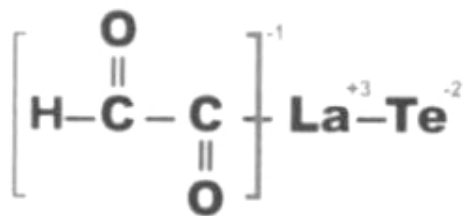


NAME & email _____

CHEMISTRY 233 – Sample Final Exam

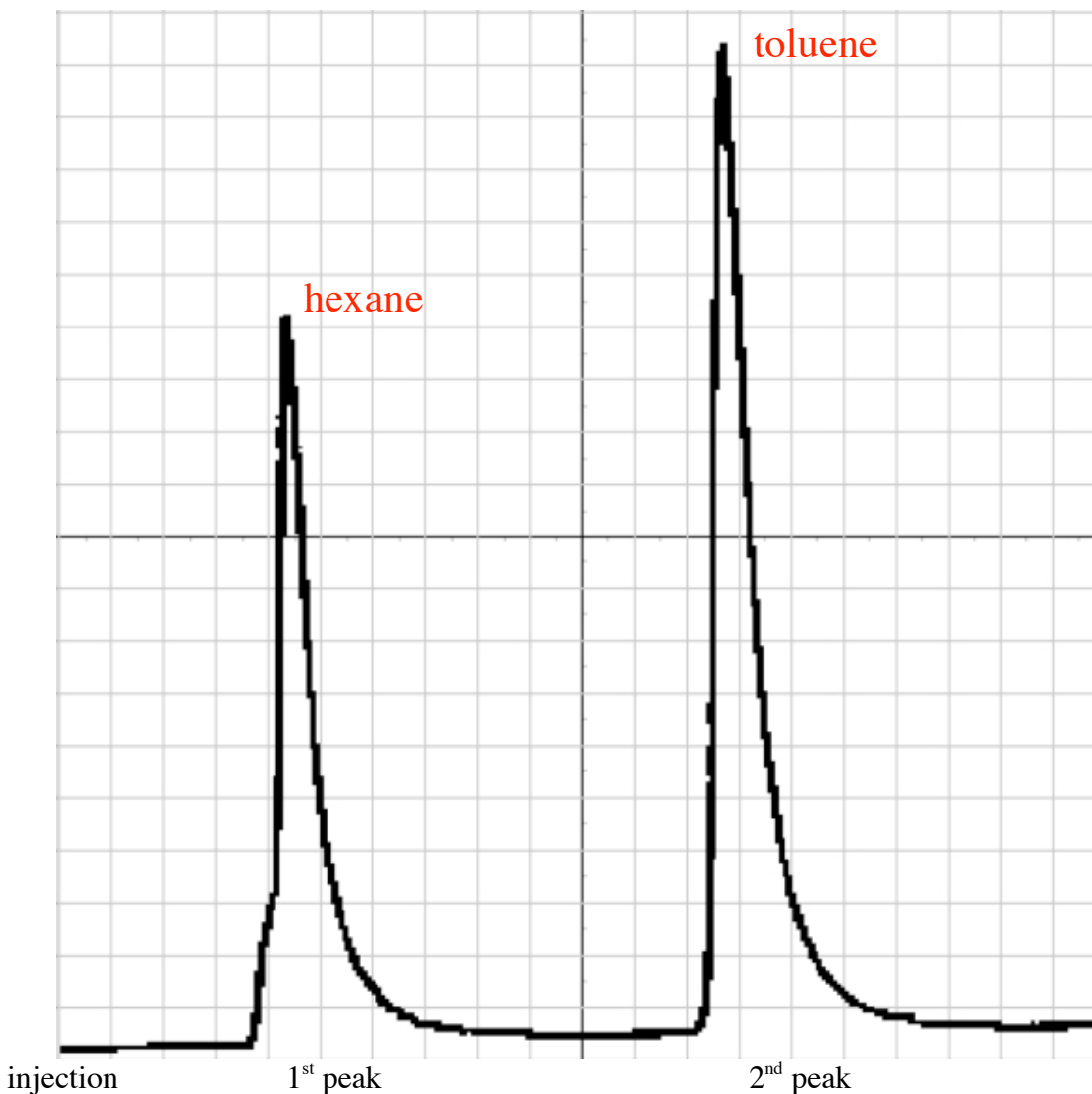


Better Eating Through Chemistry

Q 1	13 points	
Q 2	18 points	
Q 3	17 points	
Q 4	8 points	
Q 5	10 points	
Q 6	10 points	
Q 7	10 points	
Q 8	14	
TOTAL	100 points	

1. In the first lab of the semester we separated hexanes from toluene by fractional distillation. The product mixture was analyzed by gas chromatography (GC). The chromatogram shown below is of a discarded fraction.

(a) For the chromatogram shown below please identify which peak is hexane and which is toluene (5 pts).



(b) Calculate the approximate percent purity of the major compound (6 pts).

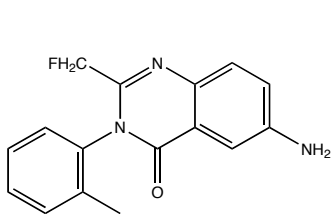
$$1^{\text{st}} \text{ peak area} \simeq 1/2 * 14 \text{ h} * 3 \text{ w} \simeq 21 \qquad 2^{\text{nd}} \text{ peak area} \simeq 1/2 * 19 \text{ h} * 4 \text{ w} \simeq 38$$

$$38 + 21 = 59 \qquad 38/59 \simeq 40/60 \simeq 66\%$$

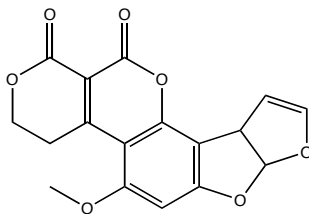
(c) How is refractive index effected by temperature (2 pts)?

It is inversely correlated.

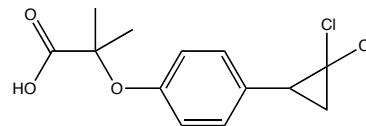
2. The second experiment you performed this year involved the separation of three compounds by a multi-step extraction scheme. You are given a mixture containing the compounds shown below, and you follow the extraction scheme given below. Indicate which compound you would expect to isolate after each step of the procedure (6 pts ea).



A



B



C

(a) Dissolve the solid in diethyl ether and extract the organic phase three times with 3.0 M NaOH. Set the organic phase aside for step (b) and combine the aqueous extracts. Treat the combined aqueous extracts with concentrated (6 M) HCl until it is acidic by litmus paper. Collect the large amount of solid precipitate by reduced-pressure filtration using a Buchner funnel. *Which compound would you expect to isolate at this point in the procedure (circle one)?*

A

B

C

(b) Extract the organic phase set aside in step (a) three times with 3.0 M HCl. Set the remaining organic phase aside for step (c) and combine the aqueous extracts. Treat the combined aqueous extracts with concentrated (6 M) NaOH until it is basic by litmus paper. Collect the large amount of solid precipitate by reduced-pressure filtration using a Buchner funnel. *Which compound would you expect to isolate at this point in the procedure (circle one)?*

A

B

C

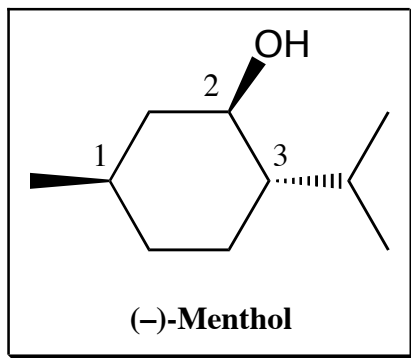
(c) Wash the organic phase set aside in step (b) three times with deionized water. Run the washed organic phase over a short column of Na_2SO_4 and rinse the column with additional ether. Combine the effluents and evaporate the solvent using a warm sand bath. *Which compound would you expect to isolate at this point in the procedure (circle one)?*

A

B

C

3. In the third experiment you performed this year, you isolated an optically and biologically active natural product by extraction of plant matter. (-)-Menthol (shown below) is another such natural product.



(a) Properly identify the absolute configuration of each of the stereogenic centers shown above (3 pts).

Stereogenic Center	1	2	3
Absolute Configuration	<i>R</i>	<i>R</i>	<i>S</i>

(b) Menthol is just one of many compounds which can be purified by recrystallization. Describe a general method for recrystallization in no more than five steps of no more than ten words each. (10 pts)

- 1) dissolve in a minimum of hot solvent
- 2) cool until no more precipitate appears
- 3) isolate by filtration
- 4) wash crystals with cold solvent



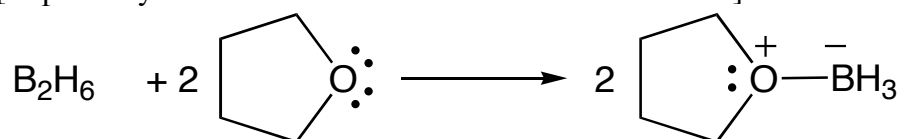
(b) You are asked to report the melting point of menthol using a MelTemp apparatus (shown to the left). What observation marks the beginning of the melting point range and what observation marks the end of the melting point range (4 pts)?

- Beginning: appearance of the first drop of liquid
 End: disappearance of last piece of solid

4. (Mayo 6-29). Why are there axial and equatorial hydroxyl isomers for 4-*tert*-butylcyclohexanol, but not for cyclohexanol itself? [Exp 5B: Reduction of Ketones Using a Metal Hydride Reagent: *cis*- and *trans*-4-*tert*-butylcyclohexanol]. (8 pts)

There are two diastereomers of 4-*tert*-butylcyclohexanol. The large *tert*-butyl group prefers the equatorial position and thus in the *trans* diastereomer, the -OH group is also equatorial. In the *cis* diastereomer, the -OH group is axial. These isomers can be separated. In cyclohexanol, the two conformational isomer interconvert readily and thus cannot be separated at normal temperatures.

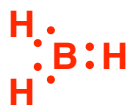
5. (Mayo 6-88). When diborane (B_2H_6) dissociated in ether solvents, such as tetrahydrofuran (THF), a complex between borane (BH_3) and the ether is formed. For example [Exp 13: Hydroboration-Oxidation of an Alkene: Octanol]:



(a) In the Lewis sense, what is the function of BH_3 as it forms the complex? Explain (5 pts).

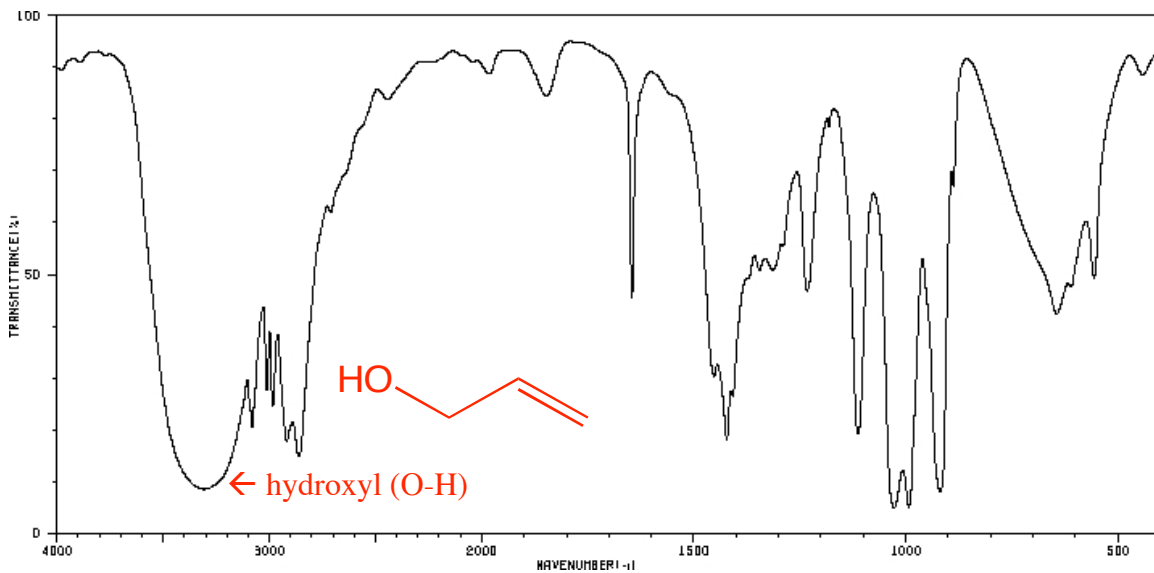
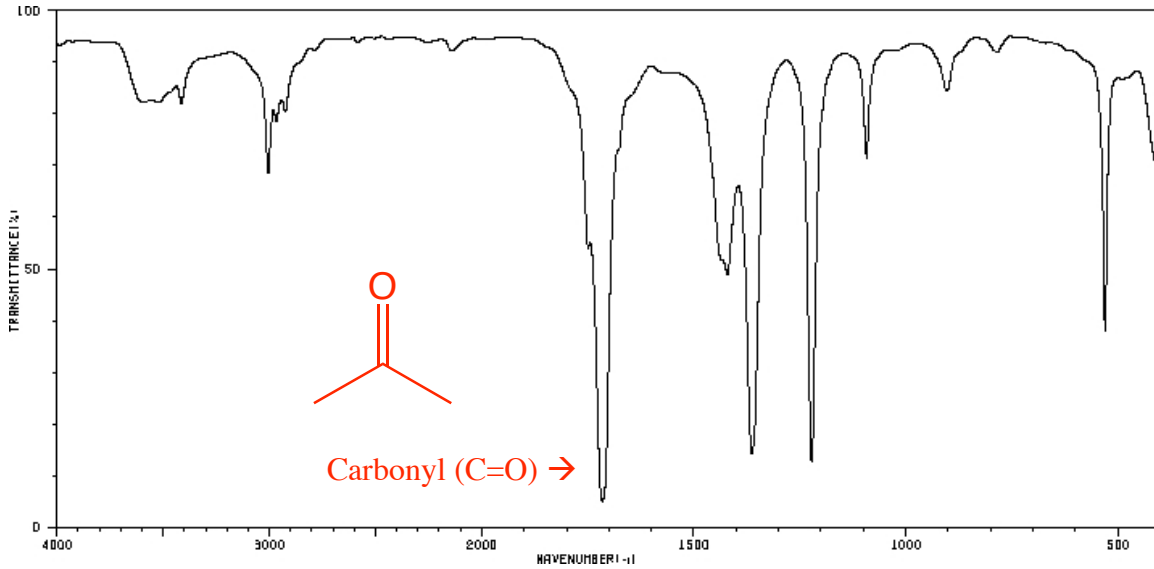
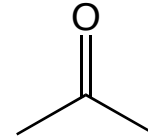
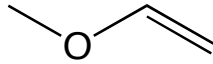
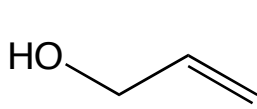
BH_3 is a Lewis acid; it accepts an electron pair from THF.

(b) Write the Lewis structure for BH_3 . Diagram its expected structure indicating the bond angles of the molecule (5 pts).

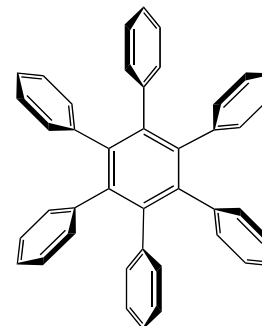


trigonal planar with 120° bond angles

6. IR spectra for two of the following three molecules (C_3H_6O) are shown below. Draw the correct molecules on their spectra and properly label the most important peak used for identification. (10 pts)

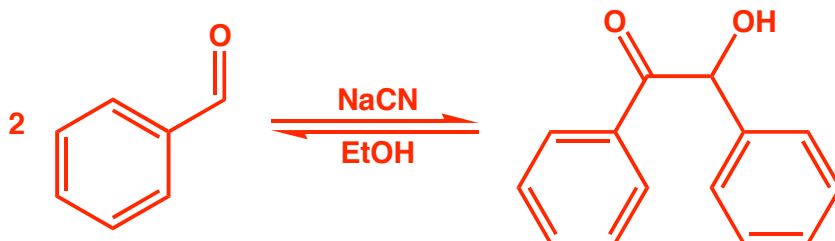


7. The focus of next semester's Organic Chemistry Lab will be the seven-step convergent synthesis of hexaphenyl benzene, shown to the right. Shown on the notebook excerpt below is the dimerization of benzaldehyde to form benzoin (MW = 212.24 g/mol), the reaction is the first step in this sequence.



(a) Fill in the missing portions of the table below. (4 pts)

1/16/2009



(cf: Mayo; Mustard; Lettuce, 867 – 5309)

chemical	Benzaldehyde	NaCN in EtOH
source	Aldrich, 98%	Stockroom (0.54 M)
purification	none	None
MW	106.13	*1.00 M*
d(g/ml)	1.0	*1.00 M*
amount	1.06 ml	1.0 ml
mmol	10	1.0
eq.	10	1.0

For benzaldehyde: $1.0 \text{ g/ml} * 1.06 \text{ ml} = 1.06 \text{ g} / 106.13 \text{ g/mol} = 10 \text{ mmol}$ benzaldehyde

For NaCN sol'n: $1.0 \text{ ml} * 1.00 \text{ mol} / 1000 \text{ ml} = 1.0 \text{ mmol}$ NaCN

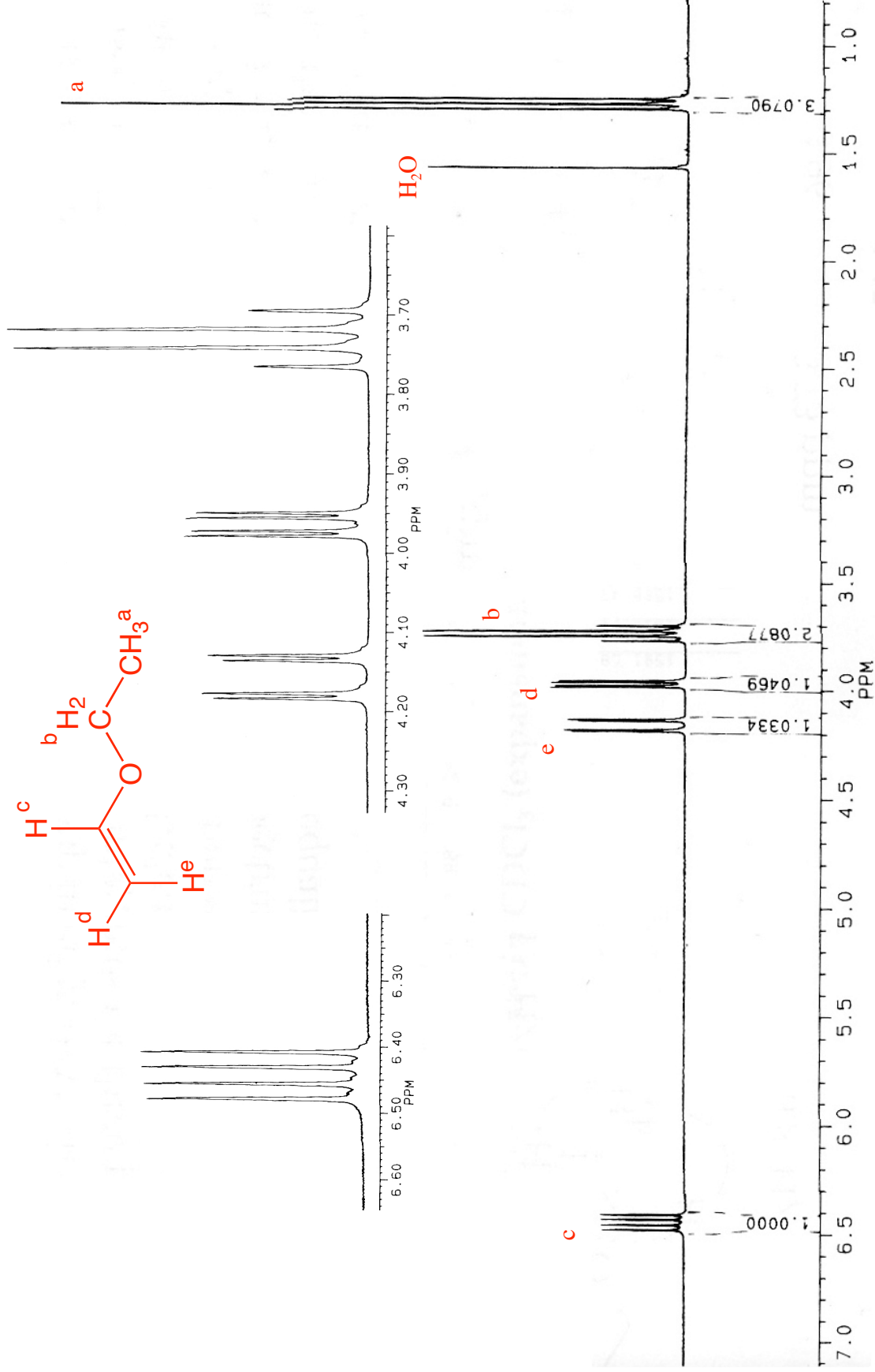
(b) Assign each of the compounds used reaction roles: starting material, solvent, catalyst or product. (4 pts)

compound	product	catalyst	solvent	starting material
benzoin	X			
ethanol			X	
benzaldehyde				X
sodium cyanide		X		

(c) What is the theoretical yield, in grams, of benzoin? (2 pts)

This is a simple dimerization; theoretical yield is 1.06 g (calculated above)

8. Fully annotate the presented ^1H NMR. (14 pts) (Microscale Organic Lab (4e), Mayo et. al. 2000, p 569).



Ethyl vinyl ether in CDCl_3 .