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

Short Answer Questions

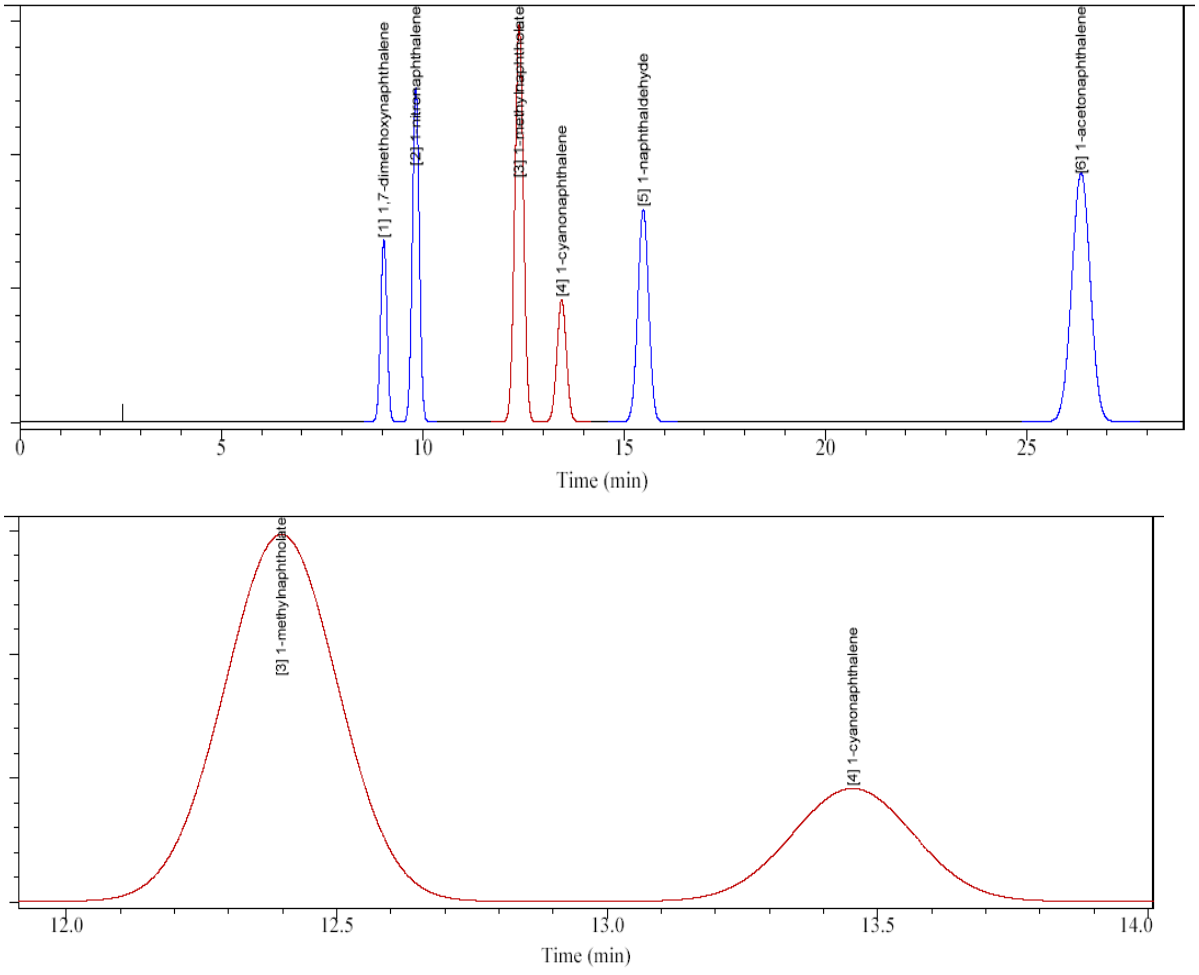
1. Classify the phase of the stationary phase in the following types of chromatographic separations as "solid", "liquid-like", or "liquid":

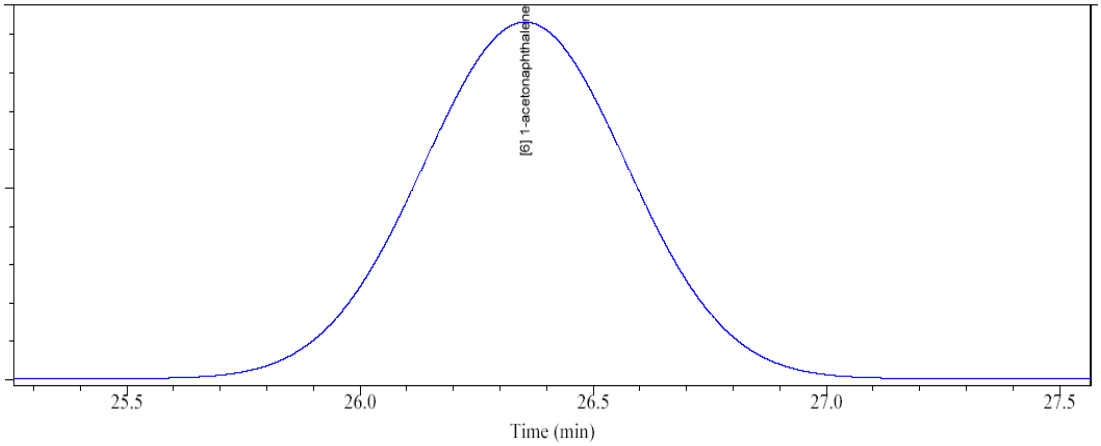
a) Coated (unbonded), open tubular gas chromatography
b) Gas chromatography with zeolite packing material
c) Bonded, open tubular gas chromatography
d) Ion-exchange chromatography
e) Normal phase HPLC with bare silica
f) Reversed-phase HPLC with C18 (octadecyl group) bonded to silica

2. What effect does an increase in K_C have on retention times?

3. In open tubular GC, which van Deemter term is normally negligible?

4. Given the chromatograms below (actually one chromatogram with two parts expanded), answer the following questions.

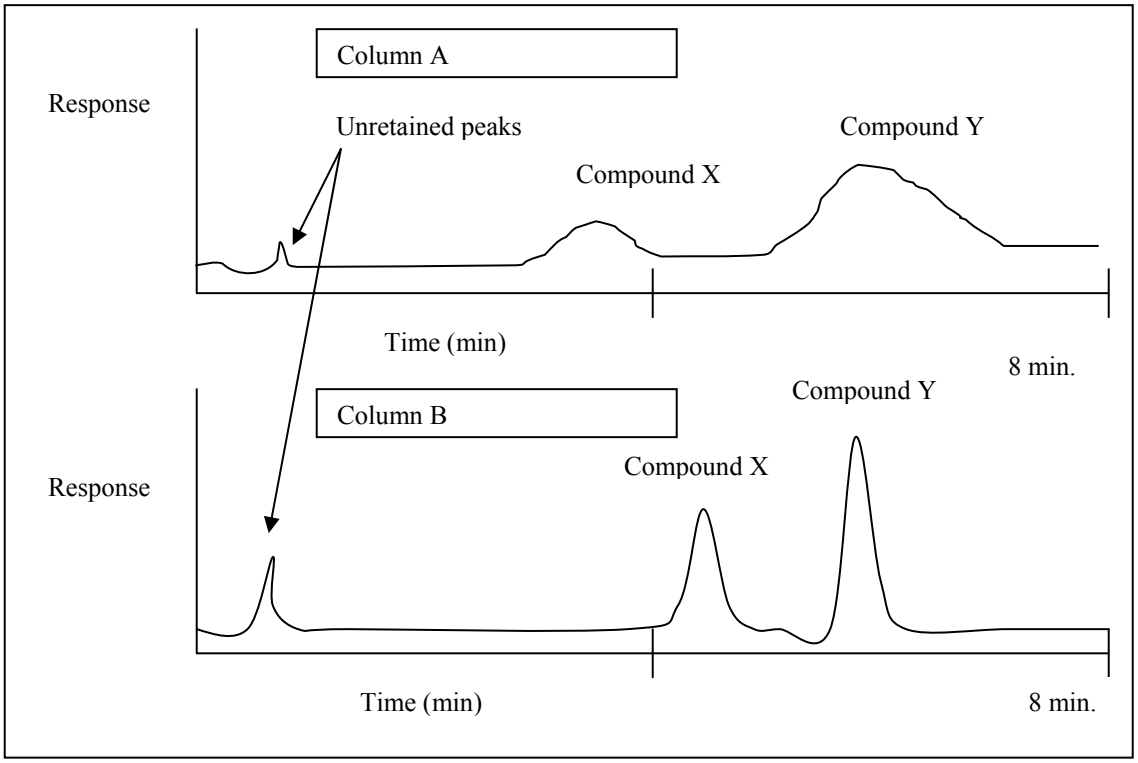




a) Calculate the resolution of the critical pair of compounds
b) Calculate the retention factor for the last peak (the hold-up time (t_M) is indicated by the vertical 'tick mark' on the baseline of the chromatogram).
c) The plate number of the column (from the last peak eluted).
d) Is the analysis time unnecessarily long? Why?

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
b) Assuming the K_C is equal to the K_{ow}, estimate the stationary phase volume.

6. The following questions regard the chromatograms shown below:



a) Which column has greater efficiency?
b) Which column gives a greater separation factor (for X and Y)?
c) Which column gives greater resolution between X and Y?

7. How does temperature affect the van Deemter B term in GC?

8. Why is the van Deemter B term in GC much greater than in HPLC?

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

11. List five major components of a chromatograph.

12. What is the most common way to increase retention of analytes in gas chromatography? in HPLC?

Longer Problems – to be turned in

1. The table below shows the retention times and peak widths for 3 compounds separated by reversed-phase HPLC using a 250 mm length x 4.6 mm inside diameter column with an eluent of 45% acetonitrile, 55% water:

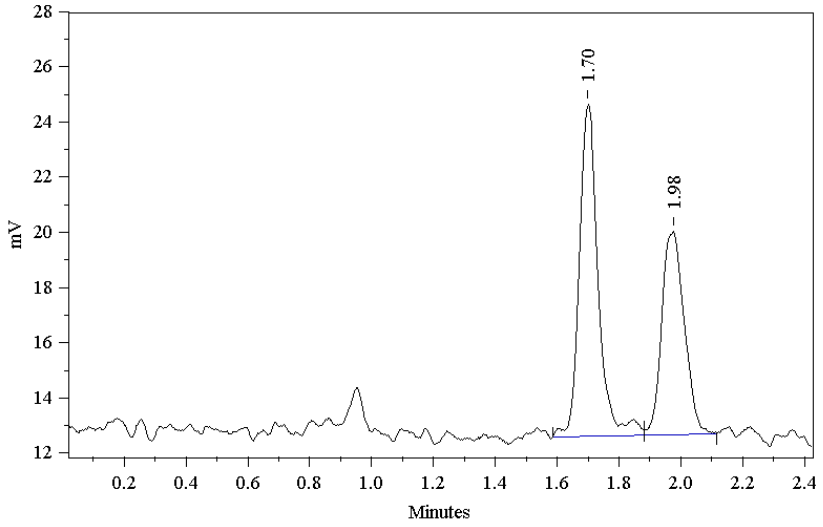
Compound	Retention time (min.)	Peak Width at half height (min.)	pK_a
phenol	4.72	0.21	9.98
2-nitrophenol	5.68	0.25	8.39
2-methylphenol	5.91	0.26	10.09

Note: $w_b = 1.70 * w_{1/2}$

a) If the flow rate is 1.00 mL/min and the column is 62% by volume packing material (including stationary phase), calculate the time it takes for mobile phase to flow through the column.
b) Calculate the capacity factor for phenol.
c) Calculate the plate number for the column using the last eluting compound.
d) Calculate the resolution for the two least well resolved peaks.
e) Calculate the separations factor for the two least well resolved peaks.

2. An open tubular GC column is 0.25 mm in diameter and 30 m long. The column wall is coated by a film that is 0.25 μm thick. The flow rate is 1.0 mL/min.
a) How long does it take He (an unretained gas to flow through the column)?
b) An unknown gas takes 12.1 min to elute from the column. What is the distribution constant (K_C) for this gas? What is its retention factor?

Questions 3 – 5 below deal with the following chromatogram:
This chromatogram shows the separation of glucose (1.70 min) from levoglucosan (1-6-anhydro-β-glucose) (1.98 min). The chromatogram was obtained with a 150 mm length C18 column using 90% water, 10% acetonitrile at a flow rate of 1.0 mL/min. A Table below gives peak widths and areas.



Name	Ret. Time (min)	Conc. (mg/L)	Peak Height (mV)	Width at base (min)
Unretained	0.95	-	-	-
glucose	1.70	1.0	12.05	0.115
levoglucosan	1.98	1.0	7.36	0.133

3. Determine retention factor (k) of glucose and the resolution (R_S) between glucose and levoglucosan.

4. Based on the chromatography, which compound is more polar. Explain.

5. Is the separation optimized for samples containing only these two compounds? If not, how could it be improved without changing the column?

6. Compounds X and Y are run by GC on two open tubular columns, a DB-1 (100% methyl) and a DB-17 (50% methyl, 50% phenyl), that have identical dimensions (diameter, film thickness and length) under identical conditions. The retention times and peak widths are show in the Table below. Note: phenyl groups interact more strongly with polar groups than methyl groups.

Compound	DB-1 Ret. time (min.)	DB-1 Peak Width (min.)	DB-17 Ret. time (min.)	DB-17 Peak Width (min.)
unretained	1.11	-	1.12	-
X	10.21	0.32	11.07	0.35
Y	10.47	0.33	10.37	0.32

Peak width at baseline listed

a) Which compound is more polar? Explain your answer.
b) What is the primary reason for improved resolution in the DB-17 column? Consider changes to N, α, and k and explain your answer.

7. An alternative fuel is to be analyzed by packed column GC. It is known to have around 10 compounds. When several standards of the 10 compounds are run by GC at 35°C (the lowest practical operating temperature), k values are found to range from 0.2 to 0.7. Is a successful analysis of this fuel likely with this column? Explain why or why not. (8 points)