The Separation of Organic Molecules using Column Chromatography

Because the acylation of ferrocene can be very tricky, we will just separate a 1:1 (w/w) mixture of the two compounds ferrocene and acetylferrocene via column chromatography.

Safety: treat all lab chemicals as if they are toxic. Most are. Keep them off your skin.

Reminder: this note is just to cover changes and suggestions. It is not a stand-alone experimental procedure. Base the prelab outline on the text and include these changes and suggestions.

Waste Disposal. When you are finished with your column, place it into your drawer to let the solvent evaporate. Next week, empty it into the Solid Waste container.

Part 1: TLC of the mixture.

Dissolve a small amount of the 1:1 mixture in dichloromethane (1-2 mL). Spot the mixture on a TLC plate (use known ferrocene and known acetylferrocene as reference spots). This means you should have three lanes. Run the TLC plate in 10:90 ethyl acetate:hexanes. Note all spots. If 10:90 is too polar, or not polar enough (how would you know?!?!), adjust accordingly and run another TLC.

Part 2: Column Chromatography – See page 196 – 197 (we are modifying this)

Packing Your Column: See figure 9.2 on page 188 for a final picture of what the column should look like. First, place a small amount of cotton (not glass wool) into the column and press into bottom neck with stopcock closed. Add a small amount of sand (just enough to fill the neck and be level at the bottom of the column). This is used to keep the bottom of the adsorbent (silica gel) level.

Because small columns can be difficult to pack using a true slurry method, or a true dry pack, we will use a modified method. See page 187 "Packing the Column," "Dry Packing Method"."

- Weight out 100 mg of the 1:1 (by weight) mixture of ferrocence/acetylferrocene).
- Weigh out ~5 g of silica gel.
- To the column with cotton and sand, add enough hexanes to fill ~ 2/3 full.
- Through your micro-powder funnel, add ~1/5 of your silica gel slowly. Let it settle. The tap the column with a rubber stopper (provided).
- Repeat above until all your silica gel is added.
- If the solvent level gets too high when you are adding the silica, slowly open the stopcock to drain some (into a collection vessel, of course). Close stopcock.
    - NEVER LET YOUR SILICA GEL RUN DRY!!
- Tap the side of the column with a rubber stopper. This will help the silica gel settle and "pack" it further. It will also help to level the "head" of the column.

To add your sample, follow "Adding the Sample" on page 189. Use a 10 mL Erlenmeyer in which to dissolve your sample. Be careful to NOT overheat the sample as it has a tendency to decompose. After adding the adsorbent to the dichloromethane solution, you need to carefully evaporate the solvent without overheating. Do this by carefully rolling the flask containing the damp sample over the surface of the hotplate (set to about 45 °C), avoiding any bumping of solvent. Bumping indicates overheating which may

lead to decomposition. If your sample turns dark, decomposition has happened. Note that dichloromethane boils at a very low temperature and does not need much heat to evaporate.

Begin separating the compounds by using first 100% hexanes (you should see a colored compound traveling down the silica gel first). Collect your fractions in disposable culture tubes (this keeps your fractions small). When this first colored band has completely eluted, change your collection tube. Drain until the solvent level is just above the surface of the sand. Now, switch the elution solvent to 100% ethyl acetate. Once you have added the ethyl acetate, switch the collection tube again, and continue until the second band has eluted.

Analyze each fraction collected by TLC. Spot using the known compounds as a reference.

Combine the like fractions (use our TLC plates of the fractions) into a larger vessel (Erlenmeyer, beaker, etc.). Evaporate the fractions with a stream of air and the hot plate on a setting of about 45 °C. Obtain a melting point of each residue obtained during limited-use-lab-hours.

Waste Disposal. Place solvents used for the chromatography into the liquid waste container. Place leftover silica gel into the container labeled as such. When you are finished with your compounds, place it into the solid waste container.

Postlab Questions – Provide answers to questions 1, 2, 4 and 5 on page 204 of the lab text.