

**WARNING NOTICE:** The experiments described in these materials are potentially hazardous and require a high level of safety training, special facilities and equipment, and supervision by appropriate individuals. You bear the sole responsibility, liability, and risk for the implementation of such safety procedures and measures. MIT shall have no responsibility, liability, or risk for the content or implementation of any of the material presented. [Legal Notices](#)

## 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
- Adsorption 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.