

**CHEM 230**  
**Fall, 2014**  
**Special Topics Additional Problems**  
**For Your Own Benefit – not to turn in**

1. **[Related to FFF]** List one type of Field Flow Fractionation and the separation field used in that type:
2. **[Related to FFF]** Relative to the direction of the flow in FFF, in which direction is the separation field pointed
  - a) with the flow
  - b) against the flow
  - c) at a right angle to the flow
  - d) it changes depending on movement
3. **[Related to FFF]** In FFF, analytes that are pushed more strongly toward a wall elute \_\_\_\_\_ (earlier or later).
4. **[Related to SFE]** A scientist testing extraction of a polar compound from tree bark through Soxhlet extraction using water notices that longer extraction periods gives lower yields. He suspects that the cause is compound degradation.
5. **[Related to SFE]** What properties of supercritical fluids make extractions of solids more time efficient than standard methods?
6. **[Related to SFE]** In the analysis of natural products in solid plant parts, it is desired to remove compounds of interest and then to obtain them at relatively high concentrations. Which step can be eliminated or minimized by switching from conventional extractions to SFE?
7. **[Related to Chiral Separations]** List one advantage and one disadvantage for separating enantiomers through derivation with chiral reagents and achiral (regular type) chromatography.  
Advantage = \_\_\_\_\_ Disadvantage = \_\_\_\_\_
8. **[Related to Zirconia in HPLC]** Running a packed HPLC column at higher temperatures, while not common, generally results in a lower H value. In considering sources of broadening from the van Deemter equation, the main reason for a decrease in H is:
  - a) shrinking of particle size resulting in a smaller A term
  - b) an increase in diffusion resulting in a smaller B term
  - c) an increase in molecular diffusion resulting in faster mass transport and a smaller C term
  - d) all of the above
9. **[Related to Zirconia in HPLC]** What type of zirconia based HPLC column would be a good replacement of silica based C18 columns (e.g. for the analysis of bases at high pH)?
10. **[Related to 2D HPLC]** In what way does the effectiveness of 2D HPLC depend on both the two methods of separation (done in the two columns) and on the types of samples?

11. **[Related to 2D HPLC]** 2D HPLC is a fairly popular method for separating proteins in biological samples. List two possible orthogonal separation methods that could be used for proteins.

12. **[Related to Zwitterion stationary phase]** Why can conductivity detection be used with zwitterionic stationary phase (and no suppressor) with better success than with ion exchange (also without a suppressor)?

Longer Answer Questions:

1. **[Related to Chiral Separations]** A researcher is carrying out an asymmetric synthesis to predominantly synthesize one enantiomer. She then is analyzing the products (both enantiomers) to determine how successful the synthesis is using GC with a chiral column. If two product peaks are observed to elute, describe how she can determine the enantiomeric ratio. Are quantitative or qualitative standards needed?

2. **[Related to Zwitterion stationary phase, ion-pairing and CE topics]**. List two special topic methods that allow analysis of neutrals, cations, and anions in a single run? In this method, what determines the elution order?

3. **[Related to 2D HPLC]** A set of peptides is separated by 2D HPLC using a SEC first column and a RP second column. Assuming the peptides can vary by 1) fragment length and 2) % of non-polar amino acids (e.g. phenylalanine, leucine and valine). Indicate where long, polar peptides and short, non-polar peptides would come out in a 2D chromatogram.

