

**Due at the beginning of class Monday, October 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 take 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.

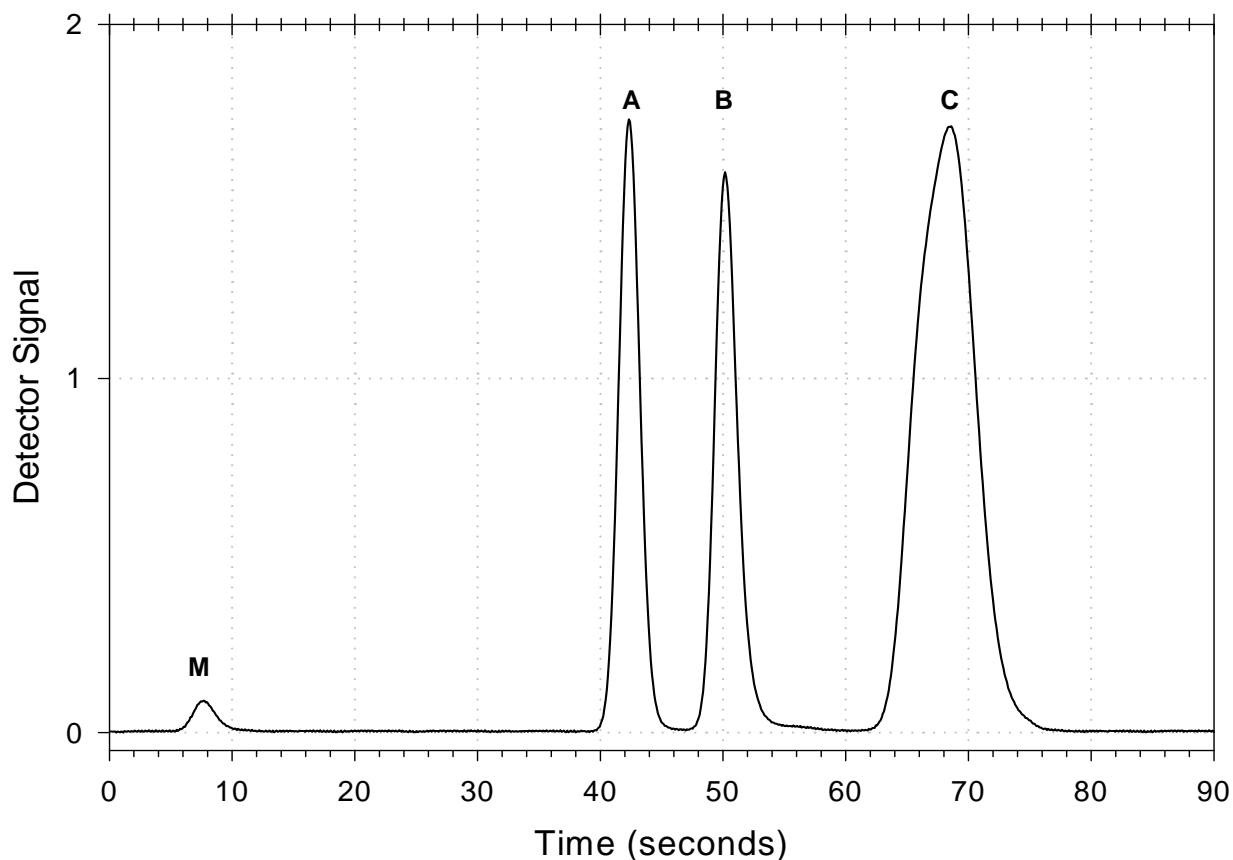
**Warm-up (2 points each)**

1. In \_\_\_\_\_, species are separated based on their ability to move in and out of the pores in the stationary phase packing material.
2. An \_\_\_\_\_ is the detector of choice for GC separations of halogenated compounds.
3. A \_\_\_\_\_ is attached to the inlet end of an HPLC column to extend its useful life.
4. In a CE experiment, \_\_\_\_\_ results in the general movement of all species toward the cathode.

**Answer in a sentence or two, or with a calculation. Complete seven of the following. Clearly indicate which problem is not to be graded. (14 points each)**

5. Why is the sample injection rate (or sample plug size) an important consideration in all separations?
6. Why is a thermal conductivity detector a much more universal GC detector than a flame ionization detector? If it is so much more universal, why use an FID at all?
7. Compare the operation of **two** of the following detectors in LC: UV absorbance, fluorescence, refractive index, electrochemical, ELSD. Consider the benefits and limitations of each detector, paying particular attention to selectivity and sensitivity.
8. In separations, we often refer to a theoretical plate as a representation of a single separation event. In general, the more events (plates) involved in the separation, the better the separation should be. Having said that, why don't we simply use very long columns to perform separations? These columns would provide large numbers of plates and good separation, right? In practice, how do we attempt to maximize the number of separation "events" that occur?
9. Describe the two general approaches to sample introduction for capillary electrophoresis. Include a description of how the sample is introduced to the capillary and any benefits or challenges of each approach.
10. Sketch a van Deemter plot for an HPLC separation using a packed column. Describe the relative contribution of all three terms in the van Deemter relationship. How does the van Deemter plot change if we considered open tubular GC instead?
11. Compare and contrast the role of the mobile phase in GC with that in LC. Include a description of the important properties of the mobile phase in each separation and its impact on the quality of a separation.

12. Clearly describe the mechanism of separation in capillary zone electrophoresis. What parameters can be changed to optimize separation conditions in CZE?
13. Answer the following questions related to the gas chromatogram below. Experimental conditions: Packed column (4 mm diameter x 2 m long), Carbowax stationary phase, 40 mL/min helium carrier gas flow rate, TCD detector, column temperature = 100°C, injector temperature = 150°C, detector temperature = 150°C. Peak M corresponds to an unretained material.



- Calculate the selectivity factor and resolution for peaks A and B.
- Calculate the number of theoretical plates for peak B.
- Based on the size of the peaks, what can you say about the relative concentrations of components A and B?
- It appears that peak C is the result of co-elution of two compounds. How would you change experimental conditions to resolve these two peaks? What effect are these changes likely to have on the separation of components A and B?

### Possibly Useful Information

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

### PERIODIC CHART OF THE ELEMENTS

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII	IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIII	IX	X	XI	XII	XIII	XIV	XV	XVI	XVII	XVIII	IX	X		
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
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† 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)
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