



(<http://www.chem.usu.edu/~sbialkow/Classes/361/GC/HETP.gif>)

1. Above is a theoretical van Deemter plot for a LC column you are running. A corresponds to the eddy diffusion,  $B/v$  to molecular diffusion, and  $Cv$  to mass transfer values of your system.

- a) Describe how these processes might lead to band broadening.

You task your assistant with optimizing your system for you. Unfortunately, he/she is unfamiliar with the concepts behind the van Deemter plot, as he/she was unfortunately not a science major in college. His/her first attempt at maximizing H is chosen in accordance with point  $\alpha$ .

- b) How might this linear velocity and H affect your LC results?

His/her next attempt is at point  $\beta$

- c) How might this linear velocity and H affect your LC results?

His/her final attempt is point  $\gamma$ .

- d) In relation to the other points chosen, why is this most effective linear velocity?

After all of this, you realize the resolution obtained from this set up is not high enough for your purposes, as all of the compounds in the mixture are not capable of being resolved. Knowing that resolution is proportional to the  $(N)^{1/2}$ , your assistant suggests you increase the length of the column, perhaps by using the set up in the lab next door. However, you have something else in mind.

- e) What's wrong with your assistant's solution, and what would be the best solution for the problem? Why?
2. With Raman spectroscopy, why is fluorescence an issue? Draw a spectrum to show the effects of fluorescence and without. What are two approaches to minimizing fluorescence?

Which of the following is not an advantage of Time of Flight analyzers?

- a. They have an essentially unlimited working mass range
- b. They have high resolution
- c. They are very efficient and sensitive
- d. They are multichannel devices

The answer is b.

2. You run a column with a packing length of 20.0 cm to separate substances A and B. The retention times for A and B are 8.40 minutes and 10.0 minutes, respectively. The base peak width for A is 1.45 minutes and for B is 1.60 minutes. The solvent peak first appeared at 1.25 minutes. Calculate the average height of a theoretical plate for this column.

Answer:

First calculate the adjusted retention times:

$$tR \text{ for A} = 8.40 - 1.25 = 7.15 \text{ min}$$

$$tR \text{ for B} = 10.0 - 1.25 = 8.75 \text{ min}$$

Then calculate N:

$$N \text{ (for A)} = 16(7.15/1.45)^2 = 389.0$$

$$N \text{ (for B)} = 16(8.75/1.60)^2 = 478.5$$

$$N \text{ (average)} = 433.75$$

Then calculate H:

$$N = L/H$$

$$H = 20.0 \text{ cm}/433.75 = 4.61 \times 10^{-2}$$

An IR spectrum of CO has a band with a peak at  $1875 \text{ cm}^{-1}$ . The related peak for Carbon-14/oxygen bond would occur at what WAVENUMBER?

First, find the force constant for the CO bond, which would be  $1.42 \times 10^3 \text{ N/m}$ . Using this constant, and the different carbon mass, solve for the wavenumber (final answer of  $1795 \text{ cm}^{-1}$ ).

Multiple Choice:

When using a gas sample for Raman Spectroscopy, it is advantageous to your signal-to-noise ratio to use a \_\_\_\_\_ pathlength.

- A) shorter
- B) longer
- C) medium-length
- D) (n) Eency Weency

Calculate the number of theoretical plates on the column for species A from the following data:

Retention time of nonretained eluent: 3.1 min

Retention Time of A: 5.4 min

Base Peak width for A: .41 min

2. An electronic transition of a molecule will likely have a hypsochromic shift if...
- a. the ground state is more polar than the excited state.
  - b. the ground state is less polar than the excited state.
  - c. the excited state is stabilized by polar solvents.
  - d. the energy required for the transition is relatively low.

Explain the difference between hard and soft ionization and list one example of each method.

2. What analytically useful information can an LCMS or GCMS spectrometer provide on the analytes of interest? (specifically what info can the column give and what info can the ms provide)

3. In what ways can you optimize both the run time and S/N for GC?

You've just taken your physical chemistry test and are feeling kinda down when some freshman chemists come up to and give you some alcohol. Feeling doubtful about their chemistry skills you decided to test the alcohol with the Kovats Index System to determine if it consists of methanol, ethanol or contains some other number of carbon atoms attached to an alcohol. Testing with benzene produced a retention index of 600 while the analyte produced an index of 300. Is the stuff safe to drink?

-These freshman chemists have no idea what they are doing because lets face it, we didn't learn anything too interesting in labs the first year. They have handed you a bottle of propanol, used for fuel. Don't Drink it!!!

The capacity factor of a molecule on a column....

- A) Is a consistent measure of a compound's relative affinity for the stationary and mobile phases.
- B) Increases the time molecules attach to the sides of the column.
- C) Describes the amount of material you can load into the column.
- D) The smaller the capacity factor, the larger the separation.

The frequency of the microwave photon that is absorbed when a diatomic molecule undergoes a transition from rotational state  $J$  to rotational state  $(J + 1)$  is given by

$$\nu_{J \rightarrow J+1} = \frac{\Delta E}{h} = \frac{\hbar}{2\pi I} (J + 1),$$

where  $I = \mu R^2$  is the molecule's moment of inertia ( $R$  is the internuclear separation) and  $\hbar = h/(2\pi)$ . The rotational transition from  $J = 0$  to  $J = 1$  is known to occur at a frequency of  $1.153 \times 10^{11}$  Hz for  $^{12}\text{C}^{16}\text{O}$  and at a frequency of  $1.102 \times 10^{11}$  Hz for  $^{13}\text{C}^{16}\text{O}$ . Identify the unknown carbon isotope, assuming that the internuclear separation for  $^{13}\text{C}^{16}\text{O}$  is identical to that for  $^{12}\text{C}^{16}\text{O}$

Mass spectrometry

- a) uses the relative solubilities of analytes in a mobile and stationary phase to separate them
- b) is a spectroscopic method
- c) is a separations technique
- d) cannot be used for biological systems (ones that include water)

Answer: C

Question:

A liquid chromatographic column has a length of packing of 17.4 centimeters, a flow rate of 0.298 mL/min, a mobile phase volume of 1.28 mL, and a stationary phase volume of 0.159 mL. Using the data given below calculate the resolution and selectivity factor ( $\alpha$ ) for species B and C. Calculate the number of plates for peak A.

	Retention Time(min)	Width of Peak Base (W), min
Nonretained	2.7	0.36
A	5.2	0.97
B	13.0	1.01
C	13.8	1.13
D	21.3	1.68

Answer:

Resolution=0.748

Selectivity Factor ( $\alpha$ )=1.07

Number of Plates for Peak A = 106.3

Which of the following is not a true advantage of a TOF mass analyzer:

- a.) virtually limitless working mass range
- b.) a very small spread of initial KE
- c.) very efficient and sensitive
- d.) yields a complete mass spectrum for each ionization event
- e.) Simple!

Answer: b