Purification of organic compounds pdf

Purification of organic compounds by crystallization practical. Purification of organic compounds by sublimation. Purification of organic compounds by recrystallization. Purification of organic compounds neet questions. Purification of organic compounds ppt. Purification of organic compounds ncert pdf. Purification of organic compounds pdf. Purification of organic compounds class 11.

Here are some of the important methods that are commonly used for the purification of organic compounds: filtration The filtration process is used to separate the insoluble solid component of a mixture from soluble components in a given solvent. It is used to separate a mixture of naphthalene and urea using water as a solvent. The urea dissolves in the water while the naphthalian remains - insoluble. After filtration, the naphthalian remains on the filter paper while the urea is recovered from the filtrate to evaporate the water. For example: a mixture of anthracene and benzoic acid is separated by dissolving the mixture in hot water and filtering the hot solution. Benzoic acid dissolves in hot water but the anthracene no. On filtration, the anthracen remains as residue on the filter paper while benzoic acid is crystallized by the cooling filtered. Filtration is very slow and takes a long time. In such cases, filtration is performed at reduced pressure using a buhner funnel and a water suction pump. The crystals of crystallization or richistallization are the most pure form of a substance with defined geometric shapes. The process with which an impure compound was converted into its crystals is known as crystallization. It is based on the difference in the solubilities of the compound and impure in a suitable solvent. The impure mixture is dissolved in a suitable solvent in which it is with a soluble parsimony at room temperature but sensitively at greater temperature. The solution is concentrated to obtain almost a saturated solution. When this saturated solution is cooled, pure substance crystals are although they will be removed from filtration. The filtered, ie, the liqueurs of the mother contains the impure together with a small amount of the mixture. If the mixture is highly soluble in a solvent and too little soluble in another solvent, therefore the crystallization can be performed in a mixture of these solvents taken in a suitable relationship. The main steps of this process are explained below: (a) choice of solvent (a) should not react chemically with the impure substance. (b) should dissolve more than the substance to heating compared to room temperature so that the excess of the substance is thrown out of cooling. (c) or the incurities should not dissolve at all in the solvent, or if they dissolve, they should be soluble to such a point that remain in the solution, ie, in the mother's liqueur to crystallization. The various solvents that are commonly used for crystallization are water, alcohol, chloroform, carbon tetrachloride, benzene, acetone, ethyl acetate, oil ether etc. (2) Preparation of the solution. The impure substance is finely powdered and therefore heated in a conical flask with a small amount of the solvent that is sufficient to dissolve all the substance on the hot. (3) Solution filtration. The hot solution obtained above is then filtered immediately both through the grooved filter paper or through hot water funnel to avoid crystallization during filtration. (4) Crystallization and separation of crystals. The plate containing the solution is then allowed to cool undisturbed when after some time crystals start to separate. If the crystals do not appear even after a long time, it may be necessary to scratch the sides of the plate with a glass rod. This process of induction of rear crystallization by adding a crystal of pure substance into its saturated solution is called sowing. (V) drying of crystals. The crystals are finally dried in the air or placing them in a steam oven or in an air oven. (vi) Color removal. Sometimes, the crystals obtained are slightly colored due to the presence of certain colored impure. In Cases, the crystals are resisted in the same solvent and a small amount of activated carbon is added. The mixture is boiled for 15-20 minutes. During this treatment, coal coal absorbs all colored impure. Coal coal is Filtered and the filtered is allowed to cool when obtaining colorless crystals of the pure substance. The crystallization of sugar. Suppose you have a sugar sample containing commonness of common, salt (sodium chloride). This can be purified for stirring of solid impure with hot ethanol at 348 K. The sugar dissolves that the common salt remains insoluble. The warm solution is filtered, concentrated, and then left to cool when the sugar crystals will separate out. The crystallization of benzoic acid. Suppose we have a mixture of benzoic acid and naphthalene mixture, Mixture.This can be purified by treatment of solid impure with hot water. Benzoic acid dissolves while naphthalene remains insoluble. The hot solution is filtered and then left to cool when the benzoic acid crystals separate. .The crystals are separated by filtration and dried. Framed crystallization This method is used to separate and purify two or more compounds that have different solubilities, in the same solvent. The mixture is dissolved in a suitable solvent in which the two components of the mixture have different solubilities. When a hot saturated solution of this mixture is allowed to cool, the less soluble substance crystallizes first while the substance remains more soluble in the solution. The crystals are then separated from mothers and focuses again and the warm solution again left to cool when the crystals of the second (ie, more soluble) composed of state. The process of separating the different components of a mixture by repeated crystallisations is known as fractional crystallization. Sublimation it involves the direct conversion of a solid to the gaseous heating state without going through the intervention of liquid and vice versa for cooling. Only those substances whose vapor pressure became equal to the atmospheric pressure very bephore, respective melting points are able to suffer sublimation. These substances are sublimable called. The sublimation process is very useful in the purification of such solids that sublimate on heating, and corresponding to non-volatile impurities. The impure substance is taken in a porcelain plate covered with a perforated paper filter on which an inverted funnel is placed.The stem of the funcut is closed with a small cotton.On heat the plate on a sand bath, vapors Of the solid volatile up hollow up, go through the filter and condensate paper holes on the cold walls by leaving the non-volatile impurities into the plate. Canfora, naphthalene, anthracene, benzoic acid, iodine, etc. are purified by this process. Simple distillation distillation implies the conversion of a steam liquid by heating followed by the condensation of the vapors as well produced by cooling. The method is commonly used for liquids that are sufficiently stable at their boiling points and containing non-volatile impurities. For example: a simple organic liquids such as benzene, ethanol, acetone, chloroform, carbon tetrachloride, toluene, xylenes, etc. can be purified for simple distillation. Procedure (1) When the muffle is heated, the temperature gradually increases and the liquid begins to boil when its vapor voltage becomes equal to atmospheric pressure. These vapors passing through the condenser are condensed. Only the liquid that distills at a constant temperature is collected in a receiver. This gives us pure liquid. Purification of a mixture of liquids. It can also be used for the separation and purification of a mixture of two or more potential organic liquid available their boiling points differ from 30-50 K. The separation is based on the Which to the BP of the most volatile liquid (low boiling) of the mixture, the vapors constituted almost exclusively of the most volatile liquids. At the boiling point of liquid less volatile, vapors almost entirely constituted less volatile liquid after the most volatile volatile It has already distiled over.The separation of the liquid mixture in individual cabin elements, obtainable respective boiling point, the distils more volatile components than the first while the distils less volatile components above later. The impurity and liquid impure having a lot of boiling point very high, of those of the two non-volatile fluids separated above are, however, to the left in the bulletproof distillation: (1) A mixture of ether (BP 308 K) and Toluene (BP 384 K) (2) a mixture of Esano (BP 342 K) and Toluene (BP 384 K), (3) Benzene mixture (BP 353 K) or chloroform (BP 334 K) and Anilina (BP 457 K) and so on . Framed distillation If the two liquids of the mixture are very close to each other, ie differ from 10 k or so Bravia separation cannot be obtained by means of the simple distillation method as described above. This is due reason that at B.P. The most volatile liquid of the mixture there will be sufficient liquid vapors less volatile so. As a result, both liquids of a mixture will distill together and the separation is denied. The separation of this liquid mixture in individual components can be obtained by fractional distillation, which involves repeated distillations and condensations. Framed distillation is performed using a fractionation column. LT usually consists of a long glass tube to a large hole, both packed with glass pearls, stones, porcelain or coke or blownã rings, in a number of spherical or pear bulbs. The actual purpose of the fractionation column is to increase the cooling surface and to provide obstacles or obstructions to ascending and descending liquid vapors. Principle: Ã, suppose we have a mixture of two fluids A and B of which AA is more volatile than B.When a mixture is heated, the temperature rises slowly and begins boiling mixture. The vapors form mainly constituted by the most volatile liquid with little of the less volatile liquids these vapors travel on a fractioning column, the vapors of the less volatile condenses b liquid more readily, of those of the most volatile liquid aa therefore, the vapors that Salt BecomeÃà, rich in a and flowing liquid becomes Richerã, B. This process of distillations and condensations is repeated at each point of the Thea splittingle column. As a result of the series of subsequent distillations, from the moment vapors reach the upper column thea and the escape in the condenser, are mainly constituted by more volatile A. Component similarly, after a series of subsequent distillations, the remaining liquid in the distillation of Getsã, enriched in A greater boiling component. Each condensation and subsequent vaporization is called theOnetic plate ,. Commercially, columns with hundreds of theoretical dishes are available. Applications: Ã (a) It is to separate crude oil in the oil industry in various useful fractions such as petrol, kerosene, diesel oil, lubricating oil, etc. (b) fractionated distillation has also been used for separate acetone (BP 329) and alcohol Methyl (BP 338 K) Distillation under reduced pressure or vacuum distillation This method is used for the purification of fluid liquids and liquid that decomposes to now under their boiling points. Principle: a liquid bubbles when its vapor voltage becomes equal to the pressure. It, outer the liquid itself would boil at lower temperature if the pressure acting on it is reduced. Since the liquid now boils at a lower temperature, its decomposition does not occur. With vacuum pumps, pressure of the order of 0.1 mm HG can be easily obtained. For example: glycerol that decomposes to its boiling point (563 k) can be distilled without decomposition to 453 k less than 12 mm HG pressure. Canna concentration Sugar in the sugar sector. Steam distillation This is a convenient method for separation and purification of compounds, biological from non-volatile volatile Or inorganic impurities. This method is applicable only ThoseãããÃ, compounds, which are steam volatile, insoluble in water, has a steam pressure of about 10-15 mm to 373 KA and contain non-volatile impure. Steam distillation is particularly important when the substance to be cleaned upper than 373 K at 760 mm and decomposes in correspondence or belowÃ, its boiling distillation Point.Steam makes the substances to distill at low temperature and therefore avoids their decomposition. Principle: In this method, a mixture of two immiscible liquids, ie water and a heated ISA organic liquid. Each one would exercise its vapor voltage regardless of the other and the mixture will begin to boiling at a temperature in which the sum of the vapor tensions of the organic liquid (P1) and that of the water (P2) a becomes equal to atmospheric pressure (P). P = P1 + P2 unless the water vapor pressure or the organic liquid is zero, the temperature to which Mix theea bubbles must be lower than the normal boiling point both of the organic liquid and water. In terms other ¢, the organic liquid bubbles at a temperature lower than its normal B.P and therefore the ISA decomposition avoided. The partial pressures of different gases in a mixture, gaseous are in a relationship of their relative number of moles. Therefore, the relationship between the number of moles of the organic liquid and water in the distillate must be in the relationship of their partial vapor tensions (in the boiling mixture,), ie. N1 / N2 = P1 / P2 where N1 and N2 are no. of moles and, P1 and P2 the partial pressures of the organic liquid and water. Leave W1 and W2 be the respective masses of organic liquid and water in the distillate. Then, N1 = W1 / M N2 = W2 / 18 where M is the molecular mass of the organic liquid. (W1 / m) A (18 / W2) = P1 / P2 O (W1 / W2) A bis (P1A Ãƒ m) / (P2A Ãƒ 18) equation gives the relative masses of the organic liquid water and in the distillate. Consider the following examples for purely illustrative purposes. 1) a mixture of nitrobenzene (BP 483 K) and water (boiling point 373 K) bubbles at 372.3 k during steam distillation. At this temperature, Nitrobenzene's vapor voltage is 20 mm and water is 760 mm. 2) a mixture of water (373 bp k) and aniline (bp 457k) bubbles at 371 k at 760 mm pressure ina vapor distillation.ã, the impure organic compound mixed with water is taken in a bottomed round ball and steam comes past. The mixture begins to boil when the combined vapor voltage becomes equal to atmospheric pressure. At this temperature, mixed vapor with vapors of the Overa compound to the condensate where they are condensed and collected in the receiver. The distillate contains water and a desired substance that can be easily separated with the help of a separator funnel. Some of the compound that can be purified through this process are o-nitrophenol, bromobenzene, ã, aniline, nitrobenzene, essential oils, turpentine oil, etc. This method is used to recover organic substances (solid or liquids) from their aqueous solutions. The process essentially provides for the agitation of the organic compound in AA funnel separator with a suitable solvent that is immiscible with water, but in which the organic compound is highly soluble. Ether, benzene, chloroform, carbon tetrachloride are some of the solvents that are generally used for extraction. Procedure The aqueous solution is mixed with a small amount of organic solvent in a separator funnel. The funnel is stupered, and the content is roughly agitated for times when the organic solvent dissolves the organic compound present in the aqueous solution. The funnelÃ, separation is now left to rest for some time when the organic solvent and water form two distinct layer.theã, lower aqueous layer is exhausted by opening the of the hank and the separate organic layer. The aqueous solution is paid again in the funnel, augarÃ, mixed with a small amount of organic solvent and the process is Times up to the entire quantity of the organic compound is extracted. Organic layers from all sizes are taken in a distillation balloon. The organic solvent is distilled, away leaving the organic compound in the distillation balloon. The efficiency of the extraction process depends on the number of times the extraction is repeated. With a quantity of solvent, the greater the number of extractions, the greater the quantity of material extract. This method is generally applicable to non-volatile compounds. For example, benzoic acid beau can extracted from its aqueous solution with benzene. Chromatography This method is widely used for separation, purification, identification and characterization, of the components of a mixture, even colored or colorless. The technique to separate the components of a mixture in which the separation is obtained from the differential movement of the individual components through a stationary phase under the influence of a mobile phase. Types of chromatography The stationary phase can be a strictly linked liquid or solid on a solid support while the mobile phase Thea can be liquid or a gas.Deeping on the nature of the stationary and mobile phases, the different types of commonly used chromatographic techniques are: types of Mobile chromatography / stationary phase Uses Adsorption or chromatography on liquid column / solid wide-scale separations Thin layer Liquid chromatography / solid quality analysis High performance Liquid liquid chromatography / qualitative solid and quantitative analysis of gas Liquid chromatography Gas / Qualitative liquid and quantitative analysis Paper or Liquid / liquid chromatography partition is qualitative and quantitative analysis of organic and inorganic compounds. Depending on the principle in question, chromatography can be divided into the following categories: two (a) chromatography adsorption (b) partition chromatography of adsorption chromatography principle. This method is based on the differential adsorption of mixture of various components, on a suitable adsoring such as silica or alumina gel. As some compounds are more strongly, absorbed of the other, they will travel through the column at different speeds and getting so separate. Types of adsorption chromatography Adsorption chromatography is of the following two types: (1) Column chromatography (2) Thin layer chromatography The column chromatography All the process is carried out in a long glass column supplied with aa of the tap at the bottom. The various phases of this process are (1) Column preparation: Ã, a cotton or glass wool buffet is placed at the bottom of the clean and dry glass aa column. Above this, a thin layer of sand washed with acid is placed to support the adsorbent adsorbent adsorbent. Which alumina (AL203), silica gel, magnesium oxide, starch, coal, etc. is Madeea in slurry with a suitable solvent (preferably not -polar) as a petroleum exane or ether. The ISA dough then carefully packed in the column gently touching so that no air bubble is trapped in the column. Thisa constitutes the stationary phase. (2) Adsorption: Ã, the mixture to be separated is dissolved in AA minimum volume of highly polar solvent suitable and applied on the upper part of the adsorbent column, with the help of a dropper or a microsyering. When the solution travels along, the mixture is adsorbed in a narrow band. A thin layer of acid washed sand is again positioned in the upper part of the column followed by a cotton sand layer loose cap from preventing the column from being disturbed during the addition of solvent from time to time. After applying the sample, a small quantity of solvent is placed above the sand layer and the column is allowed to rest for about 15-20 minutes. During this period i Mix components (we say A, B and C) are adsorbed to a different extent depending on their polarity within a narrow band. So, in the narrow band, the component a is The component B is adsorbed moderately adsorbed while the component C is weakly adsorbed. (3) elution. It is the components of the extraction process adsorbed dall'assorbente with the help of solvents of increasing polarity. The solvents usually employed in order of increasing polarity are petroleum ether, carbon tetrachloride, benzene, chloroform, diethyl ether, ethyl acetate, acetone, alcohol, etc. A solvent or a mixture of solvents that is used to extract the column constitutes the mobile phase and is usually called eluent. While the eluent passes along the column, it dissolves the different compounds. The less strongly adsorbed component of the mixture, ie, the component C is eluted first from the less polar solvent followed by moderately strongly adsorbed component B using solvents of intermediate polarity while the stronger adsorbed component A is eluted last from all more solvents high polarity. For example: a mixture of naphthalene (hydrocarbons) and benzophenone can be separated over the column of alumina and using petroleum ether containing benzene as eluent. Naphthalene is less polar is weakly adsorbed while the benzophenone is more polar is strongly absorbed the column. Election of the column will be the first elute naphthalene and then benzophenone. Thin Layer Chromatography (TLC) It is another type of adsorption chromatography in which is obtained the separation of the components of a mixture on a thin layer of an adsorbent. A thin layer of an adsorbent such as silica gel or alumina is spread over a plastic or of an appropriate size glass. (1) is taken a suitable TLC plate and two pencil lines are drawn across the width of the plate approximately 1 cm each ROM ends. The bottom pencil line is called the base line while the upper line is called the line of pull or front of the solvent. (2) A solution of the mixture to be separated is applied as a small dot with the help of a capillary at the starting line. The plate is then placed in a closed jar containing a suitable solvent. (3) Since the solvent © moves, the components of the mixture is also moving along the plate in different distances depending on the degree of adsorption or entity. When the solvent front reaches the finish line, the plate is removed and then dried in the air. (4) The points of the colored components are visible on the TLC plate because of their original color. The points of colorless components which are invisible to the eye can be observed by using the following display methods such as: (i) ultraviolet light: the fluorescisce organic compounds that can be detected by placing the plate under UV lamp having the light of 254 Nm. Since © all organic compounds do not produce fluorescence under UV light, this method is not of general applicability. (Ii) the iodine vapors. This is the most commonly used detection reagent. The developed TLC plate is placed in a covered jar containing a few crystals of iodine. Patches of compounds that adsorb iodine appear as brown spots. (Iii) chemical methods. Sometimes a suitable chemical reagent may be sprayed on the plate. For example, the amino acids can be detected by spraying the plate with ninhydrin solution. Similarly, it is possible to detect Aldehyes / Ketones by spraying the plate with the solution of 2, 4-Dinitrirophilhydrazine. The various components of the developed TLC plate are identified by their retention factor, ie RF values. It is defined as the moment that the front of the solvent moves always faster on TLC plate with respect to the compounds, the RF values, the values are usually expressed as a decimal fraction. Ã partition (or paper) chromatography or TLC chromatography column is a solid liquid chromatography, i.e., the mobile phase is a While the stationary phase is solid. On the contrary, the partition chromatography is a liquid / liquid chromatography in which it is the mobile phase and the stationary phase are liquid in the chromatography of the paper, a special quality card called chromatographic card is used. Although the card consists mainly of cellulose, cellulose, The stationary phase in the chromatography paper is not the cellulose but the water that is adsorbed or chemically bound. The mobile phase is another fluid which usually is a mixture of two or three solvents and water as one of the components. Principle: the paper chromatography works on the principle of partition, ie, is based on differential partitioning (or distribution) of the various components of the mixture between the stationary phases and mobile phases. Process: Ã 1) and a suitable starting line is drawn through the width of the paper to about 1 or 2 cm from the bottom Ã selected a chromatographic paper. (2) A point of the component of the mixture to be separated is applied to the starting line with the help of a syringe or capillary end. The chromatography paper is then suspended in a suitable solvent mixture. (3) The solvent increases the paper by capillary action and flows on the point. The different components of the mixture traveling through different distances depending on their solubility or partitioning between the stationary phases and mobile phases. (4) When the solvent reaches the upper ends of the paper, the paper is removed and allowed to dry. The well developed strip of paper is called chromatogram. The points of the separate colored compounds are visible at different heights from the starting line and are identified by their RF values. (5) The spots of colorless compounds can, however, be observed both under ultraviolet light or with the use of an appropriate spray reagent. In the descending paper chromatography, the solvent is held in a depression at the top of the room, stained the end of the paper is immersed in it and the solvent is allowed to flow down by gravity and capillary action. In this type, the flow of solvent is quick and therefore the process is less expensive in terms of time with respect to the ascending method used. The paper chromatography is particularly used for the separation of sugars and amino acids. acids.