**Modifications to CHEM237 Labs**

It is going to be important for you to thoroughly read the lab and associated background reading prior to coming to do the lab. The textbook cross references itself numerous times and determining exactly what to do requires you to pay attention while reading. I would even suggest writing up a “pre-lab” detailing the procedure you’ll be working on, from start to finish.

That said, many of the procedures in the text refer to glassware you do not have in your drawer or expensive chemicals that can be replaced with cheaper ones that are just as effective. This document discusses all of the changes to the procedures in the text and also includes some tips to make things easier.

**Experiment #5 – Column Chromatography**

- According to Gilbert and Martin [Figure 6.11 and related text on page 189], you will use a glass buret or chromatography column to perform your column chromatography experiment. You will not. In its place, you will use pipets which are very similar in size to Pasteur pipets.
- Gilbert and Martin [page 189] instruct you to use approximately 0.1g of the fluorene/9-fluorenone mixture. This quantity of material is way too much for the columns you will be using. You should use approximately 0.050g (~50mg) of the sample. Measure the mass to the correct number of significant figures.
- Column preparation: prepare your column with all materials dry, not wet as instructed in Gilbert and Martin.
- Column assembly: Insert a small amount of cotton in the constriction of your glass column. The only function of the cotton is to keep the solid column material [sand and alumina] in the column while allowing the liquid to pass through at a reasonable rate. It is vital to your performing experiment #5 that you do not use too much cotton and that you do not force the cotton too tightly into the pipet constriction. If the cotton is inserted into the constriction too tightly, it can become an impermeable barrier. After you have properly inserted the cotton into the pipet, add sand to a thickness of approximately 0.5cm. Make sure the top of the sand is level. Then add alumina to a height of approximately 7cm. The alumina [aluminum oxide, \(Al_2O_3\)] is the stationary phase. Make sure the top of the alumina is level. Finally and carefully add sand to a thickness of approximately 0.5cm. Add the sand carefully so you do not disturb the top of the alumina. When done you want the alumina-sand interface to be level and undisturbed.
After you have properly prepared your column, wash it by carefully pipetting petroleum ether to the top of the column. Make sure you add the petroleum ether carefully. It is very easy to use too much pressure when adding the petroleum ether to the top of the column. If this happens you can seriously disrupt the sand alumina interface and possibly making your column unusable. NOTE: when adding the petroleum ether to the top of your column, make sure there is a container to collect the liquid that comes through the column.

Load [apply the sample to the column] the sample as described in Gilbert and Martin. Dissolve the sample in approximately 0.5mL of petroleum ether with a little bit of methylene chloride added to it.

Begin the eluting process. Collect fractions [in clean and dry containers] that are approximately 1mL in volume. NOTE: in a rough rule of thumb, there are approximately 20 drops per milliliter. I do not know how many drops there are in 1mL using your column chromatography pipets.

All of the fluorene should come off of the column in a volume of 3-4mL of eluant collected. You should test each fraction to see if it contains fluorene. Use the procedure given in Gilbert and Martin. Combine all fractions which contain fluorene then remove the solvent by reduced pressure evaporation.

After all of the fluorene has come off of the column, change the solvent from petroleum ether to acetone. Do not change the eluting solvent from petroleum ether to methylene chloride as instructed in Gilbert and Martin. Continue eluting with acetone until all of the yellow 9-fluorenone has come off of the column.

IMPORTANT NOTE: do not allow the column to go dry while you are eluting the fluorene and/or 9-fluorenone from the column. If you do, you can seriously disrupt any separation you that you may have achieved. This means you must keep the elution solvent above the layer of sand at the top of your column.

IMPORTANT NOTE: In the beginning of the elution process, the drops should be exiting the column at the rate of 1 drop per 2-3 seconds. You should notice that the drop rate will slow down somewhat as you continue the elution process.