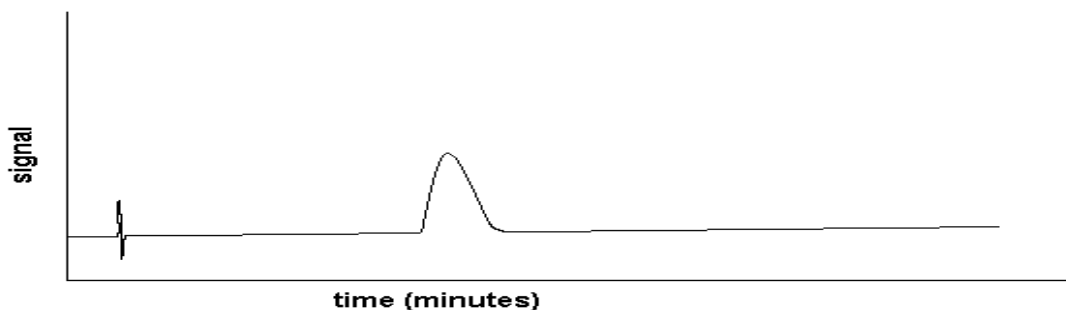


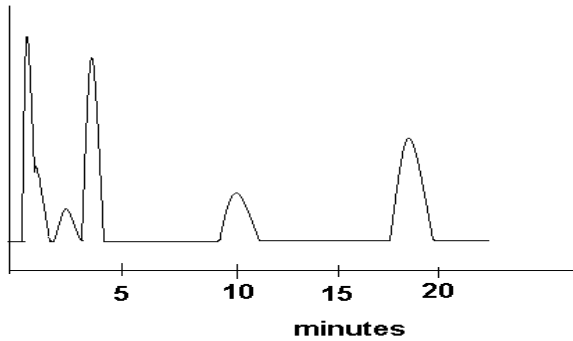
# Chromatography Problems

1. A separation on an affinity column produced the chromatogram shown below. Overtop of the chromatogram, sketch show what it would look like if a second sample with more (higher concentration) of analyte was run.

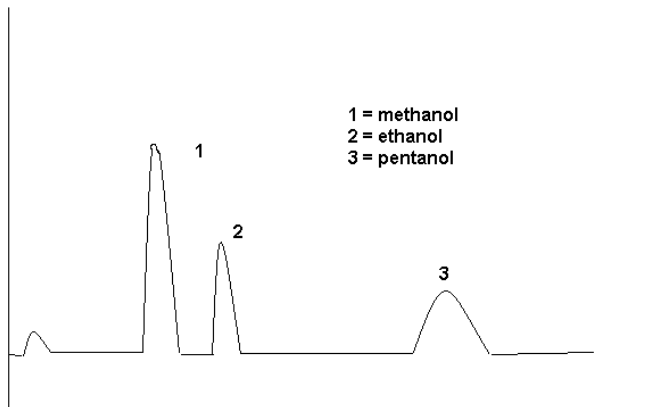


2. Are the following changes in conditions likely to increase or decrease retention time?
- faster flow rate
  - longer column
  - longer connector between column and detector
  - higher analyte concentration
  - higher temperature
  - larger sample size
  - using a less polar column while doing a reverse-phase separation
  - using a less polar mobile phase while doing a reverse-phase separation
3. In what order will the following substances elute?
- In reverse-phase liquid chromatography:
- butylamine, 2-butylene, ethylamine, ethylene
  - benzene, chlorophenol, phenol, trichlorophenol
- In gas chromatography with a polar column:
- butanol, methanol, water
- In gas chromatography with a nonpolar column:
- butanol, methanol, water
- In ion chromatography:
- bromide, fluoride, sulfide

4. For the following chromatogram, calculate retention time and adjusted retention time for each peak.



5. The following chromatogram was obtained using a C18 column and a methanol:water mobile phase.



- How would the chromatogram change if
- more ethanol were added to the sample
  - butanol was added to the sample
  - water was added to the sample
  - an unretained species was added to the sample

6. Chromatography was performed on the following solutions, where the total volume of each solution is 100.0 mL:

solution	volume 100 ppm X (mL)	volume 50 ppm Y (mL)	volume 50 ppm Z (mL)
1	5	20	10
2	10	15	20
3	15	10	5
4	20	5	15

This resulted in 4 chromatograms with the following results:

Chromatogram	peak A		peak B		peak C	
	time (min)	Area (x 10 <sup>4</sup> )	time (min)	Area (x 10 <sup>5</sup> )	time (min)	Area (x 10 <sup>4</sup> )
1	6.44	0.68	8.00	4.10	9.72	7.99
2	6.54	1.98	7.96	8.30	9.68	7.63
3	6.34	2.18	8.03	2.53	9.66	7.27
4	6.49	3.02	8.12	6.29	9.58	6.93

- Which peak corresponds to which analyte?
- Make a calibration curve for each analyte
- A unknown solution was analyzed with the same method. Based on the results below, what is the composition of the sample?

retention time (min)	peak area
6.59	33419
8.05	554193
9.68	76416

7. A calibration curve obtained from a chromatographic experiment resulting in a line with the equation

$$\text{Peak area} = (139997 \pm 5949 \text{ ppm}^{-1})[\text{analyte}] + 913 \pm 554$$

- If sample had a resulted in a peak area of 568336, what is the concentration of analyte, with error?
- In another experiment, 10.00  $\mu\text{L}$  of sample was diluted with water to make a 1.00 mL solution. The resulting analyte peak had an area of 2765422. What is the concentration of analyte in the original solution?
- In another experiment, 1.00 L of sample was processed through a solid phase extraction cartridge. The analyte stayed on the cartridge. Then 5.00 mL of methanol was used to elute the analyte from the cartridge. When the eluted solution was chromatographically separated, the analyte peak area was 6729661. What was the concentration of analyte in the original sample?

8. *Standard Addition.* The chromatographic analysis of Sample A resulted in a peak at 8.95 minutes and a peak area of 24237396. When 10.0 $\mu$ L of Sample A was mixed with 5.00  $\mu$ L of 20.0 ppm standard, chromatographic analysis of the resulting solution resulted in a peak with a retention time of 9.12 minutes and a peak area of 121220801. What is the concentration of analyte in the original sample?

9. *Standard Addition.* A chromatographic analysis of Sample B produced the following results:

Time	Peak area	% area	Height	% height
5.726	764499	39.41	62503	35.89
7.207	241332	12.44	46947	26.96
10.735	934148	48.15	64691	37.15

a. What is a likely reason that the percent areas and percent heights are different?

b. When 4.34 g of Sample B are mixed with 3.98 g of pure analyte and the mixture analyzed, the following results were obtained. What is the % analyte in the mixture?

Time	Peak area	% area	Height	% height
5.739	528045	29.50	43954	28.90
7.554	162697	9.089	33037	21.72
10.686	1099110	61.41	75114	49.38

10. *Graphical Standard Addition.* The following solutions were made and analyzed with ion chromatography--the results included in the table. Create a standard addition graph and determine the concentration of fluoride in the original sample, with error.

Note: all solutions were made with a total volume of 10.00 mL and a fluoride standard with a concentration of 35.21 ppm.

Vol sample (mL)	Volume std (mL)	Retention time (min)	Peak area
5.00	0.00	2.50	135235
5.00	0.50	2.80	396313
5.00	1.00	2.15	764095
5.00	1.50	2.46	1050673
5.00	2.00	2.41	1523699

11. *Graphical Internal Standard.* The following solutions were made and analyzed with ion chromatography using 81.7 ppm fluoride ion as an internal standard and 64.4 ppm sulfate ion as an analytical standard. What is the concentration of sulfate in the sample, with error?

Total volume of solution = 10.00 mL; last row is sample not standard

<b>Vol SO<sub>4</sub><sup>2-</sup> (mL)</b>	<b>Vol F<sup>-</sup> (mL)</b>	<b>t<sub>R</sub> peak 1 (min)</b>	<b>Area peak 1</b>	<b>t<sub>R</sub> peak 2 (min)</b>	<b>Area peak 2</b>
1.00	3.00	3.09	62817	18.30	96234
2.00	3.00	2.93	68075	18.37	204377
3.00	3.00	3.20	61262	18.54	274731
4.00	3.00	3.03	67054	18.47	402935
<b>5.00 (sample)</b>	<b>3.00</b>	<b>2.97</b>	<b>63281</b>	<b>18.39</b>	<b>278748</b>

[Solutions](#)