CHEM 322 **Name**___________________________________

Exam 2 **Spring 2015**

Complete the following. Clearly mark your answers. YOU MUST SHOW YOUR WORK TO RECEIVE CREDIT.

Warm-up (3 points each).

1. The ______________________________ utilizes a series of heated filaments in its detection mechanism.

2. The ______________________________ scales the retention characteristics of a compound to those of aliphatic hydrocarbons and provides a means of comparing chromatographic results between labs.

3. ______________________________ describes the movement of charged species in the electric field during an CE experiment.

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.

You must complete problem 5. (15 points)

5. Perhaps the single most important advance in separations science in the past few decades has been the mating of mass spectrometric detection with HPLC. Why has this been such a significant development? What two key challenges must have been overcome to make LC-MS a reality?

Complete <u>five</u> of the following. Be clear and concise. Clearly indicate which problem is not to be graded. (15 points each)

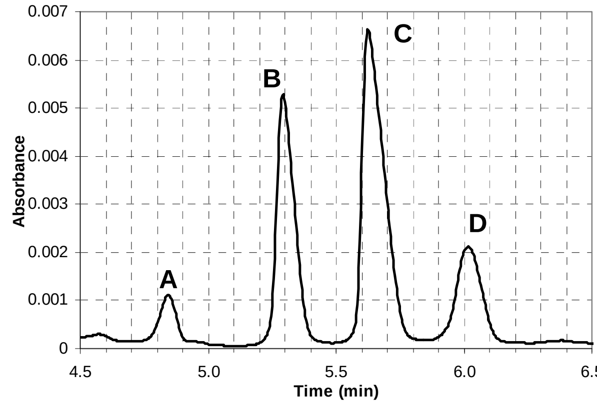
6. Briefly describe the mechanism of separation of a mixture of cations, anions, and neutrals in capillary zone electrophoresis. What parameters can be changed to optimize separation conditions in CZE?

7. 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 liquid chromatography detectors to illustrate your point.

8. Clearly describe ***one*** of the two injection methods for chromatography listed below. Include a diagram and a discussion of the key characteristics of the method in your description.
    a. Split/Splitless injection for GC
    b. An injection loop and injection valve for HPLC.

9. Consider the Van Deemter equation. Which term is likely to be the primary contributor to band broadening in capillary 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 0.85 minutes.



10. Complete the following.
    a. Calculate the number of theoretical plates for component C.
    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.

11. Your boss looks at the chromatogram and makes the following statement: "Well, it is clear to me that compound B is present at about 5 times the concentration of compound A and that compound C is caffeine since it elutes at 5.62 minutes under these conditions." Discuss the validity of this statement.

Possibly Useful Information

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

PERIODIC CHART OF THE ELEMENTS

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII	VIII	VIII	IB	IIB	IIIA	IVA	VA	VIA	VIIA	INERT GASES
1 H 1.00797																1 H 1.00797	2 He 4.0026
3 Li 6.939	4 Be 9.0122											5 B 10.811	6 C 12.0112	7 N 14.0067	8 O 15.9994	9 F 18.9984	10 Ne 20.183
11 Na 22.9898	12 Mg 24.312											13 Al 26.9815	14 Si 28.086	15 P 30.9738	16 S 32.064	17 Cl 35.453	18 Ar 39.948
19 K 39.102	20 Ca 40.08	21 Sc 44.956	22 Ti 47.90	23 V 50.942	24 Cr 51.996	25 Mn 54.9380	26 Fe 55.847	27 Co 58.9332	28 Ni 58.71	29 Cu 63.54	30 Zn 65.37	31 Ga 69.72	32 Ge 72.59	33 As 74.9216	34 Se 78.96	35 Br 79.909	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.905	40 Zr 91.22	41 Nb 92.906	42 Mo 95.94	43 Tc (99)	44 Ru 101.07	45 Rh 102.905	46 Pd 106.4	47 Ag 107.870	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.904	54 Xe 131.30
55 Cs 132.905	56 Ba 137.34	*57 La 138.91	72 Hf 178.49	73 Ta 180.948	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09	79 Au 196.967	80 Hg 200.59	81 Tl 204.37	82 Pb 207.19	83 Bi 208.980	84 Po (210)	85 At (210)	86 Rn (222)
87 Fr (223)	88 Ra (226)	†89 Ac (227)	104 Rf (261)	105 Db (262)	106 Sg (266)	107 Bh (262)	108 Hs (265)	109 Mt (266)	110 ? (271)	111 ? (272)	112 ? (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 Ce 140.12	59 Pr 140.907	60 Nd 144.24	61 Pm (147)	62 Sm 150.35	63 Eu 151.96	64 Gd 157.25	65 Tb 158.924	66 Dy 162.50	67 Ho 164.930	68 Er 167.26	69 Tm 168.934	70 Yb 173.04	71 Lu 174.97

† Actinide Series

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