**FLASH COLUMN CHROMATOGRAPHY:**

**SEPARATION OF FERROCENE, ACETYLFERROCENE, AND DIACETYLFERROCENE**

**Required Prelab Reading:** Padias, pp 174-180

**Previous techniques that you must know and be able to perform:** TLC and Rotary Evaporation



This experiment is designed to teach you the technique of flash column chromatography. What distinguishes flash column chromatography from normal column chromatography is that the solvent is pushed through the column using pressure. This usually results in a faster and better separation.

You will be given an unknown that contains at least 2 of the 3 compounds.

**Prelab question:** What is the expected order of elution for these three compounds, *i.e.* least to most polar?

Prepare your silica gel column as directed below; before beginning your chromatography, have all your solvents ready as well as collection vessels (Erlenmeyer flasks) and three labeled and tared collection vials.

Use only 5 grams of silica gel for your column.

**Packing the column: (See Padias p. 176)**

Clamp the empty column in a vertical position and add a small plug of cotton or glass wool to the bottom of the column. The cotton is covered with a thin layer of sand. The buret is then packed as follows: 1) fill the column about one-third full with hexane; 2) add a slurry of the silica gel in hexane slowly to the top of the column while gently tapping the column to obtain even packing; 3) Once packed to the correct height gently add a small layer of sand to the top of the silica; 4) Drain the solvent to the top of the sand. The column is now ready to apply the sample.

**Sample application:**

The unknown sample weighs less than 100 mg. Weigh the entire sample in the vial on the analytical balance to the nearest 0.1 mg (0.0001 g). Dissolve the sample in the smallest amount of methylene chloride possible (*ca*. 1 mL or less, if possible). Apply this solution to the top of the column with a Pasteur pipet. Add another *very small amount* of methylene chloride to the vial to rinse it and add it to the column using the same pipet used to transfer the original sample. DO NOT add too much methylene chloride or your unseparated sample will be half way down the column before you actually begin. Allow the empty vial to air-dry and reweigh it to constant weight. Subtract the vial mass from the initial mass of the vial and sample to calculate the mass of sample that was loaded onto the column.

**Elution of the column:**

Depending on your unknown mixture, you may or may not see all three bands elute off of the column. Elute the column with hexane to begin. Never allow your solvent supply to become go below the top of the silica gel. Use air pressure to push solvent through the column. To do this you will use a rubber tube connected at one end to the air line in your hood, and connected at the other end to a rubber stopper that fits the top of the buret. Turn on a gentle stream of air and using your hand, hold the stopper on to top of the buret. You must hold the stopper the entire time you are eluting the sample. If the least polar compound is present, it will elute first as a yellow band. When all of the yellow has eluted, you can change collection vessels to prepare for the second band. If there is a relatively colorless fraction in-between any two fractions, it can be collected as waste*.* At this time you may change to a 1:1 diethyl ether: petroleum ether solvent system. When you change to the ethyl ether : petroleum ether solvent, you may notice cracking within the silica gel. Do not be alarmed. This is common when using ethyl ether, as the adsorption is slightly exothermic and ethyl ether has such a low boiling point. While having cracks within a column is not ideal, it will not affect your separation in this lab. If the compound of medium polarity is present, it will be pale orange to orange in color. Change vessels again when the orange fraction begins to come off. When this fraction has eluted, change vessels again and elute with acetone to collect the last fraction. This final fraction (if present) is a darker orange-red color.

If you are uncertain about when one component has finished and the next has begun, it is best to change flasks. Then, after you’ve analyzed the contents by TLC, you can combine fractions that are identical.

Run a TLC plate (1:1 diethyl ether : petroleum ether) of your purified fractions against the known standards to determine which of the unknowns you had in your mixture. Remember to always calculate Rf values for TLC plates.

Finally, place each purified sample in a tared RB flask (weigh on analytical balances) and evaporate the solvent on a rotary evaporator, and determine the amount you recovered.

To clean out your column, let it run dry. The silica mixture is easily emptied out of your column into the appropriately labeled waste container in the hood by blowing air into the bottom of the buret.

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**DATA SHEET**

|  |  |  |  |
| --- | --- | --- | --- |
| **NAME:** |  | **Section:** |  |

|  |  |  |
| --- | --- | --- |
| **Unknown number:** |  | |
| Mass of sample loaded onto the column: | | mg |

|  |  |  |
| --- | --- | --- |
| Compound | Mass recovered, mg | % recovery |
| Ferrocene |  |  |
| Acetylferrocene |  |  |
| Diacetylferrocene |  |  |

Percent Recovery of Each Individual Fraction relative to total mass loaded on the column

(Show Calculations)

Overall Percent Recovery (Show calculation)

TAPE TLC plates to report: (DO NOT STAPLE)

**Post-Lab Questions:**

Number the four compounds in order of their elution from a silica column, using benzene as the solvent. The compound numbered one should be the first compound off of the column.

|  |  |  |  |
| --- | --- | --- | --- |
| CH3(CH2)4OH | CH3CH2OCH2CH3 | CH3(CH2)5CH3 | CH3CH2CH2CO2H |

The surface of the silica gel contains Si-OH bonds. Considering this information, explain the order of elution of the ferrocene compounds based on interactions at the molecular level.

What effect will the following factors have on a chromatographic separation? Explain your answer.   
a) Too strong an adsorbent is used.

b) The elution fractions are too large.

c) The solvent level falls below the adsorbent.

Compare the advantages and limitations of column chromatography with thin layer chromatography.