

**Due by 3:00 PM Friday, February 26**  
**NO LATE PAPERS ACCEPTED!**

Complete these problems on separate paper and staple it to these sheets when you are finished. Please put your name or initials on each sheet as well. Clearly mark your answers. YOU MUST SHOW YOUR WORK TO RECEIVE CREDIT.

**Instructions**

- This is **NOT** an open-book, open-note exam. You **MAY NOT** consult any human or nonhuman resource besides Dr. Lamp as you complete the exam. This exam **MUST** be completed **INDIVIDUALLY** and in your own words. Group work or plagiarism will result in a zero for the exam.
- You will be allowed to ask Dr. Lamp a maximum of two (2) questions regarding the exam. Additional questions may be asked at a 3-point penalty per question. If you are working on the exam in the evening, you may try to reach Dr. Lamp on his cell phone at 660-341-0067 before 10:00 PM.
- Before opening the exam, prepare for it like you would for a traditional, in-class exam. Review concepts and examples from the text, as well as those discussed in class. This preparation will help to maximize your effort on the exam and allow you to complete it more efficiently.

**Time Restriction**

You may spend no more than two (2) hours working on this exam. This must be in one continuous block of time. You are on your honor to adhere to this restriction and record the time spent in the chart below.

Date	Time Began	Time Finished	Total Time
Total Time Spent on the Exam			

**Pledge**

I pledge on my honor that I have completed the exam in accordance with the above instructions and that I have not provided or received unethical assistance. I realize that failure to comply with these instructions will result in a score of zero on the exam.

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

Complete these problems on separate paper and staple it to this sheet when you are finished. Please initial each sheet as well. Clearly mark your answers. YOU MUST SHOW YOUR WORK TO RECEIVE CREDIT.

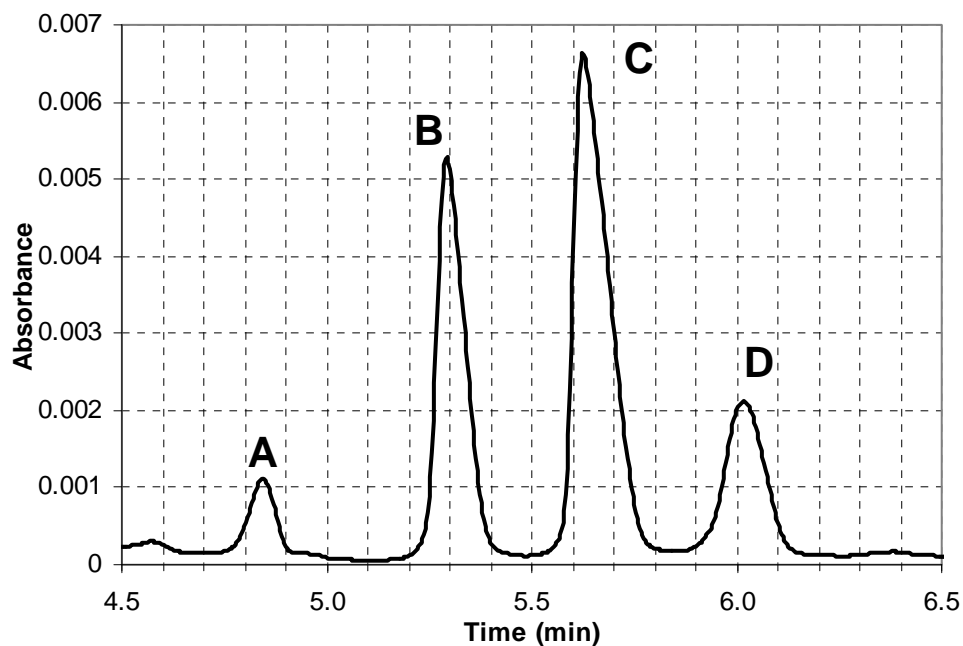
**Warm-up (2 points each).**

1. The \_\_\_\_\_ utilizes a series of heated filaments in its detection mechanism.
2. In \_\_\_\_\_ a small fiber is inserted into the sample container to allow analyte to adsorb to its surface. The fiber can then be introduced into a GC for desorption and analysis.
3. Capillary electrochromatography, aggregates of surfactants called \_\_\_\_\_ form a *pseudo* stationary phase to allow partitioning of analytes.
4. The \_\_\_\_\_ is an equilibrium constant that describes the tendency for a solute to exist in the stationary phase relative to the mobile phase during a chromatographic separation.

**Complete seven of the following. Be clear and concise. Clearly indicate which problem is not to be graded. (14 points each)**

5. Describe each of the following, indicating why each is utilized in a separations experiment.
  - a. A guard column in HPLC.
  - b. Split injection in GC.
  - c. A buffer in CE.
6. Selection of a detector for separations often involves a tradeoff between universality (or selectivity) and sensitivity. Briefly describe why this is so, using examples of specific gas chromatography detectors to illustrate your point.
7. Briefly describe the mechanism of separation in capillary zone electrophoresis. What parameters can be changed to optimize separation conditions in CZE?
8. Briefly describe how an ELSD works and why it is more universal than UV-Vis detection.
9. What is the general elution problem? Sketch a generic chromatogram to illustrate the problem. Describe one approach in gas chromatography and one approach in liquid chromatography aimed at addressing the general elution problem. From a physical chemistry perspective, how does each approach hope to alleviate the problem?
10. Consider the Van Deemter equation. Which term is likely to be the primary contributor to band broadening in GC? Justify your answer. Does the same argument hold true for LC and CE? Why or why not?

For problems 11 and 12, consider the chromatogram below that was obtained for a reverse-phase HPLC separation on a 25 cm column, using UV absorbance detection. Unretained compounds elute in 0.15 minutes. NOTE: Problems 11 and 12 are independent of one another!



11. Complete the following.
  - a. Calculate the average plate height for the separation. Based on this average, calculate the number of theoretical plates for the separation.
  - b. Calculate the selectivity factor of compound D over compound C.
  - c. Calculate the resolution of compounds C and B.
  - d. Which compound is the most polar? Justify your choice.
  
12. Your boss looks at the chromatogram and makes the following statement: “Well, it is clear to me that compound D is present at about 2 times the concentration of compound A and that compound B is methamphetamine since it elutes at 5.39 minutes under these conditions.” Discuss the validity of this statement.

### Possibly Useful Information

$A = \log(P_0/P) = \epsilon bc$	$\pi = 3.14159$
$k'_A = K_A \frac{V_S}{V_M} = \frac{t_R - t_M}{t_M}$	$\alpha = \frac{K_A}{K_B} = \frac{k'_A}{k'_B}$
$N = L/H$	$H = \frac{\sigma^2}{L} = L \left( \frac{W}{4t_R} \right)^2$
$N = \left( \frac{4t_R}{W} \right)^2 = \left( \frac{2.35t_R}{W_{1/2}} \right)^2$	$H = A + \frac{B}{u} + Cu = A + \frac{B}{u} + (C_s + C_m)u$
$R_s = \frac{\Delta Z}{W_A/2 + W_B/2} = \frac{2\Delta Z}{W_A + W_B}$	$R_s = \frac{\sqrt{N}}{4} \left( \frac{\alpha - 1}{\alpha} \right) \left( \frac{k'_B}{1 + k'_B} \right)$
$v = (\mu_e + \mu_{e0})E = (\mu_e + \mu_{e0})V/L$	$N = \frac{(\mu_e + \mu_{e0})V}{2D}$

### PERIODIC CHART OF THE ELEMENTS

	IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII	IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIIIA	INERT GASES	
1 <b>H</b> 1.00797															1 <b>H</b> 1.00797	2 <b>He</b> 4.0026		
3 <b>Li</b> 6.939	4 <b>Be</b> 9.0122											5 <b>B</b> 10.811	6 <b>C</b> 12.0112	7 <b>N</b> 14.0067	8 <b>O</b> 15.9994	9 <b>F</b> 18.9984	10 <b>Ne</b> 20.183	
11 <b>Na</b> 22.9898	12 <b>Mg</b> 24.312											13 <b>Al</b> 26.9815	14 <b>Si</b> 28.086	15 <b>P</b> 30.9738	16 <b>S</b> 32.064	17 <b>Cl</b> 35.453	18 <b>Ar</b> 39.948	
19 <b>K</b> 39.102	20 <b>Ca</b> 40.08	21 <b>Sc</b> 44.956	22 <b>Ti</b> 47.90	23 <b>V</b> 50.942	24 <b>Cr</b> 51.996	25 <b>Mn</b> 54.9380	26 <b>Fe</b> 55.847	27 <b>Co</b> 58.9332	28 <b>Ni</b> 58.71	29 <b>Cu</b> 63.54	30 <b>Zn</b> 65.37	31 <b>Ga</b> 69.72	32 <b>Ge</b> 72.59	33 <b>As</b> 74.9216	34 <b>Se</b> 78.96	35 <b>Br</b> 79.909	36 <b>Kr</b> 83.80	
37 <b>Rb</b> 85.47	38 <b>Sr</b> 87.62	39 <b>Y</b> 88.905	40 <b>Zr</b> 91.22	41 <b>Nb</b> 92.906	42 <b>Mo</b> 95.94	43 <b>Tc</b> (99)	44 <b>Ru</b> 101.07	45 <b>Rh</b> 102.905	46 <b>Pd</b> 106.4	47 <b>Ag</b> 107.870	48 <b>Cd</b> 112.40	49 <b>In</b> 114.82	50 <b>Sn</b> 118.69	51 <b>Sb</b> 121.75	52 <b>Te</b> 127.60	53 <b>I</b> 126.904	54 <b>Xe</b> 131.30	
55 <b>Cs</b> 132.905	56 <b>Ba</b> 137.34	*57 <b>La</b> 138.91	72 <b>Hf</b> 178.49	73 <b>Ta</b> 180.948	74 <b>W</b> 183.85	75 <b>Re</b> 186.2	76 <b>Os</b> 190.2	77 <b>Ir</b> 192.2	78 <b>Pt</b> 195.09	79 <b>Au</b> 196.967	80 <b>Hg</b> 200.59	81 <b>Tl</b> 204.37	82 <b>Pb</b> 207.19	83 <b>Bi</b> 208.980	84 <b>Po</b> (210)	85 <b>At</b> (210)	86 <b>Rn</b> (222)	
87 <b>Fr</b> (223)	88 <b>Ra</b> (226)	†89 <b>Ac</b> (227)	104 <b>Rf</b> (261)	105 <b>Db</b> (262)	106 <b>Sg</b> (266)	107 <b>Bh</b> (262)	108 <b>Hs</b> (265)	109 <b>Mt</b> (266)	110 <b>?</b> (271)	111 <b>?</b> (272)	112 <b>?</b> (277)							

Numbers in parenthesis are mass numbers of most stable or most common isotope.

Atomic weights corrected to conform to the 1963 values of the Commission on Atomic Weights.

The group designations used here are the former Chemical Abstract Service numbers.

\* Lanthanide Series

58 <b>Ce</b> 140.12	59 <b>Pr</b> 140.907	60 <b>Nd</b> 144.24	61 <b>Pm</b> (147)	62 <b>Sm</b> 150.35	63 <b>Eu</b> 151.96	64 <b>Gd</b> 157.25	65 <b>Tb</b> 158.924	66 <b>Dy</b> 162.50	67 <b>Ho</b> 164.930	68 <b>Er</b> 167.26	69 <b>Tm</b> 168.934	70 <b>Yb</b> 173.04	71 <b>Lu</b> 174.97
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† Actinide Series

90 <b>Th</b> 232.038	91 <b>Pa</b> (231)	92 <b>U</b> 238.03	93 <b>Np</b> (237)	94 <b>Pu</b> (242)	95 <b>Am</b> (243)	96 <b>Cm</b> (247)	97 <b>Bk</b> (247)	98 <b>Cf</b> (249)	99 <b>Es</b> (254)	100 <b>Fm</b> (253)	101 <b>Md</b> (256)	102 <b>No</b> (256)	103 <b>Lr</b> (257)
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