Lab 04: Additional Notes on Chromatography (parts A & D)

Assigned Reading:
Read Experiment 5 in Pavia, pages 80 to 86
Read Technique 12, pages 669 to 680, 684, 685
Read Technique 14, all sections except 14.3
Read the footnotes found in Notes on your Laboratory Notebook Handout regarding TLC plates.

Experimental Considerations:
Modification to the Procedure in the text
We will only do Part A & D. We will use two commercial TLC plates that are approximately 7.5 cm x 2.5 cm instead of one plate that measures 10 cm by 5.3 cm. Be sure your record the type of plate used (manufacturer, solid adsorbent, etc.) THESE PLATES ARE EXPENSIVE! DO NOT WASTE THEM!

We have available, in the lab, commercial micro pipets suitable for spotting. Most students obtain better results by making their own (your instructor will demonstrate). You may use either, but be sure your procedure indicates which ones you used.

Change in procedure:
Part A:
- Instead of 100% CH₂Cl₂, we will use 50:50 mixture of CH₂Cl₂ and hexane for the developing solvent.
- We will visualize our spots using a UV-lamp rather than using iodine crystals.

Part D:
- You need five test tubes (1,2a, 2b, 3, 4):
  - Rinse the column with 3 mL hexane and collect in TT #1:
  - Load sample
  - Switch to TT #2a
    - Continue to elute with hexane until yellow band is ~1.5 cm from cotton
    - Wash any crystals into TT#2a
  - Switch to TT#2b
    - Continue to elute with hexane until yellow band gets just above the cotton (~0.5cm). Be sure to wash any white crystals into this test tube before moving to TT#3.
  - Switch to TT#3
    - Elute with 70% hexane/ 30% Acetone until you collect all of the yellow compound.
  - Switch to TT#4
    - Allow column to dry.
    - You do not need any acetone for the last test tube.
  - Evaporate test tube 2a, 3 to ~1/4 mL and spot on TLC 3-5 times.
  - Evaporate to dryness and take two MPs.

Postlab Questions
Answer these questions in your lab notebook after your conclusion is a section titled “Post-lab Questions”

- Answer questions 1, 2, and 3 (page 87)
- Answer questions 1, 3, 4 (page 711)