4. Purification by Flash Column Chromatography

4.1. Competent Chemist Rating: *"Looks Can Be Deceiving"*

Techniques Checklist:

- Analyzing mixtures by TLC ❑
- Assembling a silica gel column ❑
- Applying crude mixtures to a silica gel column ❑
- Separating simple mixtures with a silica gel column ❑

Pre-lab Discussion and Required Reading:

- Theory of column chromatography : Zubrick Ch. 27
- TLC - polarity/solvent systems: Zubrick Ch. 28, LLP Ch. 9.3.1
- Setting up a silica gel column: Zubrick Ch. 29, LLP Ch. 11.6
- Applying crude mixtures to the column
- Running a flash column

Equipment:

- Flash Chromatography Column
- Air flow apparatus (stopper, T-valve, screw clamp, tubing)
- 100-mL Round-bottomed flask
- Test tubes - 18x150 mm
- Test tube rack
- TLC plates and spotters
- UV lamp
- Large plastic funnels

Digital Lab Techniques Manual:

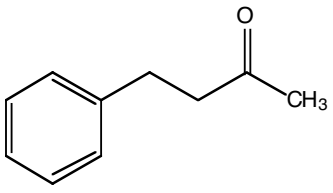
- 3. TLC: The Basics
- 10. Column Chromatography

Goal:

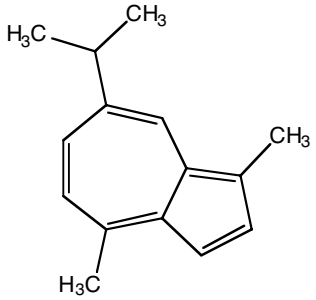
- Purify a contaminated compound using silica gel flash column chromatography.

Experiment Outline:

- You will receive 2 mL of an ether/pentane solution containing 1.00 g of benzylacetone contaminated with a small amount of guaiazulene.
- Make a TLC sample, and analyze this mixture by TLC, using 10% ethyl acetate/hexanes as the solvent system - *see TLC Guide*.
- Record the R_f values.
- Prepare the column in the hood, using 10% ether/pentane and 50 g (about 5'') of silica gel - *see Flash Column Chromatography Guide*.
- Elute the column with 10 mL of pentane.
- Apply your sample to the column, being careful not to disturb the top layer of sand. Rinse the sample flask three times with 1 mL pentane each, and use the rinses to wash the sides of the column.
- Run the column - *See Flash Column Chromatography Guide*.
- Monitor the fractions by TLC - *See TLC Guide*.
- Concentrate the set of fractions containing pure benzylacetone.
- Weigh the purified compound and prepare a GC sample.
- Obtain a TLC and a gas chromatogram of the purified compound.



Benzylacetone



Guaiazulene

Results:

- To obtain your "CC Rating" in Purification by Flash Column Chromatography, you must collect at least 0.95 g of benzylacetone. This sample must be at least 95% pure as demonstrated using gas chromatography. Your sample must also be submitted to the TA for possible weight and purity verification.