Chem 231 Quiz Number 1
Solutions

A. Short Answer/Multiple Choice Section (2 points each)
1. Which of the following safety items should NOT be worn when working on an instrument and using the keyboard
a) gloves b) goggles c) labcoat d) pocketprotector
[Contaminated gloves can contaminate the keyboard and then gloveless users]

2. An advantage of saving chromatograms that were processed with chromatographic software (e.g. ChemStation) rather than creating a plot from an exported text file include:
a) The chromatograms always look better
b) Data smoothing can only be done with chromatographic software
c) Integrations is usually better with chromatographic software and shown on plots
d) All of the above
[Homework did data smoothing using Excel; some software plots are poor quality]

3. A student is planning on running the Agilent HPLC. She has turned on the software, and used the software to turn on the pump, the column heater and the UV detector. On the Run & Control View mode, the pump and column heater are colored green and the UV detector is colored yellow. This means:
a) The system is ready for injections
b) The pump and column heater are running fine, but the UV detector is not yet ready
c) The pump and column heater are running fine, but the UV detector has a fault
d) The system has a problem and will need trouble-shooting and fixing before any sample can be run.
[yellow means getting ready]

4. When gases are sampled for subsequent injection into GC using sorbent traps, it is common to reverse the flow for injection into GC. Why is the flow reversed for desorption?
Reason = <u>This is to introduce gases into the GC in a narrower band. Compounds that are strongly retained on the sorbent have only a short distance to travel if flow is reversed.</u>

5. List two methods for solvent removal from samples. Method 1 = <u>rotory evaporator</u>
Method 2 = <u>Dry under nitrogen</u> Method 3 = <u>freeze dryer</u>

B. Calculations/Longer Answer. (4 pts)
A student is working to develop a method to separate three phthalate esters (see **Figure 1** below). He first performs the separation using a gradient separation on a 150 x 4.6 mm C18 column with the gradient program given in **Table 1**. The observed chromatogram is shown in **Figure 2**. The first peak at 2.4 minutes is also seen when a blank sample is injected (same solvent, but no phthalate esters). Describe how the separation should be improved by changing the eluent composition/gradient, and what effect would be expected.
[See solution at the bottom of next page]

Figure 1. Methyl, Ethyl, and 1-propyl phthalate esters.

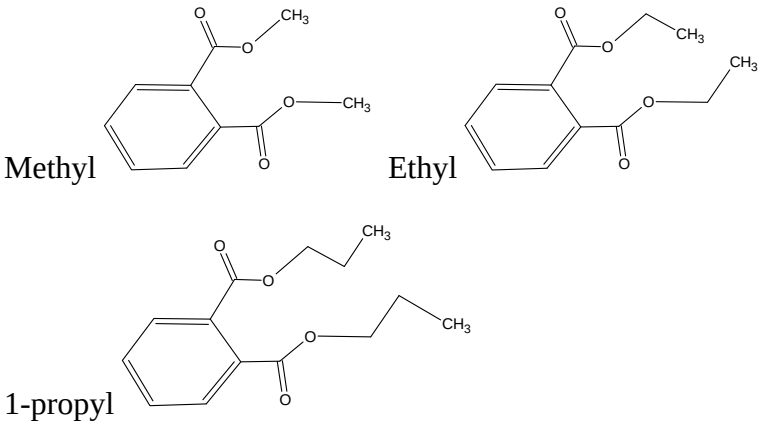
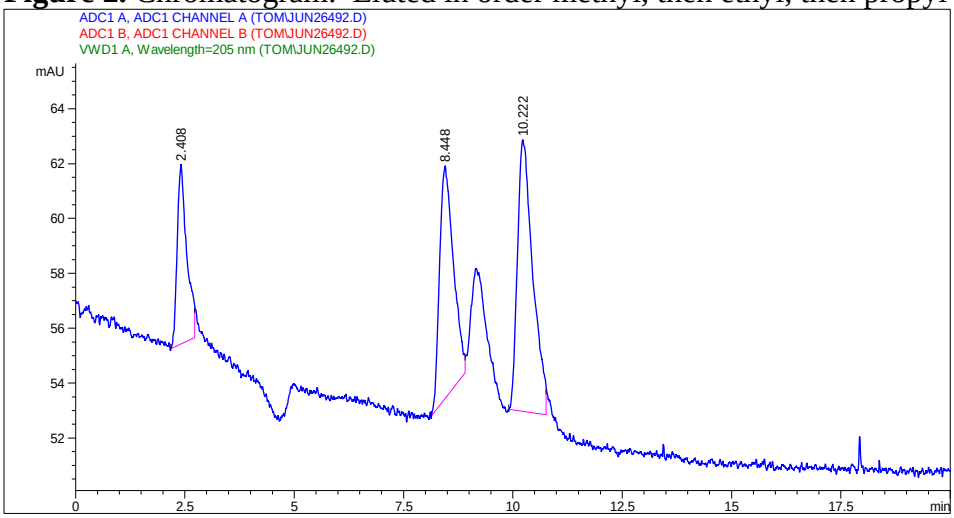


Table 1. Gradient Elution with water/acetonitrile eluent

Time (min.)	% acetonitrile
0	60
12	95
15	95
16	60

Figure 2. Chromatogram. Eluted in order methyl, then ethyl, then propyl



The main problem appears to be poor separation between the methyl and ethyl esters. This is normally "fixed" by increasing retention with an increased retention factor in better separation. This can be done by starting with a lower % acetonitrile. The peaks also tail which could indicate column overloading (although decreasing the injection volume could result in poorer signal to noise ratios).