CHEM 133 Exam 3
May 12, 2015

Name _____

Equations and constants that you may find useful:
0ºC = 273 K; Planck's constant h = 6.626 x 10^{-34} J·s
Boltzman Constant = k = 1.38 x 10^{-23} J/K
Pascal's triangle: 1
                    1   1
                 1   2   1
              1   3   3   1
           1   4   6   4   1
        1   5   10   10   5   1

SHORT ANSWER SECTION: (Each question worth 4 points)
1. What main spectrometer component is needed in atomic absorption spectrometers but not in atomic emission spectrometers? Component = _____

2. NMR is a relatively insensitive technique. Signal averaging by repeated radio frequency pulsing can be used to improve the signal to noise ratio. What needs to be considered in determining the pulsing rate?
a) the spin-lattice relaxation time   b) splitting coupling constants
c) the ppm of the absorption   d) all of the above

3. ^{14}N is an I = 1 nucleus. How many spin states does it have?
a) 1   b) 2   c) 3   d) 4

4. List a mass spectrometric ionization method that can be applied to solid samples. Method = _____ (give name or acronym)

5. What generally occurs to ions in between two mass spectrometers in MS-MS?
a) removal of excess ion energy   b) addition of an extra ion
c) isotope exchange   d) collision with reagent gas to induce fragmentation

6. List one type of mass analyzer (component of a mass spectrometer). _____

7. Triglycerides in fats and oils can be analyzed by HPLC using reversed phase columns. However, they typically are only a little more polar than the stationary phase but much less polar than solvents commonly used in reversed phase HPLC (e.g. actonitrile and water). What is a potential problem in their analysis?
a) very small retention factors
b) excessively long retention times
c) interference from other compounds that elute early
d) the need for a solvent more polar than water to elute compounds

SECTION II. PROBLEM SECTION. Show work – use the back side if needed

1. In flame atomic absorption spectroscopy a number of processes can decrease the atomization efficiency. (8 pts)
a) Give an example of one process that decreases atomization efficiency

b) Give a method to reduce the effect of this process

bonus) List a second example and method to reduce its effect (3 pts)

2. Give a block diagram for an ICP-MS instrument showing the ionization source, the way to separate ions, and the way to detection ions (6 pts)

3. In a 4.70 Tesla field, the magnetogyric ratios (γ) for ^{31}P is 1.08×10^8 T^{-1} s^{-1}.
Equations and constants you might need: ν (frequency) $= (\gamma/2\pi)H$ (H = magnetic field strength)
a) At what frequency (in MHz) does ^{31}P absorb light? (5 ppm)

b) Calculate the ratio of ^{31}P in the excited state to ^{31}P in the ground state at 298 K to 6 significant figures. $g^* = g_o$. (8 pts)

c) If the peaks from two closest (spectrally) phosphorous atoms in ATP (see structure below) are located 4.5 ppm apart, what is their difference in Hz? (5 ppm)

HO—P(=O)(O⁻)—O—P(=O)(O⁻)—O—P(=O)(O⁻)—O— Rest of adenosine

a   b   c
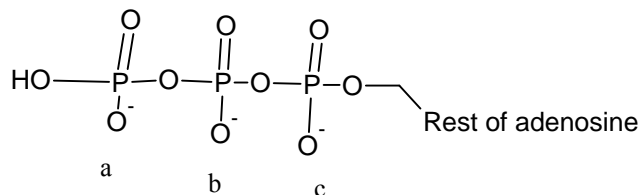
4. Sketch what the ^{1}H NMR spectrum of $Cl(CH_2)_3Cl$ looks like. You don't need to give values for the ppm scale, but make sure to give 1) the number of types of proton peaks, 2) the position of the peaks relative to other peaks and to tetramethylsilane (TMS, the standard with $\delta = 0$ ppm), 3) the relative intensity of the peaks, and 4) the splitting pattern for each peak (e.g. label as doublet, triplet, etc. or "complex" if a multiplet of multiplets. You may want to explain your answers. (12 pts)

response

Chemical shift (ppm)
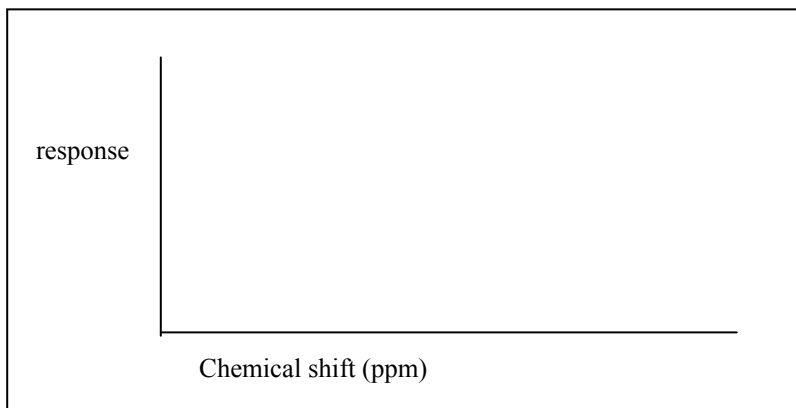
5. A halogenated hydrocarbon is analyzed by mass spectrometry with a parent ion observed with a mass to charge ratio of 238. Fragments indicate that it has at least one Br atom and one Cl atom. Given the ratio of M + 2/M, M + 4/M and M + 6/M (below), **determine the number of Br and Cl atoms** assuming no other isotopes contribute significantly to these peaks. There is no observable peak at the M + 8/M location. The relative abundances of these isotopes are $^{37}Cl/^{35}Cl = 32/100$ and $^{81}Br/^{79}Br = 97/100$. Show your work. (12 pts)

Observed ratios	M + 2/M	M + 4/M	M + 6/M
	160.2/100	72.1/100	9.9/100

6. Using the HPLC chromatograms for separating components A and B provided below and the fact that the separation occurring on column 1 (lower chromatogram) was a reversed phase separation, answer the following questions: (16 pts)
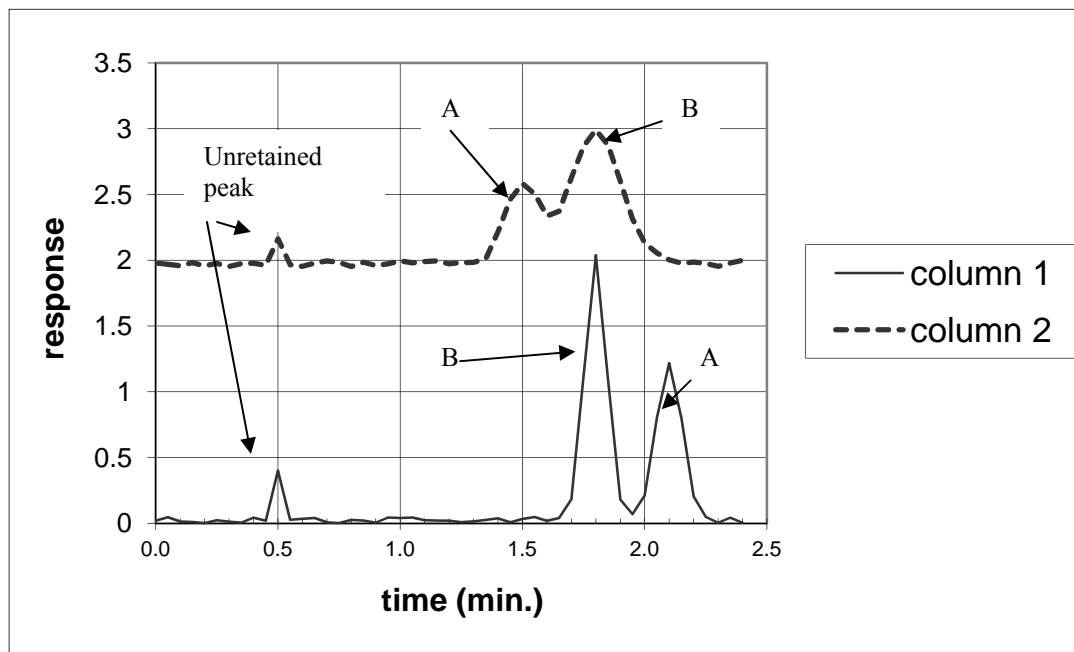
Determine:
a) Which compound is more polar: A or B? Explain your answer.

b) Is the separation occurring on column 2 most-likely reversed-phase or normal phase? You can assume that the main factor affecting separation for both columns is polarity. Explain your answer.

c) The retention factor (k) for A for the separation on column 1.

d) Whether one would want to increase the percent hexane or 2-propanol for the column 2 separation if it is desired to retain both components to increase separation.