

A Literature Review on the Separation of Chemical Compounds

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Abstract

This literature involved explanation about separation organic components in mixture such as(chromatography ,extraction ,filtration, centrifuge...) , principles of separation , methods of separation ,types of separation , purification of separated compounds , conditions of separation , physical and chemical properties of mixture.

Keywords: Separation, Chemical, Compounds.

Introduction

The separation of chemical compounds depend on differences in physical properties , differences in melting or boiling point , structures of compounds , purity of compounds and classes of chemical compounds. All chemical compounds of biochemical interest occur naturally as components of complex mixtures from which they can be isolated only with considerable difficulty.

Types of Separation

1- Separation by Chromatography

Various separation methods are based on chromatography, that is, separation of the compounds in a mixture through differences in the way they become distributed between different phases. Liquid-solid chromatography was developed for the separation of substances which is colored ,for this it is named name chromatography, which stems from the Greek word ((chroma)) meaning color and ((graphy)) because the method was used for the separation and isolation found in aromatic plants., which described in 1906 by Tswett. This is a chemical method used for the isolation of mixtures into its parts ,purification of components and also to test the purity of components.

The chromatography technique depends on the difference in the rates of the components in a mixture move in a porous medium which named stationary phase but moving phase is solvent or gas for this reason this technique contain from two phases ((a stationary phase of large surface area and second is a moving phase which is allowed to move over stationary phase)) . The stationary phase is either a solid or a liquid but the moving phase may be a liquid or a gas. Chromatography depending on the nature of the stationary phase and the mobile phase, It is constant for a given substance (component) under a given set of conditions. Therefore, it is possible to identify the different compounds through estimation their values.In an extraction, the sample is one phase and we extract the analyte or the interferent into a second phase. We can isolate and separate the components and interferents by continuously passing one sample-free phase, named the mobile phase, over a second sample-free phase that remains fixed or stationary. The sample is injected through the mobile phase and the sample's components partition themselves between the stationary phase and the mobile phase. Those components with larger partition coefficients are more likely to move into the stationary phase, taking a longer time to pass through the

system. This is the basis of all chromatographic separations. Chromatography gives both a separation of components and interferences, and a means for performing a qualitative or quantitative analysis for the analyte.

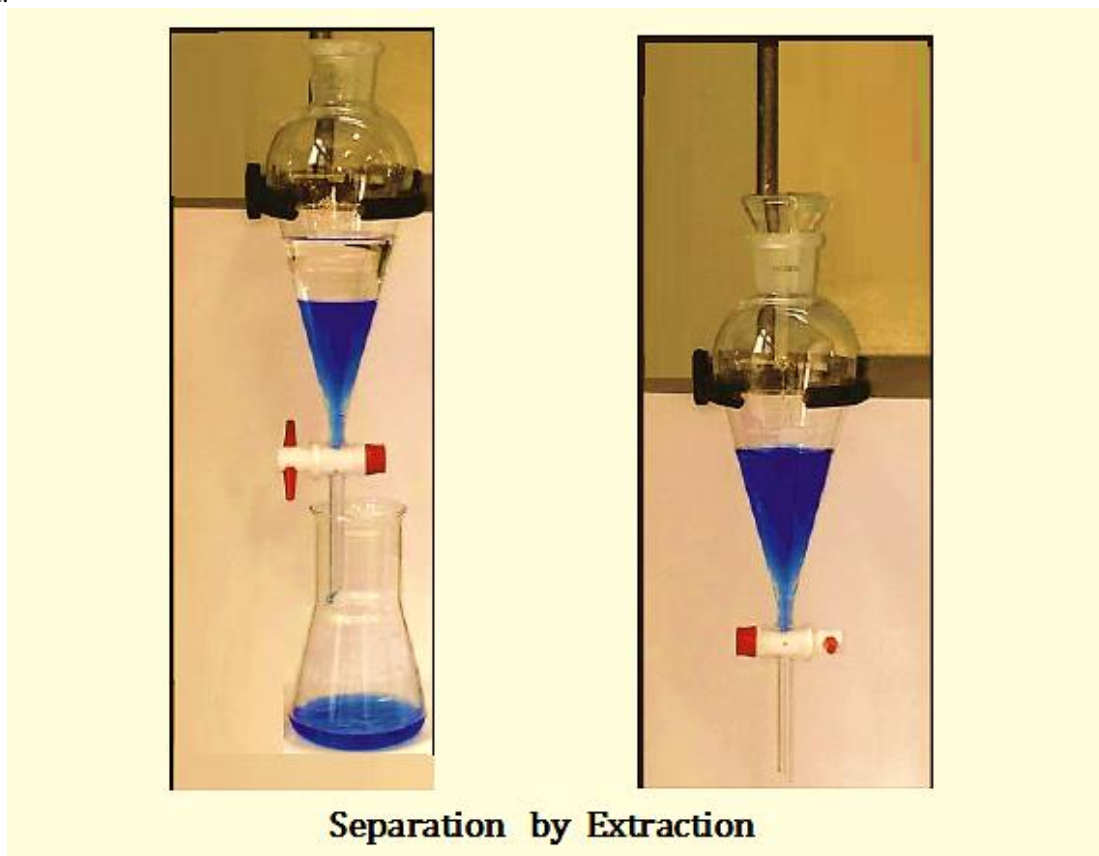
The most common phase pairs used in chromatography are a mobile liquid phase in contact with a solid phase. The liquid phase can be a pure liquid, such as water or an organic solvent, or it can be a solution, such as methyl alcohol, sodium chloride in water, or hexane in toluene. The solid phase can be a continuous material such as paper, or a fine-grained solid such as silica, powdered charcoal, or alumina. The fine-grained solid can also be applied to a supporting material, such as paper, plastic, or glass, to form a coat of continuous material. Alternatively, gas/liquid phase systems can consist of an inert gas, such as nitrogen or helium, in conjunction with a high-boiling point liquid polymer coated on the surface of a fine-grained inert material, such as firebrick. This system is called gas-liquid phase chromatography (GLPC), or simply gas chromatography (GC). In each system, both phases play a role in the separation by offering a physical or chemical characteristic that will result in differential distribution of the components of the analytical mixture being separated. Liquid-liquid phase systems are similar to gas-liquid phase systems in that one of the liquid phases is bound to an inert surface and remains stationary

2-Adsorption Chromatography

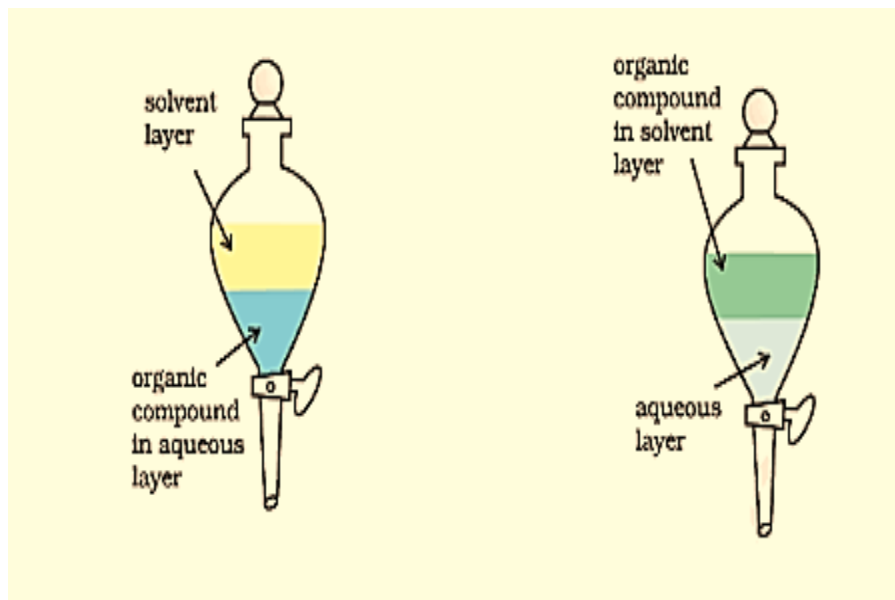
Adsorption chromatography partitions components of a mixture by means of their different adsorption characteristics onto the surface of a solid phase and their different solubilities in a liquid phase. Adsorption phenomena are primarily based on intermolecular interactions between the chemical components on the surface of the solid and the individual components of the mixture. They include van der Waals forces, dipole-dipole interactions, and hydrogen bonds. Silica is a useful adsorption medium because of the ability of its silanol OH groups to hydrogen bond or form dipole-dipole interactions with molecules in the mixture. These forces compete with similar intermolecular interactions between the liquid phase and the components of the mixture to produce the differential distribution of the components. This process causes separation to occur as the liquid phase passes over the solid phase.

3-Separation by Extraction

This method is used for the separation of an organic compound (solid or liquid) from its aqueous solution by shaking with a suitable solvent (e.g. ether, benzene, chloroform, carbon tetrachloride etc.) in a separating funnel. The selected solvent should be immiscible with water but should dissolve the organic compound to an appreciable extent.



It is important to note that extraction is more efficient (i.e., more complete) when a given volume of the extracting solvent is used in several installments.



In this technique, two liquids that do not dissolve very well in each other (immiscible liquids) can be separated by taking advantage of their unequal density. A mixture of oil and water, for example, can be separated by this technique

3-Separation by Centrifuge:

A centrifuge technique is used to isolation and separation small amounts of a heterogeneous mixture (for biocomponents). Test tubes containing the mixture are spun around very fast so that the solid gets flung to the bottom. The mixtures are spun horizontally in balanced containers, and the rotation sets up centripetal forces causing the mixture's components to separate according to their densities. Separating blood a centrifuge is used to separate blood plasma from blood cells. As the test tubes spin, the heavier blood cells sink to the bottom. Centrifuges are useful in laboratories and on an industrial scale for separating solids from suspension in liquids. For example, in the food industry, a centrifuge is used to separate cream from milk or yeast from fermented malt. Usually when the heaviest particles have settled to the bottom, the top liquid is decanted or poured off to separate the liquid layer from the bottom layer.

4- Separation by Filtration

This is a more common method of separating an insoluble solid from a liquid. An example of such a mixture is sand and water. Filtration is used in water treatment plants, where water from rivers is filtered to remove solid particles. Filtration is any of various mechanical, physical or biological operations that separate solids from fluids (liquids or gases) by adding a medium through which only the fluid can pass. The fluid that passes through is called the filtrate. In physical filters oversized solids in the fluid are retained and in biological filters particulates are trapped and ingested and metabolites are retained and removed. However, the separation is not complete; solids will be contaminated with some fluid and filtrate will contain fine particles (depending on the pore size, filter thickness and biological activity). Filtration occurs both in nature and in engineered systems; there are biological, geological, and industrial forms. For example, in animals (including humans), renal filtration removes wastes from the blood, and in water treatment and sewage treatment, undesirable constituents are removed by absorption into a biological film grown on or in the filter medium, as in slow sand filtration. Depending on the application, either one or both of the components may be isolated, and it is very important in chemistry for the separation of materials of different chemical composition. A solvent is chosen which dissolves one component, while not dissolving the other. By dissolving the mixture in the chosen solvent, one component will go into the solution and pass through the filter, while the other will be retained. This is one of the most important techniques used by chemists to purify compounds; it is important and widely used as one of the unit operations of chemical engineering. It may be simultaneously combined with other unit operations to process the feed stream, as in the bio filter, which is a combined filter and biological digestion device.



There are many different methods of filtration; all aim to attain the separation of substances. Separation is achieved by some form of interaction between the substance or objects to be removed and the filter. The substance that is to pass through the filter must be a fluid, i.e. a liquid or gas. Techniques of filtration vary depending on the location of the targeted material, i.e. whether it is dissolved in the fluid phase or suspended as a solid.

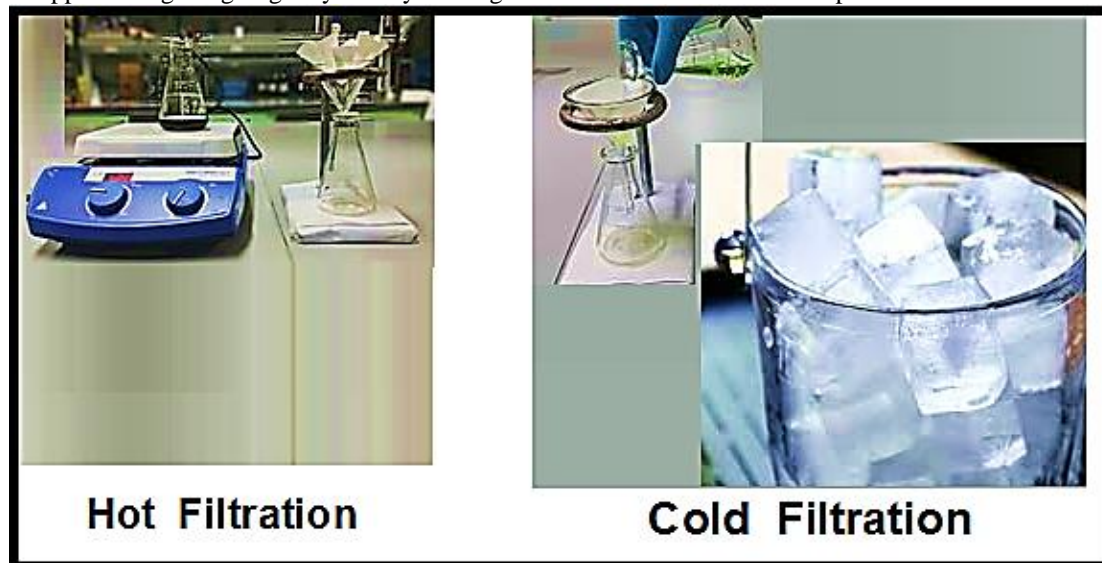
Hot Filtration, solution contained in the Erlenmeyer flask is heated on a hot plate in order to prevent re-crystallization of solids in the flask itself. There are many filtration methods depend on the desired outcome namely, hot, cold and vacuum filtration. Some of the major purposes of getting the desired outcome are, for the removal of impurities from a mixture or, for the isolation of solids from a mixture.

A- Separation by Hot filtration:

This separation method is used to separate and isolate solids components from a hot solution to prevent crystal formation in the filtration funnel and other apparatuses which comes in contact with the solution. As a result, the apparatus and the solution used are heated in order to prevent the rapid decrease in temperature which in turn, would lead to the crystallization of the solids in the funnel and hinder the filtration process. One of the most important measures to prevent the formation of crystals in the funnel and to undergo effective hot filtration is the use stemless filtration funnel. Because the absence of stem in the filter funnel, there is a decrease in the surface area of contact between the solution and the stem of the filter funnel, hence preventing re-crystallization of solid in the funnel, adversely affecting filtration process.

B- Separation by Cold Filtration :

Method is the use of ice bath in order to rapidly cool down the solution to be crystallized rather than leaving it out to cool it down slowly in the room temperature. This technique results give the formation of crystals(very small) as opposed to getting large crystals by cooling the solution down at room temperature.

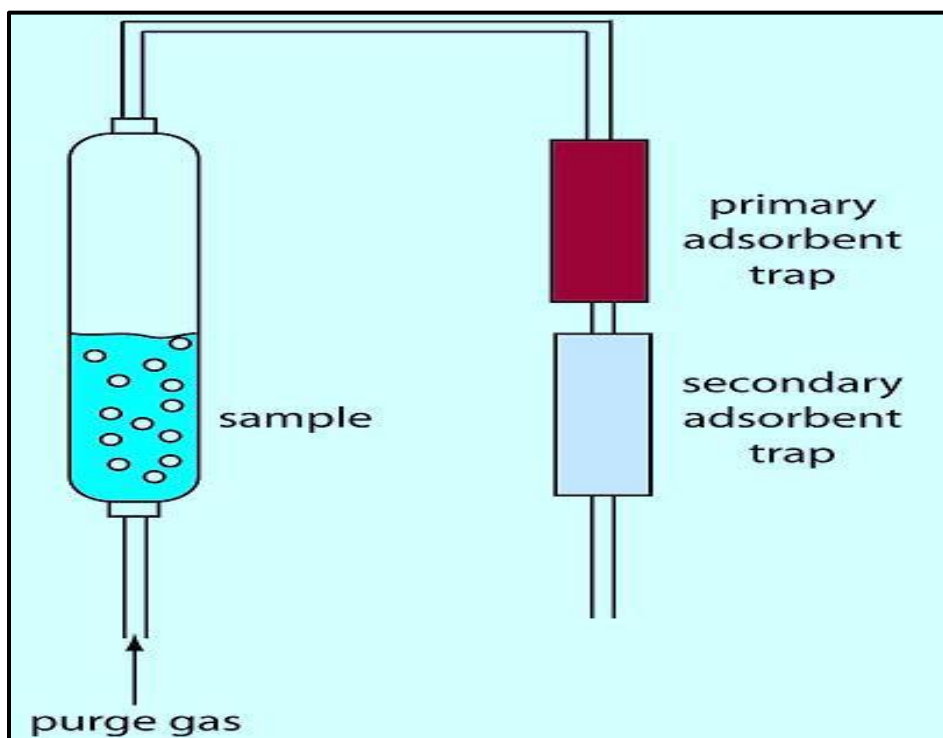


Hot Filtration

Cold Filtration

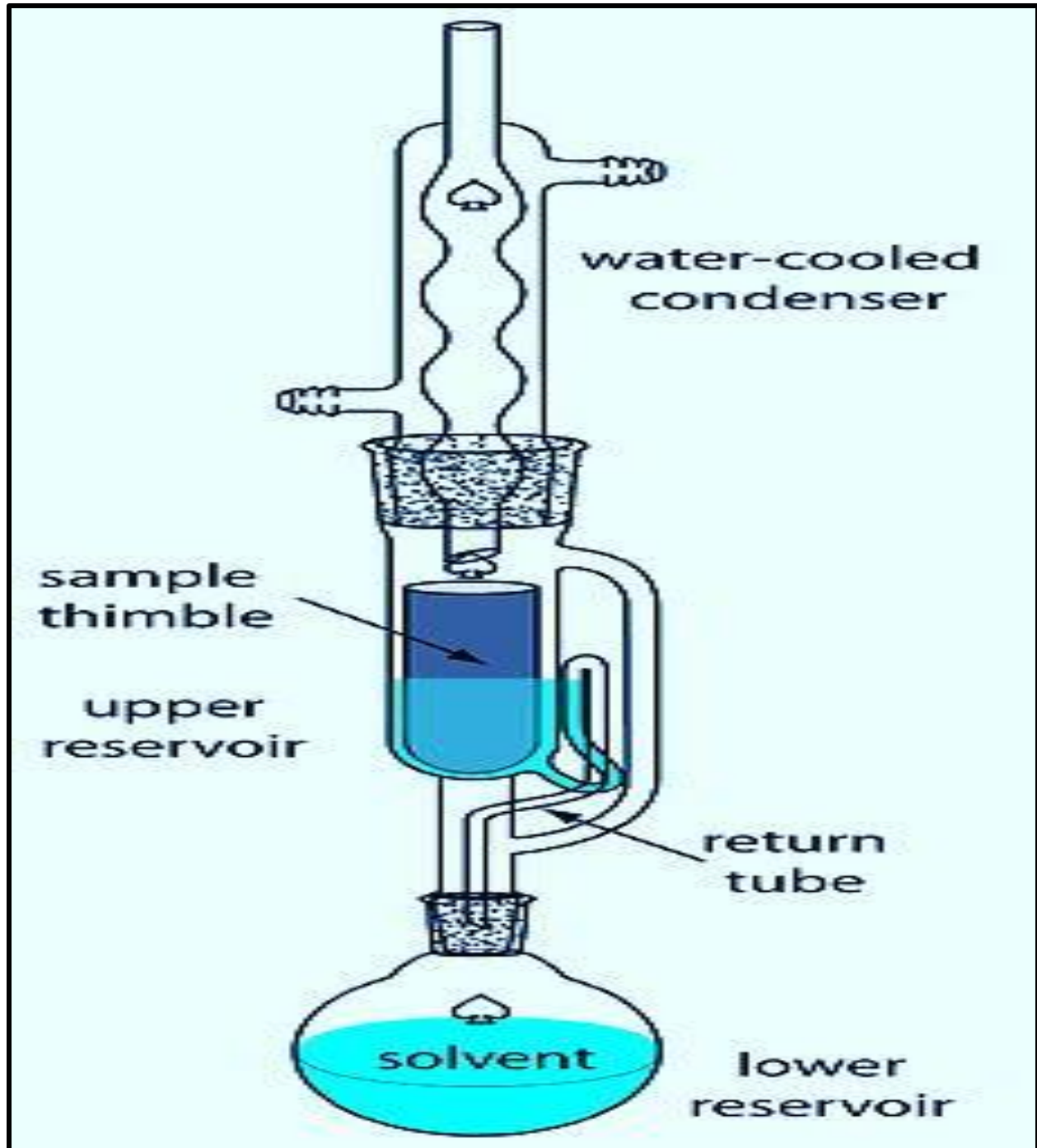
4- Microwave assisted extraction (MAE)

Microwave energy to facilitate partition of component from the sample matrix into the solvent. Its radiation interacts with dipoles of polar and polarizable materials give heating near the surface of the materials and heat is transferred through conduction. Dipole rotation of the samples induced by microwave electromagnetic disrupts hydrogen bonding; enhancing the migration of dissolved ions and promotes solvent penetration into the matrix. In non-polar solvents, poor heating occurs as the energy is transferred by dielectric absorption only. Microwave-assisted extractions have replaced Soxhlet extractions in some applications. The process is the same as that described earlier for a microwave digestion. After placing the sample and the solvent in a sealed digestion vessel, a microwave oven is used to heat the mixture. Using a sealed digestion vessel allows the extraction to take place at a higher temperature and pressure, reducing the amount of time needed for a quantitative extraction. In a Soxhlet extraction the temperature is limited through the boiling point of solvent at atmospheric pressure. When acetone is the solvent, for example, a Soxhlet extraction is limited to 56°C, but a microwave extraction can reach 150°C. Two other continuous extractions deserve mention. Volatile organic compounds (VOCs) can be quantitatively removed from liquid samples by a liquid-gas extraction. The purge gas removes the VOCs, which are swept to a primary trap where they collect on a solid adsorbent. A second trap provides a means for checking to see if the primary trap's capacity is exceeded. When the extraction is complete, the VOCs are removed from the primary trap by rapidly heating the tube while flushing with He. This technique is known as a purge-and-trap. Because the analyte's recovery may not be reproducible, an internal standard is necessary for quantitative work.



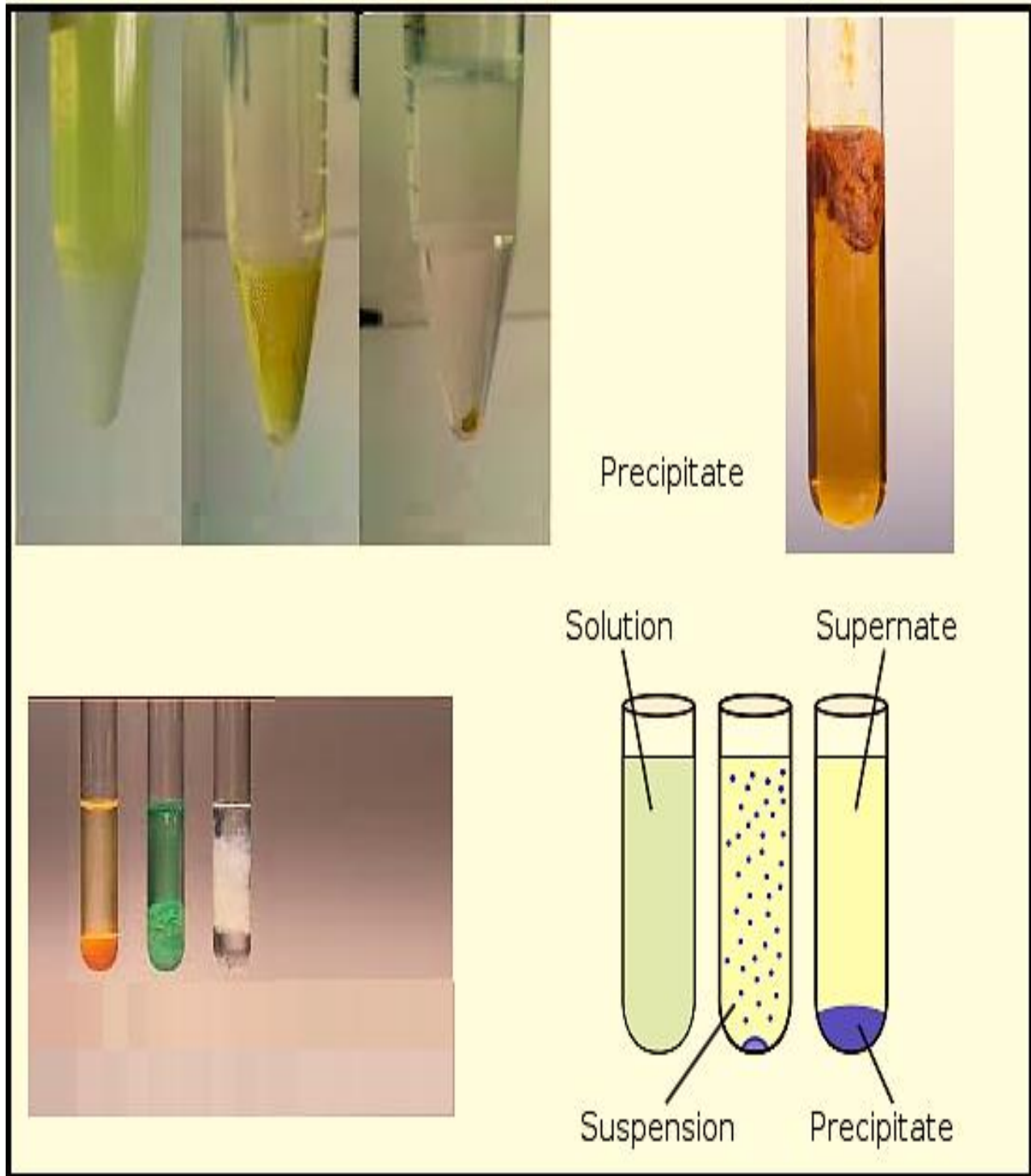
5- Continuous Extractions

The extraction by this method of a solid sample is carried out through a **Soxhlet extractor**. The extracting solvent is placed in the lower reservoir and heated to its boiling point. Solvent in the vapor phase moves upwards through the tube on the far right side of the apparatus, reaching the condenser where it condenses back to the liquid state. The solvent then passes through the sample, which is held in a porous cellulose filter thimble, collecting in the upper reservoir. When the solvent in the upper reservoir reaches the return tube's upper bend, the solvent and extracted component are siphoned back to the lower reservoir. Over time the component concentration in the lower reservoir increases.



7-Separation by Precipitation

Precipitation is separation method occurs in two steps: first the tracer is separated through coprecipitation with a carrier, after that it is isolated or separated from the carrier (Hermann and Suttle, 1961). Use of carriers that can be easily separated from the tracer is helpful, for this reason, coprecipitation through inclusion is not generally used. Coprecipitation through surface adsorption on unspecific carriers which is the most common using .

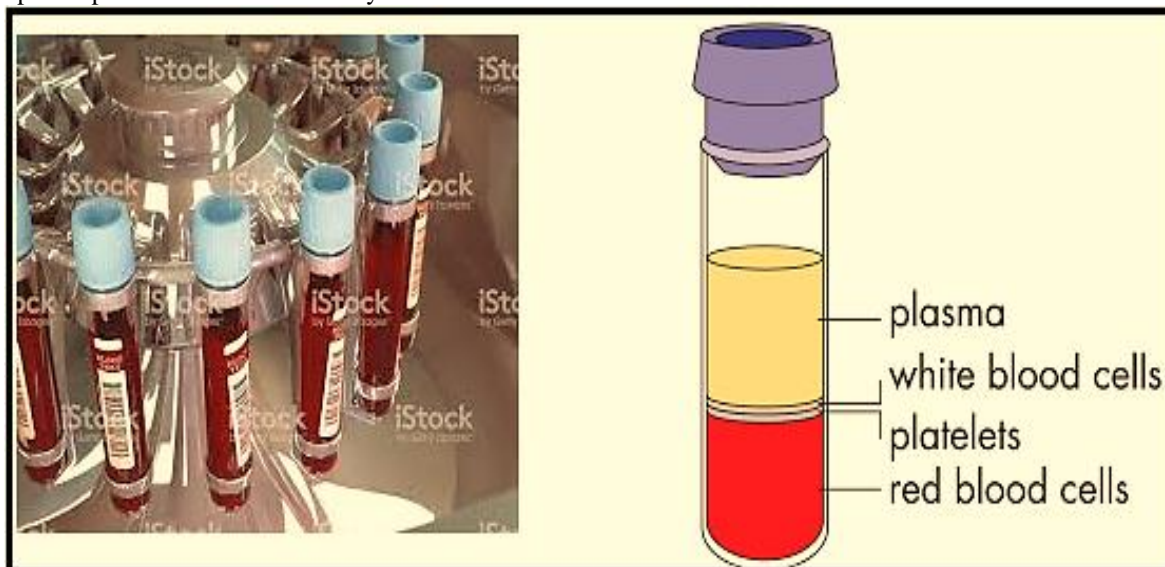


8-Separation by Centrifuge(Bio samples)

Separation by centrifuge use to separation of bio samples like blood and contents of blood , plasma , ... through rotation of samples in centrifuge. The components of a suspension can separate by centrifugation. The dispersed particles and fractions of a suspension should be put in a special tube and placed into the rotor of the centrifuge. Then the tubes is subjected to centrifugal force, generated by the rotation of the centrifuge.



Separation of Blood Components , Blood contain of two types of cells: Red Blood Cells ((RBC- erythrocytes) and White Blood Cells (WBC- leucocytes)). Erythrocytes are the blood cells which distribute oxygen from the lungs to all the organism, but the leucocytes are responsible for attacking any infectious cells, sustaining the optimal performance of immune system.



Principles of Separation

A mixture of organic compounds may be in the solid or liquid form or may include a solid contents or suspended in a liquid. If a solid contents or a liquid contents are present it is usually unwise to expect separation to be accomplished by filtration because the liquid phase almost certainly contains some dissolved solid and traces of the liquid component may be difficult to remove from the solid compound. The methods of isolating pure samples of the components from a mixture may be either physical or chemical. The chemical method consists of fractional distillation and is applicable only if there is a wide difference between the boiling points of the two compounds and provided that an azeo-trope is not formed. The chemical method of separating two chemical compounds depends on their differing solubility in water, ether, dilute base or acid.

Mixed melting point

The component, whose purity is to be tested, is mixed with a pure sample of the same compound. The melting point of the mixture is determined. If the melting point of the mixture is sharp and comes out to be the same as that of pure compound, it is sure that the compound under test is pure. On the other hand, if the melting point of the mixture is less than the melting point of the pure compound, the compound in question is not pure. A large number of methods are available for the separation of substances. The choice of method depends on the nature of components in samples ((solid or liquid)) and the type of impurities present in it.

Purification of Compounds after Separation

The separation and isolation of compounds in mixtures to give the pure components is of great practical importance in chemistry. Many synthetic reactions give mixtures of products and it is necessary for you to have a reasonably clear idea of how mixtures of compounds can be separated. Almost all compounds of biochemical interest occur naturally as components of very complex mixtures from which they can be separated only with considerable difficulty.

The classical criteria for determining the purity of organic compounds are correct elemental compositions and sharpness of melting point or constancy of boiling point. Important though these analytical and physical criteria are, they can be misleading or even useless. For instance, the analytical criterion is of no help with possible mixtures of isomers because these mixtures have the same elemental composition

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