Column Chromatography. Separation of Fluorene and 9-Fluorenone.

Reading

Please read Unit 9. For packing the micro column, see "Packing and Running the column" on pp 210-211. Use Google or Wikipedia to find the structures of fluorene and 9-fluorenone. Which compound do you think will travel down the column first?

Procedure

The column chromatography experiment has been scaled down and uses a pipette column as described in the text. Follow the instructions on page 210 to pack the column using a pipette, a little cotton, a little sand and silica gel (see Figure 9.5 on Page 202). Perform a TLC analysis of the mixture you are about to separate with a solvent mixture of 10% acetone in hexanes.

Loading the sample:

Using a glass Pasteur pipette and bulb add 0.5 mL of the fluorene/fluoreneone mixuture (prepared from 5 g of each in 200 mL of CH_2Cl_2) solution to the top of the column by slowly and carefully introducing the solution down the inside wall of the pipette column. Add ~1 mL of hexanes, then drain down to the level of the sand. The sample is now loaded on the column.

Elution:

Carefully fill the column with hexanes (~1 mL), so as to not disturb the top of the packed column, and then *elute* the column. Collect the *eluent* in a 25-mL Erlenmeyer flask (note that the *eluent* is what is added to the column (aka the mobile phase) – the *eluent* is what exits the column). Continue *eluting* the column with 2-mL portions of hexanes until the first colored band is about to come off of the column. Then begin eluting the column with 2 mL of 30% acetone/70% hexanes. Just before the colored band comes off of the column, switch to a 25-mL Erlenmeyer flask and collect the colored band. Perform TLC analysis on both fractions and the original mixture. Perform the TLC analysis on the fractions from your column chromatography and compare to the mixture that you separated using methylene chloride (CH_2Cl_2) in the TLC development chamber. Remove the solvent with rotary evaporator in a round bottom flask and collect the two products.

To Complete the Experiment

Take an IR spectrum of the fluorene and 9-fluorenone obtained from the column chromatography. Describe the success (or lack thereof) of the separation of fluorene and 9-fluorenone.