

CHEM 133 Final Exam **Solutions**  
May 21, 2015

Constants that you may find useful:

$0^{\circ}\text{C} = 273\text{ K}$     $h = \text{Planck's constant} = 6.626 \times 10^{-34}\text{ J}\cdot\text{s}$     $c = \text{speed of light} = 3.00 \times 10^8\text{ m/s}$

Boltzman Constant =  $k = 1.38 \times 10^{-23}\text{ J/K}$     $e = \text{elementary charge} = 1.60 \times 10^{-19}\text{ coulombs}$

$F = \text{Faraday's constant} = 96,500\text{ C/mol e}$

SHORT ANSWER SECTION: (Each question worth 4 points)

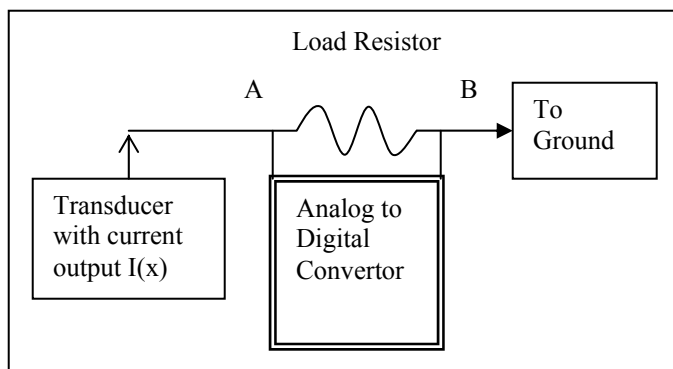
1. Chemist A is investigating the presence of highly toxic mold compounds in processed peanuts, where very small concentrations of mold compounds can have significant effects. Chemist B is measuring the types of oils in the peanuts. Oils are a major fraction of peanut mass. Compared to Chemist B, Chemist A needs excellent:  
a) accuracy      b) precision      **c) sensitivity**      d) sample throughput
2. Give the name of a transducer capable of detecting light. Transducer = photodiode, photoconductivity cell, photomultiplier, photocell.
3. What type of noise is associated with slow drifts in signal response and can be reduced by modulating part of an instrument (to perform measurements at higher frequency)?  
**a) 1/f noise**      b) shot noise      c) thermal noise      d) Johnson noise
4. In a galvanic cell:  
a) oxidation occurs at the anode which is positively charged  
b) reduction occurs at the anode which is positively charged  
c) reduction occurs at the anode which is negatively charged  
**d) oxidation occurs at the anode which is negatively charged**
5. What type of light produces transitions in vibrational motions in typical molecules?  
a) x-ray      b) UV      **c) infrared**      d) radio waves
6. Which of the following spectrometers does not need a monochromator or polychromator?  
**a) Fourier transform infrared spectrometer**      b) flame atomic absorption spectrometer  
c) fluorescence spectrometer      d) UV-visible spectrophotometer
7. Give one source of a loss of atomization efficiency that can occur in flame atomic absorption spectroscopy. Source = low volatility salts, ionization, oxide/hydroxide formation
8. One inherent disadvantage of NMR spectroscopy is that it is:  
a) low in resolution      **b) low in sensitivity**  
c) low in precision      d) not at all quantitative
9. Give the name or describe the functioning of an ion detector that can be used in mass spectrometry. Name or description: Faraday cup, electron multiplier, MCP (w/TOF), induction coil (w/FTICR)

10. The primary reason going to smaller particle sizes in HPLC results in a decrease in H (plate height) is because:

- a) the velocity will be slowed
- b) the pressure will be greater
- c) mass transport between the mobile and stationary phases is faster**
- d) the variations in paths around the smaller particles is greater

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

1. In the circuit below, the current from a transducer is being measured indirectly by feeding the voltage drop across the load resistor into an analog to digital convertor. The resistance of the load resistor is 121 kΩ, and the analog to digital convertor has 10 bits and can read from 0 to 5.0 V. Assume the transducer current is not affected by the resistance from A to B in any of the problems below.



a) If the digital display of the analog to digital convertor is 00100 11011, what is this in **binarydecimal**? (4 pts)

$$decimal = 1 + 2 + 4 \cdot 0 + 8 + 16 + 32 \cdot 0 + 64 \cdot 0 + 128 + 256 \cdot 0 + 512 \cdot 0 = 155$$

b) What voltage corresponds to the reading in a)? (5 pts)

$$0 + 155 \cdot (5 \text{ V} - 0\text{V}) / 2^{10} = \mathbf{0.757 \text{ V}}$$

c) What current does this correspond to? (4 pts)

$$I = V/R = (0.757 \text{ V} / 121,000 \Omega) (10^6 \mu\text{A/A}) = \mathbf{6.3 \mu\text{A}}$$

bonus – with problem #1) What is the maximum measurable current? (3 pts)

$$5\text{V} = \text{max voltage in so } I = 5 / 121,000 \Omega = \mathbf{41 \mu\text{A}}$$

2. A mass spectrometer is used to analyze a sample and in a 2.0 s run, a peak at 232 amu is observed. The analyst is interested in determining if sulfur is present, which will result in a 234 peak at 4.5% of the height of the 232 peak. If the signal to noise ratio on the 232 amu peak in the 2.0 s run was 75, how long of a run is needed to get a signal to noise ratio of 10.0 in the 234 peak? Assume the noise level is the same for each peak and that the time is directly proportional to the number of scans performed. (12 pts)

*For a 2.0 s run,  $S/N(232 \text{ peak}) = 75$ . Expected  $S/N(t = 2.0 \text{ s}, 234 \text{ peak}) = (4.5/100)(75) = 3.38$   
 Time is proportional to the number of scans and  $(S/N) = kn^{1/2}$  ( $n = \# \text{ scans}$ ) or  
 $(S/N) = kt^{1/2}$  ( $t = \text{time}$ ) or  $10/3.38 = (t/2.0 \text{ s})^{1/2}$  or  $t = (10/3.38)^2(2.0 \text{ s}) = \mathbf{17.6 \text{ s}}$*

3. In X-ray emission spectrometers, detectors give a peak current that is proportional to the energy of the photon detected.

a) If an X-ray of wavelength 1.32 nm gives a peak current of 7.2  $\mu\text{A}$ , calculate the peak current expected for an X-ray with a wavelength of 0.751 nm. (6 pts)

$$E(\text{photon}) = hc/\lambda \text{ and } I(\text{current}) = kE(\text{photon})$$

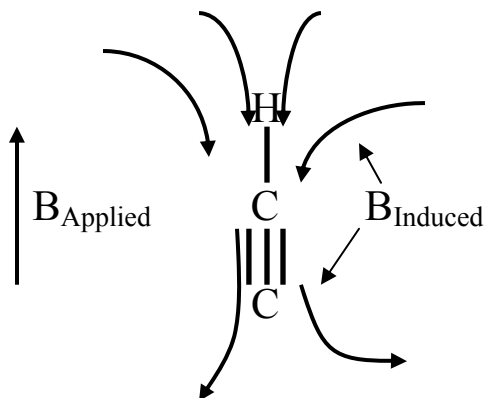
$$(hc/\lambda_1)/(hc/\lambda_2) = I_1/I_2 \text{ or } I_2/I_1 = \lambda_1/\lambda_2$$

$$I_2 = (7.2 \mu\text{A})(1.32/0.751) = \mathbf{13 \mu\text{A}}$$

b) By having a detector that gives peaks proportional to current, what part of a “typical” spectrometer is not needed with this type of detector. (4 pts)

*A monochromator would not be needed because the detector discriminates the wavelength*

4. Alkyne protons (see figure below) are influenced by electron circulation in the two pi bonds resulting in an induced magnetic field as shown below. Explain how you would expect this to affect the chemical shift (e.g. upfield or downfield) of the  $\text{HC}\equiv$  protons (compared to protons with the same electron density from the HC bond). (5 pts)



*In this case the induced magnetic field is opposed to the applied magnetic field. This effect is the same as if shielding occurs, so a **shift upfield would be expected**.*

5. Two columns, a non-polar DB-1 column and a moderately polar DB-225 column, were tested to separate methyl stearate – MS or  $\text{CH}_3\text{O}_2\text{C}(\text{CH}_2)_{16}\text{CH}_3$  from methyl oleate – MO or  $\text{CH}_3\text{O}_2\text{C}(\text{CH}_2)_7\text{CH}=\text{CH}(\text{CH}_2)_7\text{CH}_3$  (cis form) in biodiesel samples using GC in two trial chromatograms with retention times and peak widths listed below. The columns had similar dimensions (length, diameter and film thickness) and were run using the same temperature program.

Peak	DB-1 Retention Time (min)	DB-1 Peak width at base (min)	DB-225 Retention Time (min)	DB-225 Peak width at base (min)
CH <sub>4</sub> (unretained)	1.71	-	1.83	-
MO	8.53	0.185	10.31	0.213
MS	8.79	0.190	8.84	0.186

a) Determine the retention factor ( $k$ ) for MO on the DB-1 column. (4 pts)

$$k = (8.53 - 1.71)/1.71 = \mathbf{3.99}$$

b) Determine the plate number ( $N$ ) for the DB-225 column using MO. (4 pts)

$$\text{DB-225 } N = (10.31/0.213)^2 = \mathbf{37,500}$$

c) Based on their separations, which compound (MS or MO) is more polar? Explain your answer. (4 pts)

*MO – it is more retained on the more polar column.*

d) **Select a column**, which when optimized for a fast separation, should allow better analysis of biodiesel assuming MO and MS are the main biodiesel constituents of interest. Assume that both columns could be run hotter or cooler than the test conditions used above. Explain your choice (you may want to perform calculations to justify your selection). Also, **indicate what change in conditions** you would use to optimize the separation. (8 pts)

*DB-225 is better because resolution is better.  $R_s = 7.4$  (vs. 1.38 on the DB-1). The DB-225 could probably be run hotter to decrease the total separation time, while the DB-1 would need to run cooler to meet baseline resolution conditions.*

*Using the optimization equation, to run DB-225 with  $R_s = 1.5$  would require a  $k_2$  of 0.2 (estimated) or total time of 2.4 min.*