**Chem 230, Fall, 2014**
Homework Set # 2
Do by Oct. 7

Short Answer Questions

1. Classify the phase of the stationary phase in the following types of chromatographic separations as "solid", "liquid-like", or "liquid":
a) Coated (unbonded), open tubular gas chromatography **liquid**
b) Gas chromatography with zeolite packing material   **solid**
c) Bonded, open tubular gas chromatography **liquid-like**
d) Ion-exchange chromatography   **solid (possibly liquid depending on glass transition point)**
e) Normal phase HPLC with bare silica   **solid**
f) Reversed-phase HPLC with C18 (octadecyl group) bonded to silica **liquid-like**

2. What effect does an increase in K_C have on retention times? **It will cause an increase in retention times** *(greater affinity for stationary phase means more time spent in stationary phase means later elution time).*

3. In open tubular GC, which van Deemter term is normally negligible?
The A term *(hard to have multiple paths if the whole tube is open).*

4. Given the chromatograms below (actually one chromatogram with two parts expanded), answer the following questions.
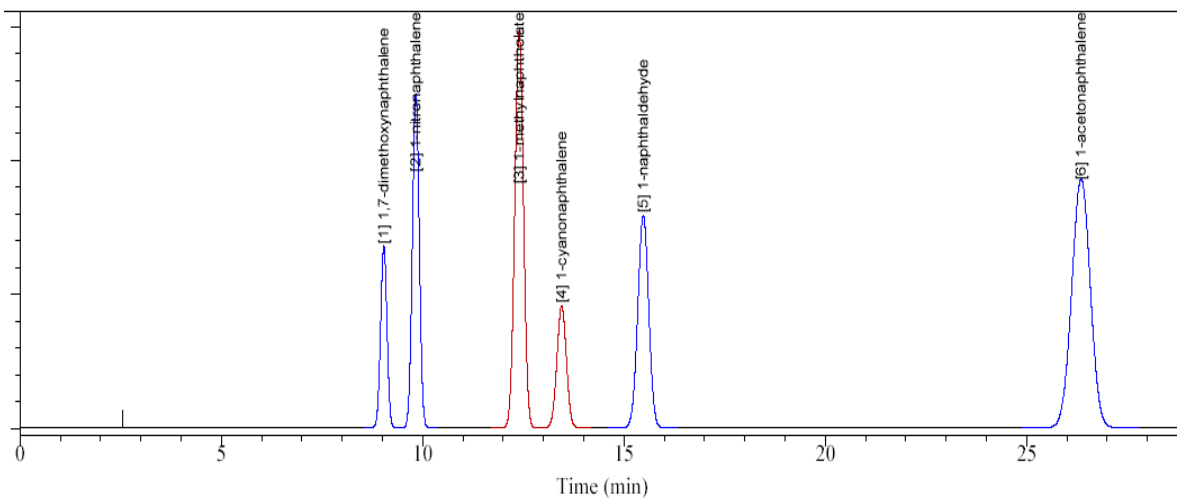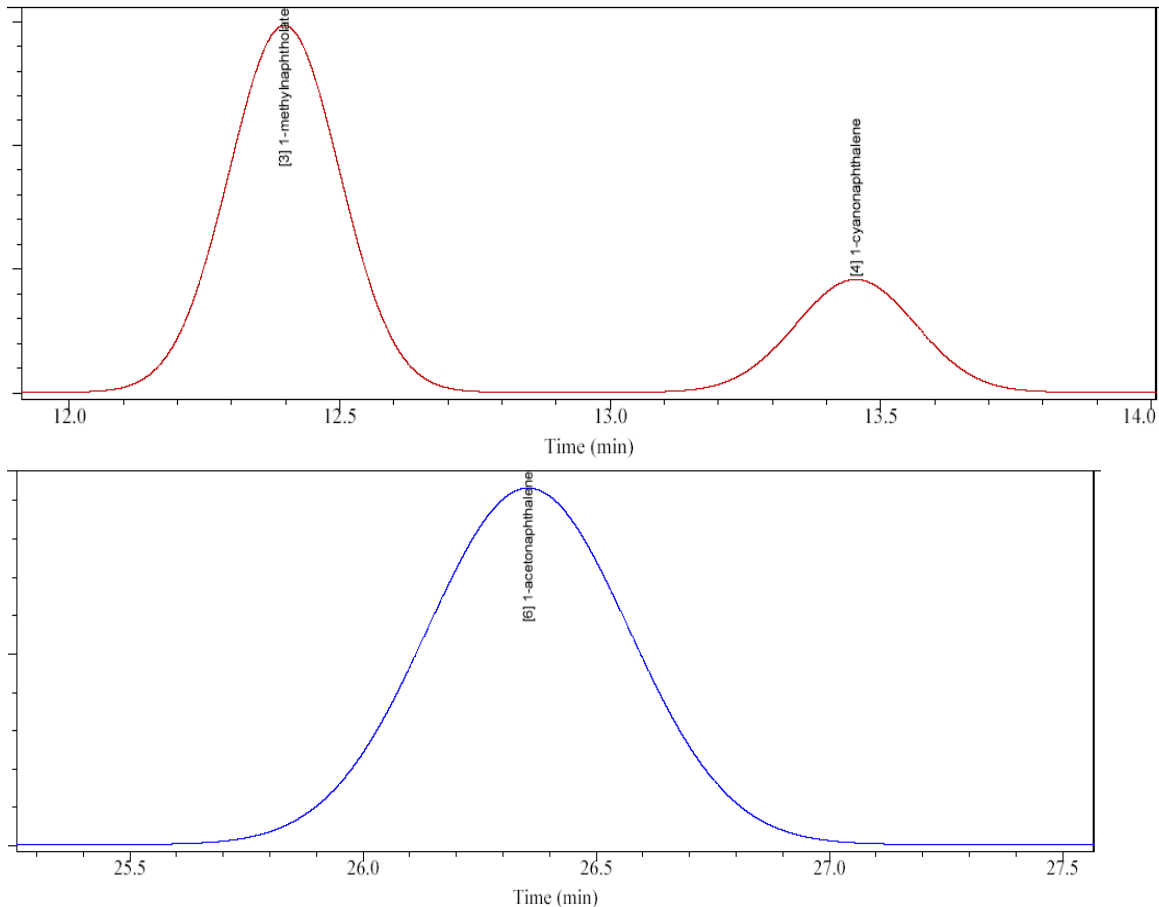
a) Calculate the resolution of the critical pair of compounds
Assuming the critical pair of compounds are those in red,
resolution = d/w_{ave} = (13.45 − 12.40)/[0.5(12.62 − 12.19) + 0.5(13.70 − 13.23)] = **2.33**
b) Calculate the retention factor for the last peak (the hold-up time (t_M) is indicated by the vertical 'tick mark' on the baseline of the chromatogram).
k = (26.37 − 2.50)/2.50 = **9.55**
c) The plate number of the column (from the last peak eluted).
N = 16[26.37/(26.80 − 25.90)]^2 = **13,700**
d) Is the analysis time unnecessarily long? Why?
Yes. If the critical was closer to baseline resolved (as opposed to the current resolution which is more than needed), the total time needed could have been decreased. If this was done by GC, it would make sense to increase the oven temperature a little to speed up the separation.

5. A reversed-phase HPLC column of dimensions 4.6 mm diameter by 150 mm length is used at a liquid flow rate of 1.0 mL/min. An unretained compound elutes after 1.12 min. A compound with a K_{ow} of 31 takes 2.63 min. to elute when using a mobile phase of 90% water/10% acetonitrile.
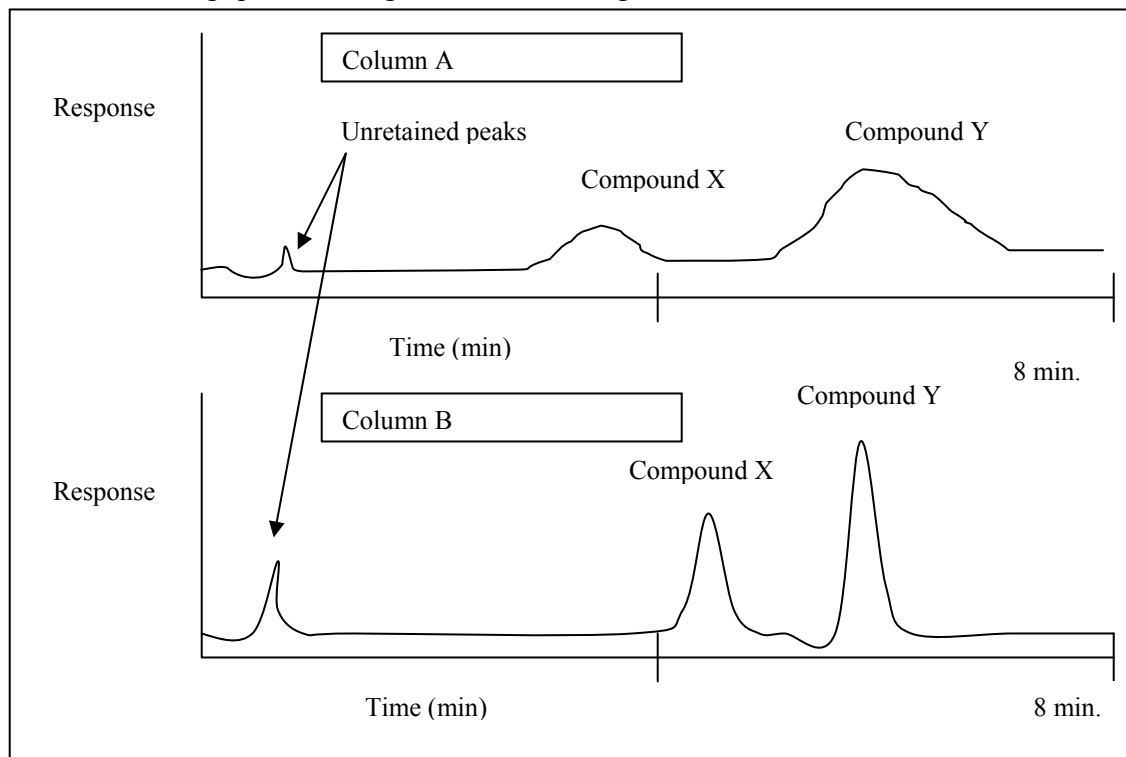a) Calculate the mobile phase volume
V = F·t_M = (1.0 mL/min)(1.12 min) = **1.12 mL**

b) Assuming the K_C is equal to the K_{ow}, calculate the stationary phase volume.
k = (2.63 min. - 1.12 min)/1.12 min = 1.35
$K_C = k(V_M/V_S)$ or $V_S = kV_M/K_C$ = 1.35(1.12 mL)/31 = **0.049 mL**

6. The following questions regard the chromatograms shown below:

a) Which column has greater efficiency?
Column B (narrower peaks at the same elution time)
b) Which column gives a greater separation factor (for X and Y)?
Column A – Compound X and Y are better separated
c) Which column gives greater resolution between X and Y?
Colum B (It looks like two peaks could fit between X and Y vs. one for Column A).

7. How does temperature affect the van Deemter B term in GC?
Higher temperature increases the B term due to faster diffusion.

8. Why is the van Deemter B term in GC much greater than in HPLC?
Diffusion of molecules in the gas phase is much greater than in the liquid phase.

9. List the following columns in order of increasing susceptibility to tailing or fronting for injection of equal masses of analyte:
0.25 mm diameter, 0.25 μm film thickness, coated open tubular GC column
0.45 mm diameter, 1.0 μm film thickness, coated open tubular GC column
3.2 mm diameter packed GC column

The first column would be most susceptible to overloading causing tailing or fronting and the packed column would be the least suceptible.

11. List five major components of a chromatograph.
1. Mobile phase reservoir
2. Fluid flow/pressure control or delivery
3. Injector
4. Column
5. Detector
6. Data Processor
7. Fraction Collector
(Note: not all chromatographs will have all of these components)

12. What is the most common way to increase retention of analytes in gas chromatography? in HPLC?
In GC – decrease temperature
In HPLC – increase % of weak solvent (more polar solvent in reversed phase HPLC or less polar solvent in normal phase HPLC).