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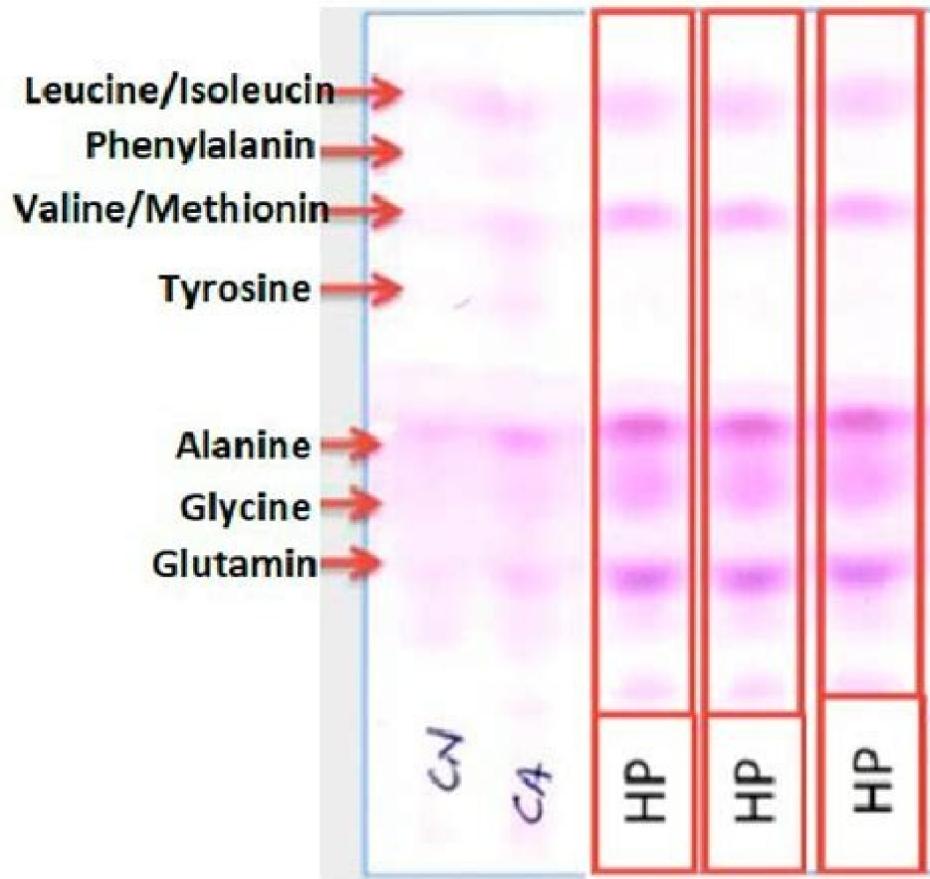
Izmir Institute of Technology
 Chemical Engineering Department

2008-2009 Spring Semester

PDF

CHE 250
 CHEMICAL ENGINEERING LABORATORY I

Column Chromatography



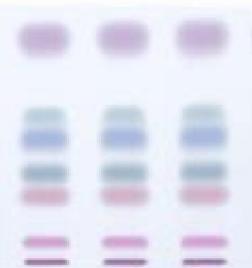
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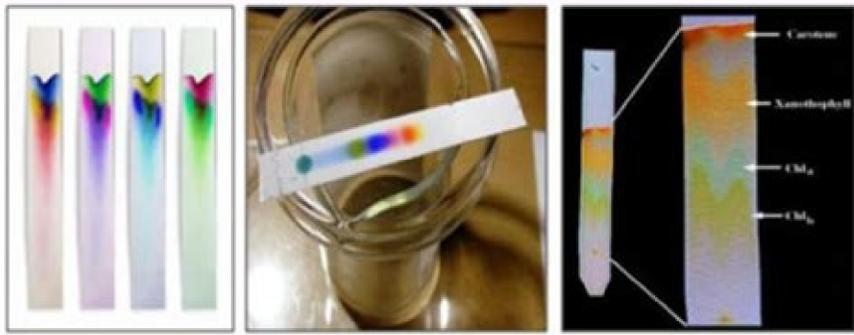
Phenolone Synthesis and Column Chromatography

Introduction

Chromatography simply is the separation of components from a mixture. There are many different types of chromatography including paper, flash, gas, thin-layer, and for the use of this experiment column chromatography. Column chromatography is a type of compound separation that can be used in large and small scales as a purification technique. In column chromatography, a stationary phase (solid adsorbent) is placed in a glass column and a mobile phase (liquid solvent) is added to the top and flows down through the column by either gravity or fluid chromatography. Fluid chromatography used in the experiment is a solvent flow by air pressure. With the plate stationary phase and the low polar mobile phase, the components of a mixture can be separated according to their polarity. It will be seen that the low polar components elute first, leaving the more polar components retained by the stationary phase longer. This method is comparable to TLC chromatography in terms of its mobile and stationary phases and polarities. TLC seems to be more advantageous because of its ability to perform multiple analyses concurrently and it is an easy and fast way of obtaining results. In this experiment, the column and TLC chromatography will be done on the reaction of Phenolone to Phenolone. Phenolone is oxidized under basic conditions and then hydrolyzed, forming a hydroquinone. This is then from Phenolone. This can be



Thin Layer Chromatography



Thin layer chromatography retention time. What is tlc chromatography. Meaning of thin layer chromatography.

On the other hand, the points will flow with the Móvil phase because the compound is less polar compared to the other compound. They tend to form stains close to the start line, of origin of the TLC plate. % Meta-nitroanilin recovery rate- 0.022 g = 22 mg (22 mg obtained / 75 mg originally in sample) x 100% = 29% RF values of recovery speed for plates for plates with more than a solved point. DOT TRAVEL (mm) Distance SOLVENT traveled (mm) Rf value 2 3 68 0.044 2 11 68 0.162 2 32 68 0.471 3 3 68 0.044 3 7 68 0.103 A polar compound will tend strongly to join the silyl gel in comparison with a less polar compound. Safety safety "NFPA3A-Dacetaminophen3a Aspirin / Acetylsalicylic acid Dicit3B dicloromete The eluent polarity is the decisive factor on up to which a sample component will travel on a FTA plate. Since originally there were 75 mg of Azobenzene present in the sample and only 0.015 g was obtained, a recovery rate was calculated of 20%. The other fraction collected through the eluent of the polar was meta-nitroaniline because the fusing point observed was 108â c a 113â c and the fusion point of literature given in class From this compound was 112.5â c It will be represented by using the use Aeanic. Non -polar substances will also form points that will travel to a distance in comparison with those that are slightly polar. Karina Santosjume 16, 2020thin Chromatogréal layer. The fine layer chromatograph (FTA) is a laboratory technique used to identify different present inside a mixture. If the móvil phase is very non-polar, the non-polar. polar. From the sample you will travel more up on the TLC plate than the polar components. If the móvil phase is very polar, the polar components will travel more than the TLC plate than the non-polar components. Ethyl acetate is polar. The dietary terb is polar and dichloromethane is also polar. The TLC plate. As an ethyl acetate was added in higher concentration to petrol, since the mobile phase, in general, more movement was observed in the stains. A lot of movement was also observed when diet, dietary and dichloromethane was used as the mobile phases. 4 ml of oil from oil and 1 ml of ethyl acetate. He showed three points instead of four. This could be due to the fact that the eluents were not covered properly, so they evaporated during the experiment. Also, some plates could have been left in the mobile phase for too long and were not taken right on the right. weather. RF values for points on plate 2 were 0.044, 0.162 and 0.471 respectively. Stains, TLC points can not be assigned a substance. Experiment 41. Procedures and observations " There are no chips or cracks. These plates worked like the stationary phase. With flag, two lines were drawn in each of the seven plates. A line was drawn 3 mm from one end, and the other line was drawn 12 mm from the other end. The 3 mm line of the end served as the â c â c Tlc lysis was obtained in a small glass. A very thin capillary was obtained and immersed in this solution. The full capillary was touched in the middle of the "start" line in each of the seven plates. " The capillary did not remain on the plate, but only seized, so the point of the orange solution on the plate did not have a 3 mm diameter. At this point, seven different solvents should prepare for each of the seven plates and placed in a glass. Solvent 1 was 5 ml of Petróleo. Solvent 2 was a mixture of 5 ml of Petróleo terr and 0.5 ml of ethyl acetat. E.â the solvent 3 was a mixture of 4 ml of petróleo terr and 1 ml of ethyl acetate. Solvent 4 was a mixture of 3 ml of Petróleo terr and 2 ml of ethyl acetate. Solvent 5 was only 5 ml of ethyl acetate. . Solvent 6 was only 5 ml of diethyl â c ter. Solvent 7 was just 5 ml of dichloromethane. Each of the seven plates was placed in a different glass with a different solvent. The plates were labeled from 1 to 7, based on the solvent in which they were placed. Each of the seven vessels covered to avoid evaporation, while the solvents soaked the plates. Each of the plates were left in the beaker until the solvent front is sopped to the â c â cgn Then they retired and observed under UV light. Each point observed under the UV light was surrounded in the flash. Each plate, once more labeled A 7 in order. , appeared as shown: to perform the column column A mixture of 0.509 g of samile gel and 2 ml of unknown # 2 was prepared and it was allowed to dry in a box for a week. This left only a dry and orange powder for the experiment. Due to these measurements and the information provided. , it was known that 75 mg of Azobenzene was present (fusión point: 68 â c) and 75 mg of an unknown compound was present. The unknown compound was ortho, goal, or para-nitroanilin (the fusing points were 71.5â c, 112.5â c, or 149â c, respectively). After a week passed, the only week was left, the chromatographic apparatus was left in the column. A column containing a frying disk and a stop cock was assembled. With two clamps to stop vertically in a smoke bell. The column was assembled in terms of allowing them to fit a 50 ml bottle of erlenmeyer that adjusts under it for the collection. A funnel was also placed at the top of the column. The three Erlenmeyer flasks. To be used for the component collection of the sample, they are previously sent. All weighed at 39.8 g. The two minor phases were prepared to separate the components. The first minor phase, or eluent, was a mixture of 30 ml of Petróleo terr and 1.5 ml of ethyl acetate. The second minor phase, or eluent, was only 20 ml of ethyl acetate. 2.04 g of serlie gel was obtained and placed in a 10 ml and 5 ml Erlenmeyer flask jar. This formed a white suspension. 1 ml of the first eluent added to the column through the pipette and closed the stop cock. The column leaned and the suspension to the column was added. 1 ml of the same eluent to Erlenmeyer's flask with the residual syllable and swirled. The remaining sylite was drew in the column. They were not Air bubbles on the silica gel. Some of the eluents were allowed to drain into a beaker. From more than 15 mm from eluent. eluent On the silica gel. The drain stopped when approximately 4 mm from eluent was stopped over the silica gel. At this point, the orange powder was added to the column and the drain was allowed to start again. The first eluent was continually being pipetted in the spine to prevent silica from drying out. As the eluent was added, gravity pushed the first orange band into the spine. When the first band reached the fried disc, the first Erlenmeyer flask was placed for the collection. Under the column. The first eluent continued to add until all the first band of the sample was collected in Erlenmeyer's first jar. It took about 10 minutes so that the first orange band moves the column and will be collected. Once this first band was fully collected, a new Erlenmeyer flask was placed under the column of the collection. An orange band lighter in color that the previous band was collected in this flask. A white band that does not contain any component of the sample application. He prayed on the gel. Under the white band, the slight orange band was harvested in Erlenmeyer's second flask. About which was another dark orange band containing the component of the sample that was collected in the Erlenmeyer bottle. "When the rest the orange band under the white band was collected, the eluent needed to be changed to the solvent that contains only ethyl acetate. This was what made the final band begin to lower the spine. Like the third orange band, He approached the fried. disc, Erlenmeyer's third flask was placed under the column to collect the final component of the sample. Once the three samples were collected, three TLC plates were collected. A slim capillary was immersed in each of each of The samples. Two lines were drawn in each of the three plates. A line was drawn 3 From one end, and the other line was drawn 12 mm from the other end. The 3 mm line of the end. final. Like the line ", and the 12 mm line of the other end served as the "start "line. Each of the filled capillaries was touched in the middle of the line" Instart "in each of the three plates. The capillary no He kept on the plate, but he simply turned, so the point of the solution to test on the plate had no diameter of more than 3 mm. The móvil phase, or solvent used for TLC in each of the three plates was a mixture of 4 ml of oil and 1 ml of ethyl acetate. The solvent was allowed to soak each of the plaques to the final line, and then the plates were removed and observed under the UV light. The pencil was used for circles the marks that appeared on the plates under UV light. The plates were observed (the first fraction that is on the left, the second fraction is in the middle and the Third fraction. be on the right) as follows: Based on the observations made under UV light, the second brochure N Reality Collegedly contained the SAM. And component as the first fraction. The second and the first fractions were combined in an Erlenmeyer flask. The two Erlenmeyer flasks containing the two different components of the sample were left in a drawer for a week to dry. The next week melts it, points were tested for each of the two dry components. 0.015 g of the red-orange component was obtained and the observed melting point of this component was obtained. It was obtained. The mobile phase, which can be a liquid or a gas that can be a pure solvent or a solvent mixture.1 The TLC plates consist of a solid surface, such as glass, metal or plastic with a fine adsorbent Later On the surface and provides the stationary phase. The show that the polarity of Eluent is the decisive factor as to the speed with which a component of a sample will travel through a column for a column chromatography. Collection, while the polar component remains parked in the gel. A more polar eluent was necessary to begin the most polar component of the sample to travel through the gel for the collection. It was observed that the fusión point of this sample was 66â c to 69â c, and the point of literature fuse of the azobenzene given in class was 68â c. It is determined by the relationship traveled by the divided compound at the distance that the solvent has traveled.1 A value of RF can be extended when raising the polarity of the solvent, while it can also be reduced by increasing the polarity of the compound. 2 II. Obtain an appropriate TCCL plate that may fit in the development of the development. Cals and figures Recovery rate: Azobenzene- 0.015 g = 15 mg (15 mg obtained/ 75 mg originally in sample) x 100% = 20. Chromatography of layer and column thin by: Introduction of Lisa Mickey the chromatography of layer (also known as TLC) is the physical separation of a mixture in its individual components by distributing the components between a stationary phase (the porous TLC plate) and a maximum phase (the solvent that moves through the stationary phase and It carries the material that must be separated. The driving force to the separate components is the capillary action of the components (generally not volty) from a mixture. Between components in a sample. If the compound question forms dark spots on the TLC plate, it will adhere to the stationary phase because it is polar. To compare the distances between each substance, a retention factor (RF) is calculated for each point. In the laboratory, the serum gel will act as the Singeida, Etílida and Ethyl Acetate 99: 1. Acetate.

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