Name: _____

Name: _____

Locker Number: _____

CHEMISTRY 354-355

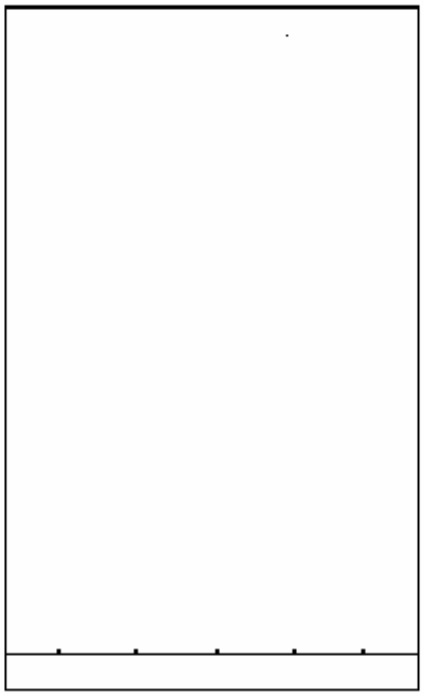
Experiment 5

CHROMATOGRAPHY

PART A. THIN-LAYER CHROMATOGRAPHY

1. Write the identification code for your unknown here.

2. Make a sketch of the TLC plate, based on your laboratory results. Label each spot by the name of the compound.



3. Assign an R_f value to each substance in the sample.

4. Identify the unknown substances in the mixture that you were assigned.

PART B. SELECTING THE CORRECT SOLVENT FOR THIN-LAYER CHROMATOGRAPHY

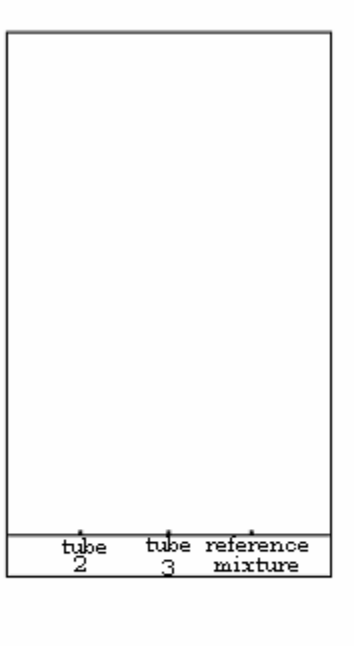
5. Record the names and structures of the two compounds that you ran on thin-layer chromatography.

6. Which solvent resolved the two compounds successfully?

7. For the other two solvents, explain, in terms of their polarities, why they failed.

PART D. COLUMN CHROMATOGRAPHY

8. Sketch the TLC plate obtained after your tests on the samples in test tubes 2 and 3.



9. What do the TLC results indicate about the purity of the two samples obtained from the column chromatography?

10. Record the melting points for the dried solids found in test tubes 2 and 3. What do they indicate about the purity of the two samples?

ANSWER THE FOLLOWING QUESTIONS

1. Each of the solvents given should effectively separate one of the following mixtures by TLC. Match the appropriate solvent with the mixture that you would expect to separate well with that solvent. Select your solvent from the following: hexane, methylene chloride, or acetone. You may need to look up the structures of the solvents and compounds in a reference book.

a. 2-phenylethanol and acetophenone

b. bromobenzene and *p*-xylene

c. benzoic acid, 2,4-dinitrobenzoic acid, and 2,4,6-trinitrobenzoic acid.

2. Consider a sample that is a mixture composed of biphenyl, benzoic acid, and benzyl alcohol. Predict the order of elution of the components in the column chromatography of this mixture. Assume that the chromatography uses a silica column and that the solvent system is based on cyclohexane, with an increasing proportion of methylene chloride being added as a function of time (HINT: See Table 12.3)

Fastest _____ > _____ > _____ Slowest

3. A yellow compound, dissolved in methylene chloride, is added to a chromatography column. The elution is begun using petroleum ether as the solvent. After six liters of solvent had passed through the column, the yellow band still had not traveled down the column appreciably. What should be done to make this experiment work better?

4. You have 0.50 g of a mixture that you wish to purify by column chromatography. How much adsorbent should you use to pack the column? Estimate the appropriate column diameter and height.

5. In a particular sample, you wish to collect the component with the *highest* molecular weight as the *first* fraction. What chromatographic technique should you use?

6. A colored band shows an excessive amount of tailing as it passes through the column. What can you do to rectify this problem?

7. A student spots an unknown sample on a TLC plate and develops it in dichloromethane solvent. Only one spot, for which the R_f value is 0.95, is observed. Does this indicate that the unknown material is a pure compound? What can be done to verify the purity of the sample?

8. A student spots an unknown sample of a TLC plate and develops it in pentane solvent. Only one spot, for which the R_f value is 0.05, is observed. Is the unknown material a pure compound? What can be done to verify the purity of the sample?

9. A *colorless* unknown substance is spotted on a TLC plate and developed in the correct solvent. The spots do not appear when visualization with a UV lamp or iodine vapors is attempted. What could you do in order to visualize the spots if the compound is:

   a. an alkyl halide

   b. a ketone

   c. an amino acid

   d. a sugar