INTRODUCTION:- The branch of chemistry which deals with chemical characterization of a substance is called ANALYTICAL CHEMISTRY. A complete chemical characterization includes two types of Analysis.

(i) Qualitative Analysis (ii) Quantitative Analysis

QUALITATIVE ANALYSIS:- It is concerned with determination of nature of substance. In this type of analysis we are concerned with determination of which elements are present in a compound.

QUANTITATIVE ANALYSIS:- In this type of analysis we are concerned with determination of amounts of different elements present in a chemical compound.

STEPS IN QUANTITATIVE ANALYSIS:- It involves four major steps (i) Obtaining a sample for analysis
2) Separation of desired constituent
3) Measurment and calculation of result
4) Drawing conclusion from analysis

Q:- 2:- WRITE A NOTE ON FILTRATION PROCESS.

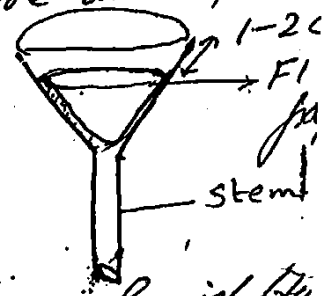
It is a process by which insoluble particles are separated from liquid. It can be performed by different types of filter media. The nature of precipitates, ~~nature of solvent~~ and nature of solvent determine which type of filter media should be used. The most commonly used filter media are (1) Filter paper
2) Filter Crucibles.

FILTER PAPERS:- Filtration by glass funnel and filter paper is a slow process. When mixture of liquid and solid particles is poured on to the filter paper, the solvent passes through filter paper and suspended particles are left behind as residue. The liquid which passes through filter paper is called FILTRATE

CHARACTERISTICS OF FILTER PAPER: The filter papers have different pore sizes. The choice of filter paper depends upon size of particles in the precipitate.

SIZE OF FILTER PAPER: — The size of filter paper should be such that it should be one fourth to one half full of precipitate at the end of filteration.

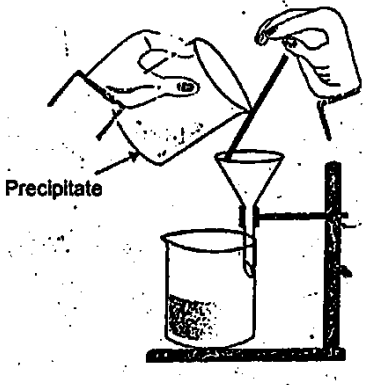
SIZE OF FUNNEL: The size of funnel should be such that its upper rim is 1—2 cm above the top circumferance of filter paper



STEM OF FUNNEL: The stem of funnel should be several inches long so that it can extend a few centimeters down into the receiver beaker. The tip of stem should touch with walls of beaker so that liquid runs down along the side of beaker without splashing. Smoothly the stem of funnel should remain continuously fi of liquid, as long as there is liquid in conical portion

HOW TO FOLD FILTER PAPER: The paper should be fol twice. The first fold is along diameter of paper. Nat sec fold is such that edges do not quite match. The paper should be opened on slightly larger sect there is three fold thickness on one side and one thickness on other side. The apex angles is slightly grea than 60°.

The paper is moistened with water and pressed down in to 60° funnel,

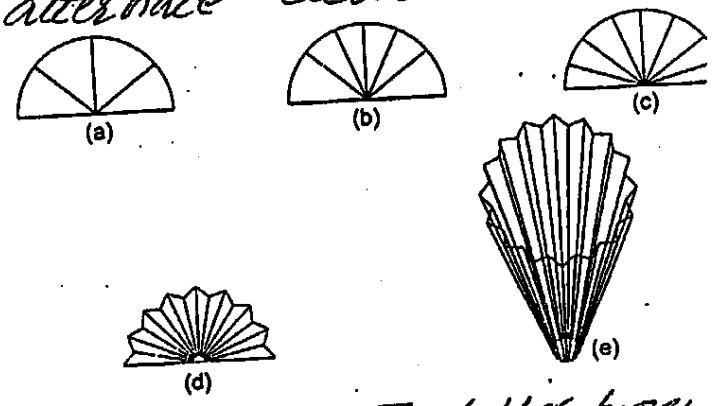


TIME OF FILTERATION: Filteration process is quite time consuming. However it is aided by gentle suction, produced when liquid passes down the stem of funnel. For this suction the

should closely attached with walls of funnel.

FLUTED FILTER PAPER

The rate of filteration could be increased by using fluted filter papers. For this purpose ordinary filter paper is folded in such a way that there is fan like arrangement with alternate elevations and depressions.



The rate of filteration is slightly increased by using fluted filter paper.

FILTER CRUCIBLES

It is method to ~~filtered~~ filter precipitate through suction. Two types of crucibles are used

GOOCH CRUCIBLE It is made of porcelain. It has a perforated bottom. The bottom is covered with paper pulp or filter paper. The Gooch crucible is placed in suction filtering apparatus.

ADVANTAGES It is useful for filtering precipitates which are to be ignited at high temp.

If perforations are covered with Asbestos mat then it may be used to filter solution which react with ppt. For example solution containing Conc HCl, $KMnO_4$, etc.

SINTERED GLASS CRUCIBLES Sintered glass crucible is a glass crucible with porous glass disc sealed into bottom. It is quite easy to use because no preparation is required for using it.

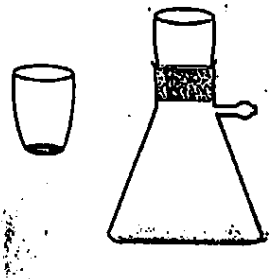


Fig. (2.3a) Gooch Crucible with filtering apparatus

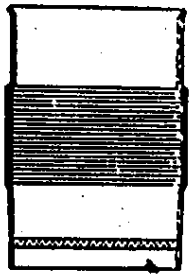


Fig. (2.3b) Sintered glass Cruci

CRYSTALLIZATION:-

NEED OF CRYSTALLIZATION:- In most of the synthetic reactions, chemical compound is obtained in an impure form. It is purified by crystallization from suitable solvent. Thus Crystallization is used to purify crude products

BASIC PRINCIPLE:- The solute should be more soluble in a suitable solvent at its B.P. When solution is cooled the solute should be separated as crystals

The process of fetteration involes following steps.

CHOICE OF SOLVENT:- The solvent is chosen on HIT and TRIAL BASIS. A number of solvents are tried before reaching a conclusion

CHARACTERISTICS OF SOLVENT:- An ideal solvent should posses following characteristics

1:- It should dissolve a large amount of solute at its boiling point and only a small amount at room temperature

2:- It should not react chemically with solute

3:- It should not dissolve impurities. If impurities are soluble then they should not crystallize out along with desired substance when solution is cooled.

4: On cooling solution, well formed crystals of pure compound must be obtained

5:- It should be inexpensive

6:- It should be safe to use and it should be easily removable.

COMMONLY USED SOLVENTS:- The solvents which are mostly used as for crystallization are WATER, ABSOLUTE ETHANOL, DIETHYL ETHER, RECTIFIED SPIRIT (95% ETHANOL), ACETONE, CHLOROFORM, CARBONTETRACHLORIDE, ACETIC ACID, PET. PETROLEUM ETHER

COMBINATION OF SOLVENTS

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

If solvent is inflammable, water bath is used for heating purposes.

2. **PREPARATION OF SATURATED SOLUTION**:- The substance is heated on water bath or burner with minimum amount of solvent to prepare saturated solution. Add more solvent to boiling solution, if required, until all solute has dissolved

3. **FILTRATION**:- The hot saturated solution is filtered through a normal or fluted filter paper. The insoluble impurities are removed as residue. The premature crystallization during filteration is avoided by using hot filter funnel, saturated solution or by using hot water funnel

4 **COOLING**:- The hot filterate is cooled at a moderate rate so that medium size crystals are formed. V. Slow cooling produce large size crystals which carries larger amount of solvent containing impurities in it. Further, larger size crystals are difficult to dry.

5- **COLLECTING THE CRYSTALS**:- The mixture of liquid and crystals is filtered through Gooch crucibles using a vacuum pump. Full suction is applied to remove mother liquor from crystals. When filter cake is rigid, it is pressed with a cork to remove remaining liquid.

WASHING OF CRYSTALS:- The crystals are then washed with a small amount of cold solvent. The process of washing is repeated several time.

The filterate (mother liquor) is concentrated by evaporation, and second crop of crystals is obtained by cooling. The success of process depends upon % of crystallized product.

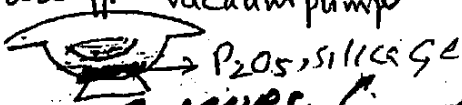
6 **DRYING OF CRYSTALLIZED PRODUCT:** There are three methods of drying crystals.

BY PRESSING the crystals between several folds of filter paper. The process is repeated several times

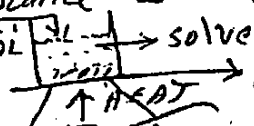
DISADVANTAGE: 1) The crystals are crushed to a fine powa
2) Fibres of filter paper contaminate produc

BY USING OVEN The crystals are dried in an oven provide the substance does not melt or decompose on heating at 100

VACUUM DESICCATOR Its a safe and reliable method The crystals are spread over a watch glass. These are placed in a vacuum desiccator for several hours. The drying agent used in a desiccator are $CaCl_2$, Silicagel or Phosphorous pentoxide (P_2O_5).



7 **DECOLOURIZATION OF UNDESIRABLE COLOURS:** Some cr undesirable coloured due to some impurities. The crys are boiled with sufficient quantity of animal charco The coloured impurities are adsorbed by animal charcoal. The pure decolourized substance is crystallized out from cooling filtrate.

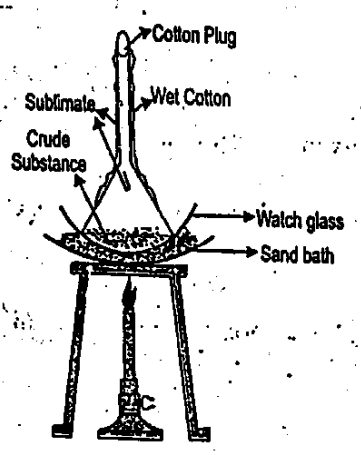


WRITE A NOTE ON SUBLIMATION:

DEFINATION: The process in which a solid is converted directly into vapours without passing through liquid phase is called sublimation. When vapours are cooled they are directly changed into solid. separation of volatile solids from non volatile to purify a solid. For

EXAMPLE AND USES:- It is used example Ammonium Chloride, Benzoic acid, naphthalein, iodin can be purified by Sublimation

PROCESS: The substance is taken in a watch glass. It is covered with an inverted funnel. The funnel is covered with wet



cotton to keep it cool. The substance is heated slowly over a sand bath. The solid vaporizes and the vapours condense as solid on inner walls of funnel.

In a better method, the process is carried out in a cold finger.

SOLVENT EXTRACTION:- The process in which a substance is transferred from one solution to another immiscible solvent is called

BASIC PRINCIPLE According to this technique a solute can be separated from a solution by shaking solution with an immiscible solvent. The solute should be more soluble ~~solvent~~ solute in that solvent.

PROCEDURE:- Usually it is done by placing the solution and second solvent in a separating funnel. The funnel is stoppered and two liquids are shaken together.

EXAMPLE The most common laboratory example of solvent extraction is ether extraction. This is used to separate the product of organic synthesis from water. The aqueous solution of organic compound containing some inorganic impurities is taken in separating funnel. Then some ether is added to it. The mixture is shaken and layers are allowed to separate. The organic compound mostly go into ether layer and impurities remain in aqueous layer. The ether layer is separated. Same process is repeated with aqueous layer once again. It is more suitable to use small portions of ether several times rather than using all of the volume just once. The ether layer is easily evaporated and organic compound is obtained. This technique is particularly useful when product is volatile or thermally unstable.

DISTRIBUTION LAW Solvent extraction is an equilibrium process and it is controlled by distribution law or partition law which states that;

A mathematically constant ratio exists between Concentration of a given compound in two immicible solvents. This ratio is independent of amount of solute added. mathematically it could by written as $\frac{C_1}{C_2} = K$ "K" - This law is based on experimental evidence.

DISTRIBUTION OF IODINE BETWEEN AQUEOUS SOLUTION OF KI AND CARBON TETRACHLORIDE

In aqueous solution of KI, iodine react with I^{1-} ion to form triodide ion, in a reversible reaction. The following equilibrium is established

$$\underset{\text{soluble in } CCl_4}{I_2} + \underset{aq}{I^{1-}} \rightleftharpoons \underset{\text{(soluble in water)}}{I_3^{1-}}$$

At this point the rate at which iodine passes from CCl_4 to aq. layer, becomes equal to rate at which it passes from water to CCl_4.

Thus when CCl_4 is added to brown coloured solution of I_3^{1-} ion in water, the brown colour of aq. solution fades and a purple colour appears in CCl_4 layer due to I_2.

For good separation, the two liquids are gently shaken to increase area of contact of two layers. The ratio of concentration of iodine in two layers remains constant. It is independent of amount of iodine added. This constant is called distribution co-efficient denoted by K

$$K = \frac{[I_2 \text{ (in } CCl_4)]}{[I_2 \text{ as } I_3^{1-} \text{ (in Aq)}]}$$

CHROMATOGRAPHY:-

The word chromatography originates from KHROMATOS meaning coloured writing. It is an important application of distribution phenomenon. It is used for separation of sample mixtures.

PRINCIPLE:- The solute distributes itself between a stationary phase and mobile phase. On the basis of stationary phase chromatography may be divided into two types

A **ADSORPTION CHROMATOGRAPHY** The chromatography in which stationary phase is a solid is called Adsorption chromatography. The solid may be supported as a thin film on the surface of an inert solid. The substance leaves the mobile phase and get adsorbed on the surface of solid phase.

B **PARTITION CHROMATOGRAPHY** Chromatography in which stationary phase is a liquid is called Partition Chromatography. The substance being separated distribute themselves throughout between ~~the~~ stationary phase and mobile phase.

MOBILE PHASE The mobile phase flowing over the surface of stationary phase may be liquid or gases

In chromatography substances are separated due to their relative affinities for stationary and mobile phases. The separation of the components of mixture is based upon their distribution co-efficient "K" which is different for each component

$$K = \frac{\text{Conc. of component in mobile phase}}{\text{Conc. of component in stationary phase}}$$

SMALL K VALUE:- The component with small "K" value mostly remains in stationary phase as mobile phase passes over it

LARGE K VALUE:- The component with large "K" value remains dissolved in mobile phase and passes over the stationary phase quickly.

There are different techniques ~~of~~ chromatography. Most commonly used is paper chromatography

PAPER CHROMATOGRAPHY: It is a type of PARTITION CHROMATOGRAPHY. Both mobile phase and stationary phases are liquids.

STATIONARY PHASE:- Stationary phase is water adsorbed on paper. The adsorbed water is immiscible liquid to mobile phase which paper passes over it.

MOBILE PHASE:- The mobile phase is usually an organic liquid. The nature of solvent depends upon sample mixture being separated.

The paper chromatography may be (1) ASCENDING (2) DESCENDING (3) RADIAL/CIRCULAR

In ascending chromatography solvent is taken in a pool at the bottom of chromatographic tank. The lower end of paper dips in solvent. The solvent travels upwards by capillary Action.

PROCEDURE The solvent mixture is taken in chromatographic tank. Cover the tank to homogenise its inner atmosphere.

Take about 20 cm strips of whatmans' Chromatographic paper no. 1. Draw a thin pencil line about 2.5 cm from one end.

The sample mixture is applied in the form a spot on pencil line, with a capillary Tube. The spots of pure components may also be applied along sides of sample spot on same line.

The spots are dried.

The paper is suspended with clips such that the impregnated end dips into the solvent mixture but base line remains outside the liquid.

Cover the tank to homogenise inner atmosphe

The solvent rises due to capillary action. When the solvent passes the spots the solute begins to move upward. The rate at which spots move upwards, depend upon their distribution co-efficients.

When the solvent front has moved $\frac{3}{4}$th length of the paper, the strip is removed. The solvent front is marked with pencil. The strip is allowed to dry

Once the paper is dried, the pattern is called Chromatogram. If component of mixture are coloured they can be easily identified. If compone are colourless, the chromatogram has to be develope by chemical method or physical method and spots are identifie

Each component has a specific retardation factor called "R_f" value

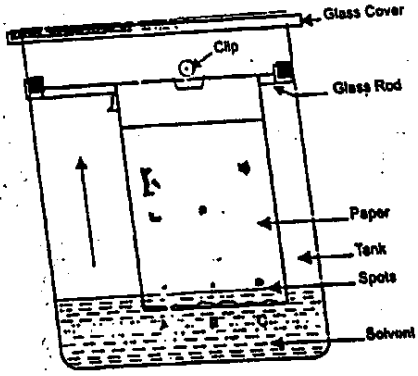
The "R_f" value is related to its distribution co-efficien.

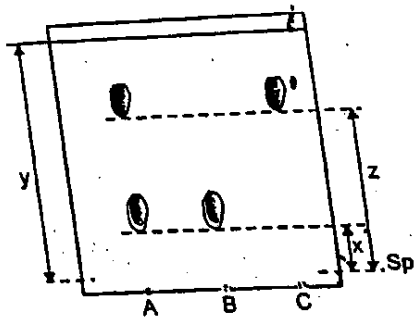
$$R_f = \frac{\text{Distance covered by component from original spot}}{\text{Distance covered by solvent.}}$$

Consider following diagram. It shows that mixture contain components A and B. Their R_f values are

$$R_{f(B)} = \frac{x}{y}$$

$$R_{f(A)} = \frac{z}{y}$$





Uses:- ① It is quite useful for identification, separation isolation and purification of products of organic Syntheses.

2) It is used to identify purity of a substance

3) It is equally important for qualitative and quantitative analysis.

GIVE SHORT ANSWERS TO FOLLOWING QUESTIONS.

1:- WHAT ARE ADVANTAGES OF FILTERATION BY CRUCIBLE:

FILTERATION BY CRUCIBLE HAS FOLLOWING ADVANTAGES:-

1 **RATE OF FILTERATION:-** RATE OF FILTERATION IS GREATLY INCREASED BY USE OF VACUUM PUMP.

2. **IGNITION OF RESIDUE:-** THIS METHOD IS USEFUL IF RESIDUE IS TO BE IGNITED AT HIGH TEMP.

3. **FILTERATION OF REACTIVE CHEMICALS:-** AS PORES OF CRUCIBLES ARE COVERED WITH ASBESTOS SHEET, IT CAN BE USED TO FILTER ACID, BASE, OXIDIZING AGENTS WHICH REACT WITH PAPER.

4. **EXTRA ADVANTAGE OF SINTERED GLASS CRUCIBLE.**

SINTERED GLASS CRUCIBLE REQUIRE NO PAPER OR ASBESTOS SHEET. NO PRELIMINARY PREPARATIONS ARE REQUIRED.

QNO. 2

WHY ETHER IS MOSTLY USED IN SOLVENT EXTRACTION

ANS:- ETHER IS MOSTLY USED DUE TO FOLLOWING PROPERTIES

2) **IMMISCIBLE:-** ETHER IS IMMISCIBLE WITH WATER. IT FORMS UPPER LAYER.

(ii) **ORGANIC COMP. MORE SOLUBLE:-** ORGANIC COMPOUNDS ARE MORE SOLUBLE IN ETHER THAN WATER.

(iii) **INORGANIC IMPURITIES INSOLUBLE:-** IN ORGANIC IMPURITIES ARE INSOLUBLE IN ETHER. THEY REMAIN IN AQ. LAYER.

(iv) **EASY TO EVAPORATE:-** IT IS EASY TO EVAPORATE ETHER DUE TO ITS LOW B.P.

Q. 3.

WHAT IS DISADVANTAGE OF LARGE SIZE CRYSTALS

ANS:- LARGE SIZE CRYSTALS ARE PRODUCED IF HOT SATURATED SOLUTION IS COOLED VERY SLOWLY. IT HAS FOLLOWING DISADVANTAGES:

2) LARGE SIZE CRYSTALS CONTAIN MORE SOLVENT CONTAINING IMPURITIES IN IT.

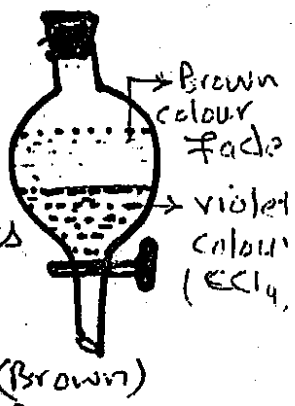
(ii) IT IS DIFFICULT TO DRY LARGE SIZE CRYSTALS.

HENCE HOT SATURATED SOLUTION IS COOLED AT MODERATE RATE TO GET MEDIUM SIZE CRYSTALS.

Q NO. 4

WHAT HAPPENS WHEN CCl_4 IS ADDED TO Aq. SOL OF I_2.

WHEN CCl_4 IS ADDED TO AQUEOUS SOL OF IODINE BROWN COLOUR OF AQ. SOLUTION FADES AWAY AND PURPLE COLOUR APPEARS IN CCl_4. IODINE MOVES FROM AQ. SOLUTION TO CCl_4. FOLLOWING EQUILIBRIUM IS ESTABLISHED



$$I_2 + I^{1-}_{aq.} \rightleftharpoons I_{3\,aq}^{1-}\,(\text{Brown})$$

I_2 in CCl_4 (violet).

DISTRIBUTION CONSTANT "K"

$$K = \frac{\text{CONC. OF } I_2 \text{ IN } CCl_4}{\text{CONC OF } I_2 \text{ AS } I_3^{1-} \text{ IN } H_2O}$$

Q NO. 5

WHAT IS DEVELOPING OF CHROMATOGRAM?

IF COMPONENT OF MIXTURE ARE COLOURLESS, THEY CAN BE MADE VISIBLE BY PHYSICAL OR CHEMICAL METHAD IT CALLED DEVELOPING OF CHROMATOGRAM.

EXAMPLE: IF MIXTURE OF AMINO ACIDS ARE SEPARATED BY CHROMATOGRAPHY, THEY ARE DEVELOPED BY **NINHYDRIN**. Pb^{2+}, Cd^{2+} IONS ARE MADE VISIBLE BY H_2S.

Q NO. 6

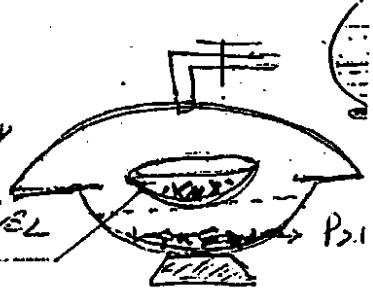
WRITE FUNCTION OF FOLLOWING APPARATUS/CHEMICALS

HOT WATER FUNNEL: IT IS USED TO AVOID PREMATURE CRYSTALLIZATION DURING FILTERATION OF SATURATED SOLUTION

COLD FINGER: IT IS USED FOR SUBLIMATION PROCESS

ANIMAL CHARCOAL: IT IS USED TO ADSORB COLOURED IMPURITIES AND DECOLOURIZE CRYSTALS.

SEPARATING FUNNEL: IT IS USED IN SOLVENT EXTRACTION TO SEPARATE TWO IMMISCIBLE LIQUIDS.



VACUUM DESSICATER: IT IS USED TO DRY CRYSTALS IT CONTAINS P_2O_5/$CaCl_2$/SILICA GEL CRYSTALS ARE PLACED IN WATCH GLASS.

Q:. WHY THERE IS NEED OF CRYSTALLIZATION? IS

WHENEVER A COMPOUND IS PREPARED IN LABORATORY, IT IS OBTAINED IN CRUDE FORM. IT CONTAINS SOME SIDE PRODUCTS AND MAY ALSO CONTAIN UNREACTED REACTANTS IT IS PURIFIED BY CRYSTALLIZATION. MOST OF IMPURITIE AND COLOURED COMPOUNDS ARE REMOVED BY CRYSTALLIZA

Q:- SEPARATION OF ASPRIN? (EXERCISE Q NO: 5).

ASPRIN IS SEPARATED BY FILTERATION. REACTION MIXTU IS POURED IN ICE COLD WATER. ~~IT~~ ASPRIN SOLIDIFIES WHILE OTHER COMPONENTS REMAIN IN SOLUTION FOR IT IS SEPARATED BY SUCTION FILTERATION BY GOOCH CRUCIBLES OR SINTERED GLASS CRUCIBLE

Q: EXTRACTION OF ORGANIC COMPOUND FROM AQUEOUS LAYER? (EXERCISE Q NO. 6)

SOLUBLE ORGANIC COMPOUND CAN BE SEPARATED BY SOLVENT EXTRACTION. AQUEOUS SOLUTION IS TAKEN IN SEPARATING FUNNEL SOME CHLOROFORM IS ADDED IT. IT IS WELL SHAKEN. COMPOUND GOES FROM AQ. SO TO CHLOROFORM MIXTURE IS ALLOWED TO STAND CHLOROFORM LAYER IS REMOVED AND ALLOWED TO EVAPORATE. PURE COMPOUND IS OBTAINED IN SOLID STATE.

REPEATED SOLVENT EXTRACTION? EXERCISE Q NO. 8

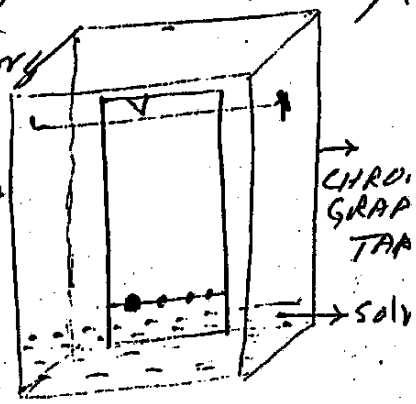
EACH TIME SOLUTE DISTRIBUTES ITSELF BETWEEN TWO IMMISCIBLE SOLVENTS IN CONSTANT RATIO OF CONC. MORE THE NO. OF TIMES, SOLVENT EXTRACTION IS CARRIED OUT MORE COMPOUND CAN BE EXTRACTED HENCE REPEATED EXTRACTIONS ARE MORE EFFICIEN

SEPARATION OF MIXTURE OF INKS.

MIXTURE OF INKS CAN BE SEPARATED BY CHROMATOGRAPHY.

PROCEDURE

(i) TAKE A CHROMATOGRAPHIC TANK CONTAINING SOME ORGANIC LIQ (ETHANOL — H_2O). COVER THE TANK TO HOMOGENIZE INNER ATMOSPHERE

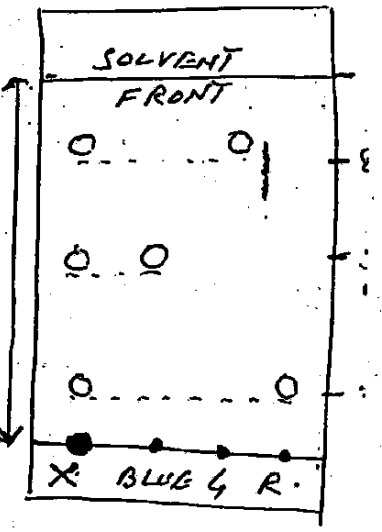


(ii) TAKE A STRIP OF WHATMANN'S FILTER PAPER NO. 1. DRAW A LINE WITH LEAD PENCIL 2.5 cm FROM ONE END.

(iii) THE SAMPLE MIXTURE IS APPLIED IN THE FORM OF A SPOT ON LEAD PENCIL LINE WITH CAPILLARY TUBE.

(iv) THEN PLACE SPOTS OF PURE "RED" "BLUE" & GREEN INKS ON SAME LINE.

(v) WHEN SPOTS ARE DRIED, SUSPEND THE PAPER IN CHROMATOGRAPHIC TANK IN SUCH A WAY THAT PAPER END IS DIPPED IN SOLVENT BUT BASE LINE REMAINS OUTSIDE.



CHROMATOGRAM.

(vi) SOLVENT RISES DUE TO CAPILLARY ACTION. PURE INKS AND MIXTURE ALSO RISES

(vii) WHEN SOLVENT HAS ARISEN $\frac{3}{4}$ OF PAPER, REMOVE PAPER, MARK SOLVENT FRONT.

(viii) DISTANCE COVERED BY SOLVENT AND EACH COMPONENT IS DETERMINED AND R_f VALUE OF EACH IS DETERMINED

$R_f = \frac{\text{DISTANCE OF COMP.}}{\text{DIST. COV. BY SOLV.}}$

S. No	COLOUR	DIST. OF COMPONENT	DIST. OF SOLVENT	R_f	
1	BLUE	5 cm	10 cm	$\frac{5}{10} = 0.5$	
2	GREEN	8 cm	10 cm	$\frac{8}{10} = 0.8$	
3	RED	3 cm	10 cm	$\frac{3}{10} = 0.3$	