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

1. [**Related to CE topic not in Exam 4**] In capillary electrophoresis, hydrodynamic injection means:
a) using an injection valve before the capillary
b) using pressure to force the sample into the capillary
c) using a voltage to migrate sample into the capillary
d) using a change in height to force the sample into the capillary

2. [**Related to CE topic not in Exam 4**] The main reason narrow capillaries are used in capillary electrophoresis is:
a) to limit dispersion when analytes partition to and from the capillary walls
b) thick walls are needed to avoid current leaks through the walls
c) to limit "joule" heating in the capillaries at high voltages
d) to keep the multiple flow term of the van Deemter equation small

3. [**Related to CE topic not in Exam 4**] Which term of the van Deemter equation contributes the most to band broadening in capillary zone electrophoresis?
a) the A term b) the B term c) the C term d) the u term

4. [**Related to CE topic not in Exam 4 and MEKC**] List a capillary electrophresis method that can be used for uncharged molecules.
Method = _____

5. [**Related to MEKC**] Micellar electrokinetic chromatography (MEKC) is performed with sodium dodecyl sulfate (SDS). The SDS concentration must be greater than the critical micelle concentration or:
a) No micelles form b) inverted micelles (hydrophobic exteriors) form
c) A reversal of polarity occurs on the fused silica surface (from negative to positive charge)
d) There are insufficient charge carriers for electrophoretic mobility

6. [**Related to SMB Chromatography**] How is the moving bed simulated in simulated moving bed chromatography?
a) by computer data reduction methods
b) by switching valves going to, between, or from an array of connected columns
c) by moving the column walls instead of the bed
d) by using a high potential to stimulate migration of the bed toward the cathode

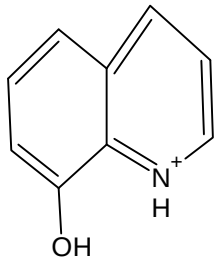
7. [**Related to SMB Chromatography**] Why would SMB not be useful for isolation of natural products from plant extracts?

8. [**Related to SPME-HPLC**] For what type of compounds can SPME-HPLC be used but not SPME-GC?

9. [**Related to SPME-HPLC**] List two factors that influence the time it takes for analytes to diffuse off of an SPME fiber in an SPME type injection. factor 1 = _____ factor 2 = _____

Longer Answer Questions:

1. [**Related to CE topic not in Exam 4**] Given hydroxyquinoline's pK_a values (pK_{a1} = 4.94 for NH^+ proton and pK_{a1} = 9.8 for OH proton – see structure below) can be separated from neutral molecules by capillary electrophoresis using a silica capillary. Separations are attempted at pH = 4, 7.5, and 11. Indicate whether hydroxyquinoline will be well separated from neutral compounds at those pH values and the relative migration speed (fastest to slowest).



2. [**Related to MEKC**] Discuss how the answers to question 1) would be changed if the separations were performed at the same pH but using MEKC.

3. [**Related to SMB Chromatography**] A large-scale synthesis produces two diastereomers and also has left over excess reagents.
a) If the diastereomer of interest is less polar than the other diastereomer and the other reagents, could SMB (simulated moving bed) chromatography be used to collect at good purity the diastereomer of interest in a single run? Explain your answer.
b) Could the other diastereomer also be collected in good purity?

4. [**Related to ion-pairing HPLC**] Alkyl amines are bases with conjugate acid pK of around 10. Why can these be separated easily using C18 columns and ion-pairing HPLC while it is very difficult to do so by reversed-phase HPLC.

5. [**Related to ion-pairing HPLC**] Glycine, $NH_3^+CH_2CO_2^-$, shown in its neutral pH zwitterionic form, has a pK_{a1} for $-CO_2H$ acid of 2.35 and a pK_{a2} of 9.78 for its $-NH_3^+$ acid. At what pH and with what type of counter ion could glycine be retained through ion-pairing HPLC on a reversed phase (C18) column? Can it be separated at any pH using standard reversed phase HPLC?