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 (3 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.

Complete 6 of the following. Be concise in your answers and show work for problems involving calculations. Clearly indicate which problems are not to be graded. (15 points each)

5. Why is the sample injection volume (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 the TCD is so much more universal, why use an FID at all?

7. Compare the operation of a UV absorbance detector with **<u>one</u>** of the following detectors in LC: fluorescence, refractive index, electrochemical, ELSD. Consider the benefits and limitations of each detector, paying particular attention to selectivity and sensitivity.

8. You intend to perform a separation of a mixture of the five components below using capillary electrophoresis with pressure injection and absorbance detection at 200 nm at the cathode end of the capillary. The table below describes the properties of each of the components under the conditions of the separation. Sketch an electropherogram you would expect for two experiments:

(1) capillary zone electrophoresis in a fused silica capillary **and**
(2) capillary zone electrophoresis in a capillary whose surface has been reacted with trimethylchlorosilane.

Identify each peak in your electropherograms and describe why you chose to draw them as you did.

species	concentration (ppm)	molar mass (g/mol)	molar absorptivity @ 200 nm ($M^{-1}cm^{-2}$)	charge
A	50.0	101.3	1000	+1
B	100.0	100.9	500	-1
C	50.0	100.2	2000	+2
D	100.0	99.9	1000	0
E	50.0	100.5	1000	-2

9. 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 in a chromatography experiment?

10. 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.

11. 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, FID detector, column temperature = 100°C, injector temperature = 150°C, detector temperature = 150°C. Peak M corresponds to an unretained compound.
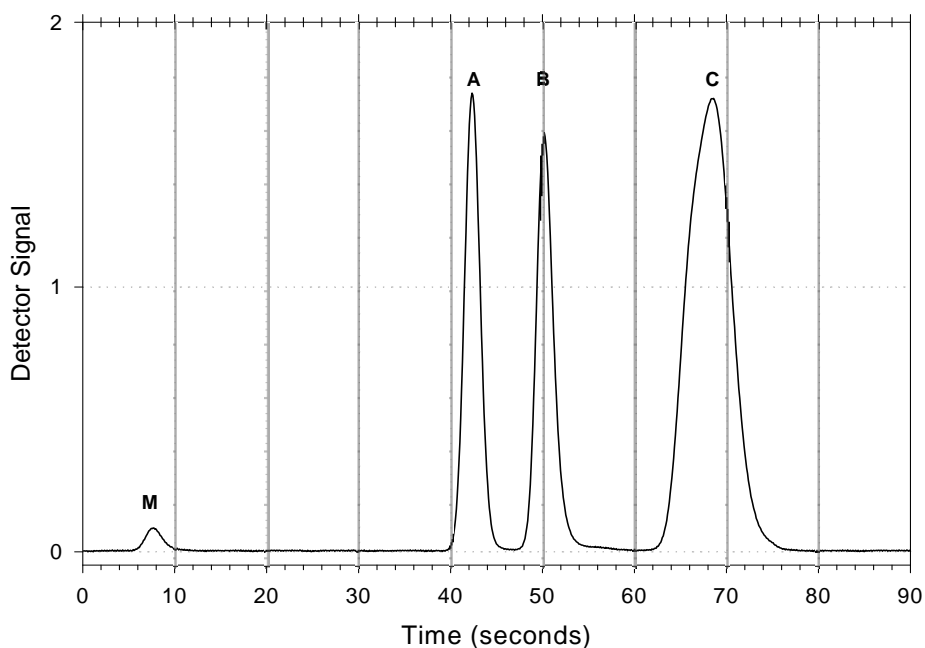
a. Calculate the selectivity factor and resolution for peaks A and B.
b. Calculate the number of theoretical plates for peak B.
c. Based on the size of the peaks, what can you say about the relative concentrations of components A and B?
d. It appears that peak C is the result of co-elution of two compounds. 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

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

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