Chromatography

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Chromatography is a general term that is applied for a wide variety of separation techniques based on the partitioning or distribution of a sample (solute) between a moving or mobile phase and a fixed or stationary phase.

Stationary Phase

It is characterized by a high surface area and get the separation of the result physiological interaction for the Material to be estimated Material to be estimated and portable by the mobile phase and this phase either solid or liquid.

Mobile Phase

Is the phase that carries the material to be separated and passes from the top of the stationary phase and this phase is either liquid or gas.
There are a large number of chromatography methods and for accurate study, chromatography methods should be classified.

Chromatography classification

The chromatographic procedures can be subdivided according to the various techniques applied, or to the physicochemical principles play role in the separation.

A **The chromatographic procedures according to the various mobile phases can be divided into three parts:**

1-Gas chromatography (GC).
2- supercritical fluid chromatography (SFC).
3- liquid chromatography (LC).

B **The liquid chromatography can be divided into:**

1- Paper chromatography (PC).
2- Column chromatography.
3- Thin layer chromatography (TLC).

Ion - Exchange Chromatography

The term "ion exchange" means the exchange between ions have the same charged between a solution and a solid material that contact with the solution but does not dissolve in it. It is called an (Ion - Exchanger).

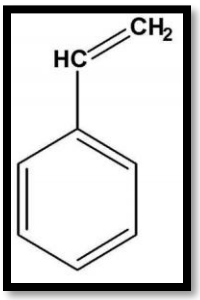
The ion-exchanger is characterized by its own charge, and have a porous molecular structure that allows the movement of ions and solvent molecules through it in and out freely. There are many materials that are suitable for this purpose, such as natural materials, including some types of clay and soil where the phenomenon of ion exchange was discovered for the first time in clay and soil, where it was noted that the dissolved salts ions in the water are exchange with the ions associated with soil, The clay usually contains in its crystalline structure an increase of the positive or negative charge, This charge is equivalent with the ions that have the opposite charge that are can change able with ions have the same charge soluble in the solution and in contact with the clay particles

$$2R^{-}\,Na^{+} + Ca^{+2} \rightleftharpoons (R^{-})_2\,Ca^{+2} + 2Na^{+}$$

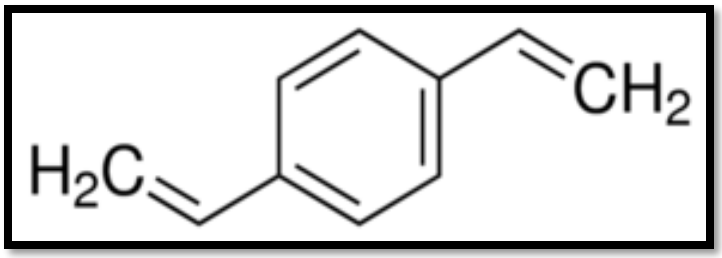
On the exchanger **in solution** **on the exchanger** **in solution**

R^- : Is the body of the exchanger that carries a negative charge in the case of the cation exchanger which is always equated with a positive ion charge such as **Na^+** an ion exchange component $R^- Na^+$.

The stationary phase in ion-exchanger chromatography is Polymer particles (polystyrene) which is linked by side with (Di vinyl benzene), that called (Resin). The vinyl group in the ring can be easily exchanged when added an active acidic groups.



Styrene



Di Vinyl benzene

There are four main types of ion exchange resins used in analytical chemistry:

Exchanger Type	Effective functional groups
A-*Cation exchanger*	
1- Strong acid	Sulfonic acid SO_3H^+
1- Weak acid	Carboxylic acid $COOH^+$
B-*Anion exchanger*	
1- Strong base	Quaternary ammonium groups $R\text{-}CH_2N^+(CH_3)$
2-Weak base	Poly amine

Preparation of ion exchanger

The column is prepared by filling the solid material (resin) inside the column by adding it to a column filled with solvent and left until it precipitates. The column can be mechanically shaken or the solvent is crushed by a long piston during filling. The air bubbles formed in the column should be expelled and made less efficient.

A glass wool or perforated glass disc is placed in the bottom of the column to support the stationary phase and we can be use the burette as a column.

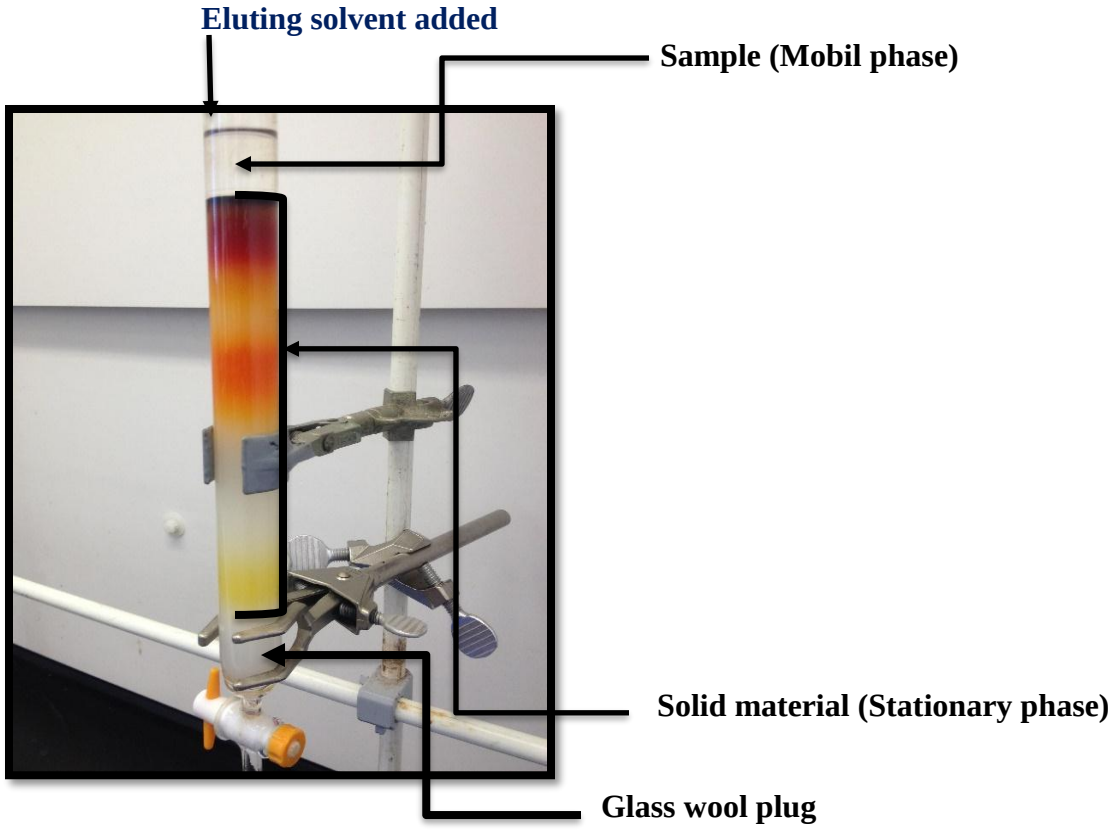
The dimensions of column depend

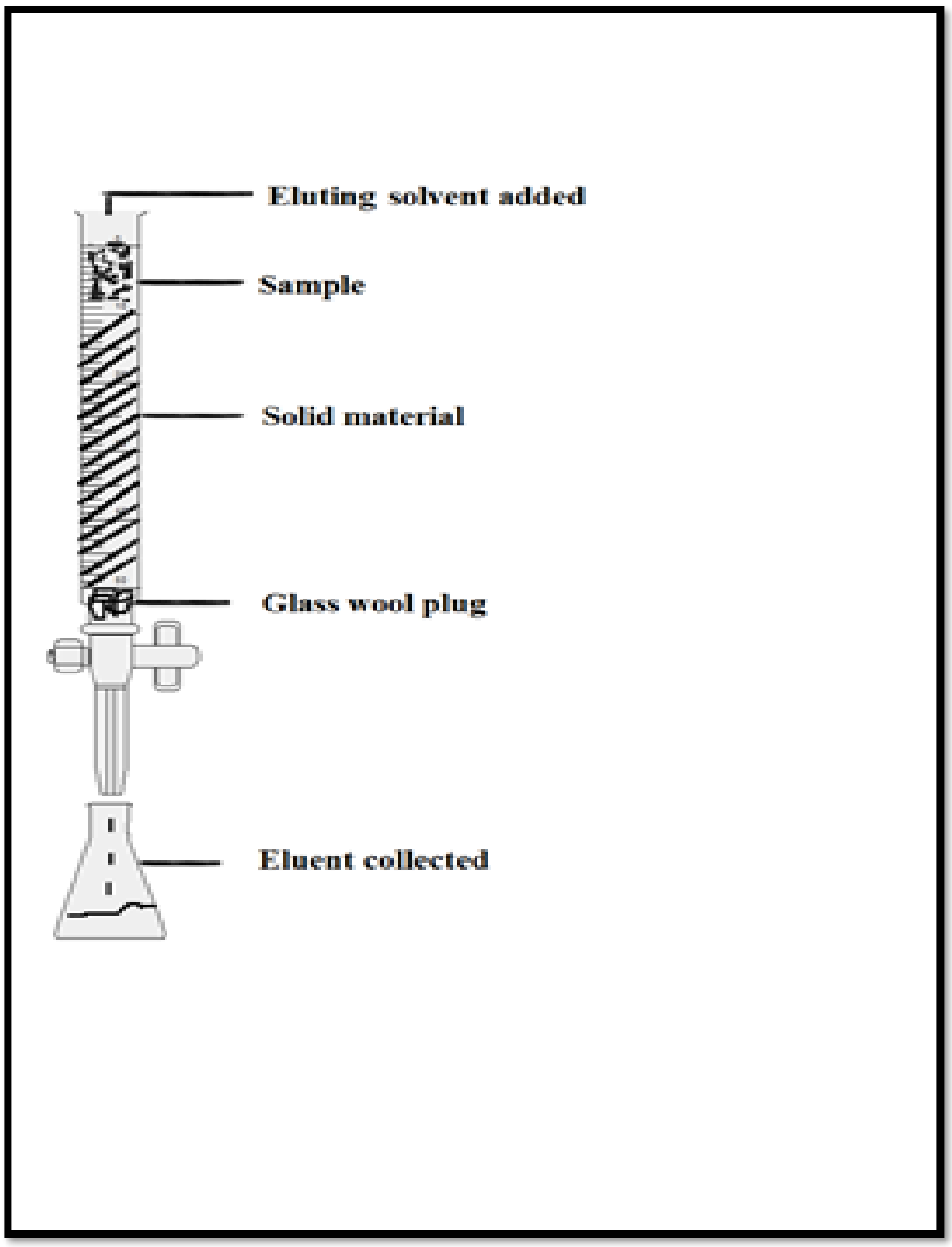
1- Separation efficiency required.

2- The size of the sample

3- The type of the chromatography separation method.

The dimensions of the ion exchanger column are range from a few millimeters in diameter and a few centimeters in length to a few centimeters in diameter and several tens of centimeters in length.




Eluting solvent added
Sample
Solid material
Glass wool plug
Eluent collected

Cation exchange resins (positive ion exchanger)

These resins contain acid groups linked to the aromatic ring, the positive ions exchangers (strong acid) are sulfonic acid groups (SO_3H^+) and it's a very strong acid similar to sulfuric acid while The positive ion exchangers (weak acid) are carboxylic groups ($COOH^+$), They partially ionize so that protons of these groups can be exchanged with other cations.

$$n\ Rr^-\text{-}SO_3H^+ + M^+ \rightarrow (\ Rr\text{-}\_\ SO_3\text{-}\ )n\ M^+n + nH^+ \quad \text{(strong acid)}$$

$$n\ Rr^-\text{-}\ COOH^+ + M^+ \rightarrow (\ Rr\text{-}\ \_COO\text{-})n\ M{+}n + nH{+} \quad \text{(weak acid)}$$

Note

Rr	**the resin**
n	**the number of moles**
M^+	**the positive ions**

Anion Exchange Resins (Negative ion exchanger)

These resins contain basic groups (hydroxyl ions OH^-) can be exchanged with the other anions. The (Quaternary ammonium groups) are very strong base while the Amine groups are a weak base groups.

$$n\ Rr^+\text{-}NR_3\ OH^- + A^{-n} \rightarrow (Rr^+\text{-}NR_3)n\ A^{-n} + n\ OH\text{-} \quad \text{(Strong base)}$$

$$n\ Rr^+\text{-}NH_3\ OH^- + A^{-n} \rightarrow (Rr^+\text{-}\ NH_3)n\ A^{-n} + n\ OH\text{-} \quad \text{(Weak base)}$$

Note

Rr	**the resin**
n	**the number of moles**
A^{-n}	**the negative ions**

Properties of Exchangers

Ion exchangers must have certain properties in order to be use several times. **Some of these properties** that must be known and controlled are:

1- The size of the exchanger granules must be large.

2- Degree of branching.

3- Degree of inflation.

4- Capacity.

Capacity

It is the number of the (M. eq) of (H^+ or OH^-) per gram of solid resin.

Or It is the number of the (M. eq) of (H^+ or OH^-) In the one ml volume of the wet resin.

$$\text{Capacity} = \frac{\text{M.eq.}(OH^- \text{ or } H^+)}{\text{g (Solid R.)}} \qquad \text{Capacity} = \frac{\text{M.eq.}(OH^- \text{ or } H^+)}{\text{ml(Liquid R.)}}$$

The ion- exchanger activated

To converting the resin (exchanger) to the desired shape depends on the type of exchanger (cationic or anionic) and it depends on the purpose of the experiment. In the process of water desalination (deionized water, removing the ions from the water), the cationic exchanger is converted into (H^+- form) by adding a suitable dilute acid to the column containing the cation resin. The anionic exchanger (OH^-- Form) by adding a suitble dilute base to the column containing the anionic resin and then wash the column with the distilled water to remove the trace of acid or base. The washing process is done by adding distilled water to the column with the opening of the column faucet and maintaining the flowing speed. The cation exchanger activates by added an acid like HCl (3M), then added of distilled water as a few batches, for example (10 ml) and then we get rid of the solution in the column to the conical flask and then check it by using silver nitrate ($AgNO_3$).

If the solution is turbid, that is mean the solution is contain salts that is formed by the reaction of the added acid with the ions in the resin, then we remove of the solution and washing the resin by distilled water. The solution must be remove from column again and check the solution by silver nitrate, if solution is not turbidity this refer of the absence of salts and complete the check by adding a drop of the methyl orange indicator. when the color of the solution is pink that is mean the presence of the acid in the column while if the solution is yellow color that is mean no more acid in the column.

The separation by the ion exchanger

Is one of the types of chromatography (liquid - solid) which are separated all similar ions in the charge.

Types of ion exchangers

1- Natural exchangers such as zeolite, soil.

2- Structural exchangers: can be **divided into:**

A Non-organic ion exchangers

These exchangers are divided into **two parts:**

1- Aluminum silicate

It is a cation exchange consist from the mixing of aluminum sulphate and sodium silicate to produce a gel containing aluminum ions that can be replaced with calcium ions or ammonium or other ions. Although these exchanges have a high exchange capacity but it's easily decomposed by acids and base. The most important use of these exchanges in the estimation of ammonium ion.

2- Hydroxyl Oxides

The precipitate (iron oxide or aluminum oxide) which have a positive charge (as a cationic exchanger) and their ability to ions exchange are small, While the hydroxyl oxides of quaternary metals (Sn, TN) are more useful in the ion exchange.

B Organic exchangers

are industrial resin have a high molecular weight containing an effective group of organic polymers that are insoluble in water and can combine with a large number of electrically charged groups and are used in analytical chemistry and are available in the granules form ranging from (0.02 - 0.04 mm)