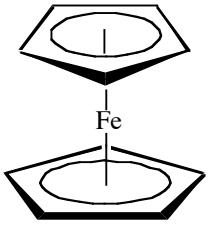
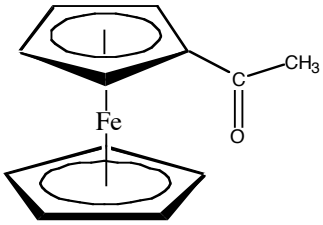
Experiment 5: Column Chromatography

Read pp 270-275 and 283-287, Chapter 19, in *LTOC*. Pay particular attention to pages 283-284. You may view the video: 19.1 Chromatography Column – Packing the Column at http://www.macmillanlearning.com/Catalog/studentresources/mohrig4e; however, be aware that you will be performing a small scale chromatographic separation, and the steps of the procedure that you follow will be somewhat different from those of the video.



Ferrocene

Bring Figure Page, Expt. 5 to the laboratory with you. You will separate a mixture that consists of two related compounds: ferrocene and acetylferrocene. **Outline** the steps of the following procedure:



Acetylferrocene

Obtain a Williamson microscale column with a plastic funnel and stopcock (see page 284 in *LTOC*). Check to make sure the stopcock is not loose. To measure the amount, fill the column about two-thirds full with alumina, then pour it from the column into a 10 mL Erlenmeyer flask. Place the column in a *buret* clamp in the fume hood and close the valve. Pour hexane (**caution! flammable**) into the column until the level of liquid reaches to the top of the glass. Add about 8 mL of hexane to the alumina in the flask and stir the mixture with a thin stirring rod to eliminate bubbles. Swirl the flask to suspend the alumina in the hexane, then *quickly* pour the entire slurry into the funnel at the top of the column (this is the tricky part!). Place the flask that contained the alumina under the column, open the valve and drain out some of the hexane. Do *not* allow the level of hexane to drop below the top of the alumina! If a significant amount of alumina was left behind in the flask, close the valve, (add a little more hexane to the flask, if necessary) and finish transferring the alumina. Do not worry if a small amount of alumina is left behind in the flask. Open the valve and drain enough hexane into an empty flask until the level of liquid in the column is about 5 mm above the top of the alumina. **It is very important when performing column chromatography that the column *never* be allowed to run dry! Never let the level of liquid fall *below* the top of the alumina!**

Weigh a clean, dry 10 mL Erlenmeyer flask and record this mass in your notebook.

The 1:1 ferrocene/acetylferrocene mixture is provided for you. Obtain 0.090 g of the mixture on weighing paper. Pour this solid mixture through the funnel onto the column of alumina. Using a Pasteur pipet, wash down any mixture that adheres to the funnel or walls of the column with a few drops of hexane. Tap the column to remove air bubbles. Place a 25 mL side-arm flask under the valve of the column. Fill a pipet with hexane. Open the valve on the column, then place the tip of the pipet close to the top of the liquid in the column, and carefully add the hexane in such a manner that the top of the alumina is not disturbed. When the sample has been applied in a narrow band at the top of the column, fill the column to the top of the glass with hexane.

As the column is eluted with hexane, ferrocene will travel down as a yellow band. Collect this yellow solution in the side-arm flask. Add hexane to the column as necessary so that it does not run dry. If you see crystalline material at the tip of the valve, wash it into the flask with a drop or two of hexane. When the yellow has been completely eluted, place a beaker under the column, and let the level of hexane lower until it is about 1 cm

above the top of the alumina. Fill the column to the top of the glass with a 50/50 mixture of hexane and diethyl ether. Open the stopcock and continue to collect the colorless solution that elutes off the column in the beaker. During this time, an orange band will travel down the alumina. When this orange band (acetylferrocene) reaches the bottom of the column, replace the beaker with the tared 10 mL flask and collect the orange solution. Once again, it is very important throughout the elution that the column never be allowed to run dry!

When elution of both yellow and orange bands is completed, shake out the contents of the column into a beaker. Rinse out the glass column with water, adding the rinses to the beaker. The mixture of alumina and liquid (including the solvents that eluted in between the bands) must be disposed of in the appropriate **Laboratory Byproducts** jar.

A solution of the original mixture will be provided for you. Obtain a silica gel TLC plate and spot this solution on the left-hand side of the plate, allowing enough room on the right-hand side for two more spots. Spot the right-hand side of the plate with the solutions from the yellow band and the orange band (the plate should have 3 spots in all). Develop the plate with 30:1 toluene–absolute ethanol. Measure the distances from the origin to the solvent front and to all spots on the TLC plate.

Place a rubber stopper on the side-arm flask that contains the yellow ferrocene solution. Attach the side-arm to a vacuum outlet with rubber tubing. Slowly open the vacuum to evaporate the solvent (see Figure Page, Expt. 5). Use a steam bath if heat is required.

You will purify this ferrocene by sublimation. **Read** pp 236-239, Chapter 16, in *LTOC* (see Figure Page, Expt. 5). Push a centrifuge tube through your black neoprene adapter and place it in the mouth of the side-arm flask. Place the apparatus in a thermowell and clamp it so that the bottom of the flask is touching the sides of the thermowell. Attach the side-arm of the flask to the vacuum with rubber tubing. First, turn on the vacuum. Next, fill the centrifuge tube with pieces of ice (this will serve as a "cold finger"). Finally, turn on the thermowell to a high setting.

Watch as the ferrocene sublimes onto the surface of the cold finger and the upper walls of the flask. When the bottom of the side-arm flask is empty of solid material, wrap the sides in aluminum foil so all the ferrocene collects on the cold finger. Turn off the thermowell, then turn off the vacuum. *Very slowly and carefully* disconnect the rubber hose from the side-arm (a blast of air can knock the sublimate off the cold finger). **Carefully**, remove the centrifuge tube from the flask. Scrape the ferrocene off of the tube and onto *tared* weighing paper. Obtain the weight and melting point of the ferrocene this week, since some of it will sublime away. The side-arm flask will become more difficult to clean over time, so be sure to wash it before leaving the lab.

Evaporate the solvent from the orange solution by heating the flask on a steam bath. Obtain the weight and melting point of the acetylferrocene next week.

Melting points of the compounds

Ferrocene	174-176°
Acetylferrocene	81-83°

Name _____ Date _____

T. A. _____ Lab period _____

Results and Calculations (to be handed in two days after the next lab period, with the discussion for Expts. 4 & 5)

Calculate the percent recovery of sublimed ferrocene from the mixture.

Melting range for ferrocene _____

Calculate the percent recovery of acetylferrocene from the mixture.

Melting range for acetylferrocene _____

Determine the R_f values for all spots seen on the thin layer chromatogram. Indicate the color of the spot that corresponds to each R_f.