4.2. Expert Experimentalist: *"Setting the Pace"*

***** This is a two-day experiment. Do not start this experiment unless you have two free lab days to complete it.** ***

Techniques Checklist:

- Picking the correct eluent ❑
- Adsorbtion of a crude mixture onto silica gel ❑
- Separating complex mixtures using gradient elution ❑

Pre-lab Discussion:

- Suggest limited list of eluent solvent systems
- Discuss sample adsorption and gradient elution strategies

Equipment:

- Identical to CC Level

Goal:

- Separate a complex mixture of three compounds using gradient elution flash column chromatography.

Experiment Outline:

- You will receive 20 mL of an ether/pentane solution containing 0.20 g of guaiazulene, 0.20 g of 9-fluorenone, and 0.20 g of 3-methylanisole.
- Analyze the mixture by TLC using the solvent systems discussed in the pre-lab lecture - *see TLC Guide*.
- Decide on an appropriate starting eluent.
- Decide on the silica gel to compound ratio.
- Prepare the column - *See Flash Column Chromatography Guide*.
- Adsorb the mixture onto a small amount of silica gel, according to the instructions provided in the pre-lab lecture and the DLTM.
- Apply the mixture to the column, being sure to rinse the sides and apply an extra layer of sand to the top of the column.
- Run the column.

• TLC all of the fractions from your column, have your TLC plates checked by an instructor, and reproduce them in your lab notebook.

• Calculate R_f values for the three compounds in the chosen TLC solvent mixture.

Results:

• To obtain your "EE Rating" in Purification by Flash Column Chromatography, you must successfully separate all three components of the mixture by TLC and correctly calculate the corresponding R_f values.