
Name

CHM 410 / ENV 1410 Analytical Environmental Chemistry

~ Midterm Exam ~

Fall, 2008

53 points over 6 questions on 6 pages.

No external devices (ie. calculators) are permitted.

Marks for the test are allocated on a 'mark-a-minute' basis, so please judge the amount of detail required based on the number of marks allocated for each question.

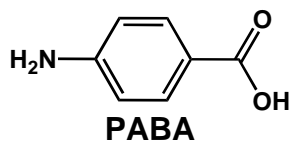
Solvent Properties Table

Solvent	Viscosity at 25 °C (cP)	UV Cutoff (nm)	Boiling Point (°C)	Dielectric Constant	% H ₂ O solubility
Methanol	0.54	205	65	32.7	miscible
Acetone	0.30	330	56	20.7	miscible
Water	0.89	190	100	80	miscible
Acetonitrile	0.34	190	82	37.5	miscible

“Education has failed in a very serious way to convey the most important lesson science can teach: skepticism.”

~ David Suzuki

Question 1. Until recently *para*-aminobenzoic acid (PABA) was a common component of commercial sunscreens.



(a) Draw the structure of PABA under the following aqueous conditions:

Water at pH 2 (**1 point**)

Water at pH 7 (**1 point**)

Water at pH 10 with an organic ion pair (**2 points**)

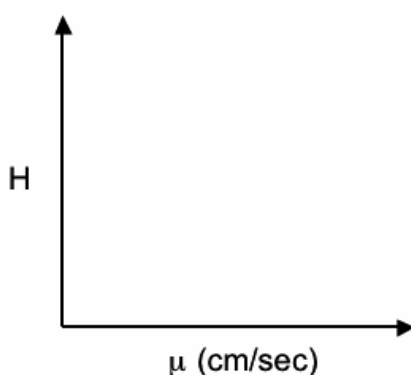
(b) You are interested in extracting PABA from surface water samples. State an extraction technique that would be appropriate for this analyte and matrix. (**2 points**)

Question 2. As a solvent acetone has low viscosity, is miscible with water and has a low boiling point. Because of these properties it has been suggested as an appropriate solvent for LC-MS applications. Would acetone be appropriate for LC-UV applications? Briefly explain your answer. (**3 points**)

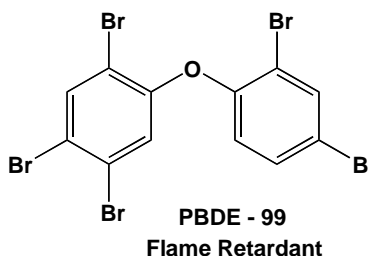
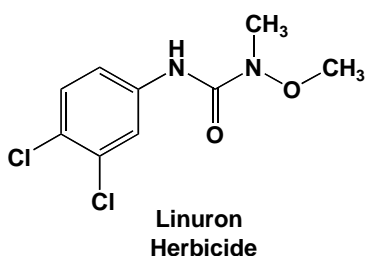
Question 3. The C-term of the Van Deemter equation describes the efficiency of mass transfer in a chromatography column.

(a) Define the C-term for a packed column (LC)? (2 points)

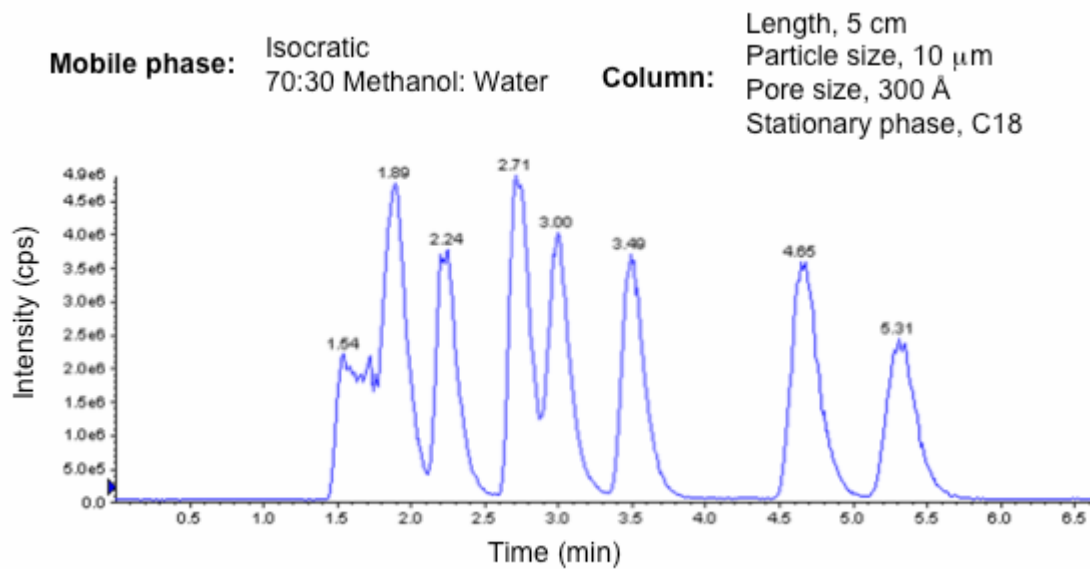
(b) Using the solvent properties table draw the C-term of the Van Deemter equation on the axis provided for water, methanol and acetone. (Assume all column parameters remain constant) (3 points)



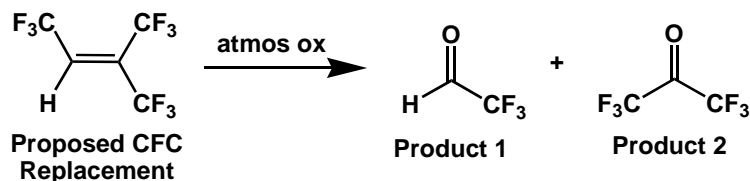
Question 4. For the two environmental contaminants shown below, which would be more appropriate for ELISA analysis? Why? For the chosen compound draw an appropriate immunogen (protein-bound complex to be administered to an animal) and antigen (enzyme-linked complex used in ELISA). (5 points)



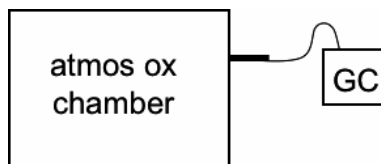
Question 5. You have joined the Mabury Lab as a summer student and your first task is to separate a mixture of fluorinated acids by LC-MS. On your first try you produce the chromatogram shown below. You know your resolution is not good enough. State **three** changes to the column or mobile phase that you would investigate to improve your chromatography. Explain why you would expect these changes to improve your chromatography. (9 points)



Question 6. A proposed CFC replacement is shown below on the left. This compound will not deplete stratospheric ozone or warm the climate as it can easily undergo atmospheric oxidation by addition to the double bond. Two potential degradation products of this reaction are shown below.



- (a) An atmospheric chemist, Lucy, has contacted you for analytical support. Lucy has a chamber in which she can simulate atmospheric oxidation reactions, and has recently had a grant approved to buy a GC to attach directly to her chamber (as shown below). She would like to perform simulated atmospheric oxidation reactions on the proposed CFC replacement shown above, and during those experiments analyze for the starting material as well as potential degradation products, Product 1 and Product 2. Unfortunately, she does not have enough money to buy a GC-MS and has asked your advice on an alternate GC detector that would be appropriate for this analysis. Propose an alternate detector; describe how it works (with a schematic if necessary) and why it would be the most appropriate for the analysis in question. **(6 points)**



- (b) It turns out Lucy's grant application has been re-evaluated and she has been granted enough money to purchase a GC-MS. She's very excited! Her supplier has indicated that the GC-MS will come standard with an EI ion source, but if she is interested in using PCI or NCI for ionization she will need to purchase an additional CI ion source. She has asked for your opinion on whether the CI source would increase the sensitivity of her analysis for the CFC replacement and products that she is interested in. Tell Lucy what type of ionization (EI, PCI or NCI) would be ideal for her analysis and why. Describe how the ions are produced in this source. **(8 points)**

(c) Using the system you helped Lucy design she has found that both Product 1 and Product 2 are major degradation products from the atmospheric oxidation of the potential CFC replacement in question. To continue her investigation she would like to sample indoor air in a building where this CFC replacement is being used as a coolant. What sampling media would you recommend she use to look **specifically** for Product 1 and Product 2 and why? (Assume Lucy has access to the ANALEST facility for analysis) **(5 points)**

(d) Lucy has tried to publish her results, but her paper was rejected due to insufficient QA/QC protocol. She doesn't understand. Explain to Lucy how accuracy and precision are experimentally determined in a sampling campaign. (No need for specific experimental details related to Lucy's sampling campaign) **(6 points)**