the crystals over watch glass and keep in a vacuum desiccators for many hours. The drying agents used in desiccators are CaCl₂, P₂O₅, Silica gel.

vii. **Decolourization of Undesirable Colours:**
Sometimes crystals become coloured due to impurities. To prevent it, the substance is boiled with animal charcoal. Animal charcoal absorbs the coloured impurity. The pure colourless crystals are formed by cooling the filtrate.

Q.10. **Write down the procedure to separate the mixture of three inks with the help of paper chromatography.**

Ans: Take a 20cm strip of whatman filter paper No 1. The strip is 2.5cm wide. Mark a pencil line 2.5cm from lower end. A spot of mixture inks is placed on pencil line. The separate spots of three pure inks are also placed on the pencil line. Then filter paper is suspended into the tank containing solvent. The lower end of filter paper dips 5-6mm into the solvent. When mobile phase (solvent front) rises 3/4 the paper, then remove it. Every ink gives a coloured band. But the mixture gives three bands. The Rₐ values of all bands are calculated. In this way we can separate mixture of three inks.