S343 Midterm Exam Preparation

Important midterm information:

- The exam will be held in Woodburn Hall 120. More information will be sent to you via email.
- The exam will be Tuesday, October 14, from 7:15PM-9:15PM.
- You may not use calculators. You should be able to do the small amount of necessary math by hand.
- You will not be given typical IR stretch frequency tables. You need to know the typical values as given in class.
- **You must bring your student ID with you.** If you do not bring your ID, this could lead to a delay in grading at the least, and possibly a zero on the exam.

Content: The midterm exam will be comprised of all topics covered to this point in the course and all lab experiments through the Dihydroxylation Mechanisms Lab. Major content includes, but is not limited to:

- Structural basis of intermolecular forces
- Degrees of unsaturation
- Solvents: nonpolar, polar aprotic, polar protic, relative density, water miscibility
- Solubility of compounds in aqueous and organic solvents
- Acid/base extraction
- Recrystallization
- Melting point data
- Thin layer chromatography
- Gas chromatography
- Mass Spectrometry
- IR spectroscopy
- The main purposes of labs conducted, and how the above topics applied to each lab

How should I prepare?

- Review quizzes. Answer keys are posted on the website
- Review class notes
- Review homeworks 1-3. Selected answers are/will be posted on the website
- Review labs. You do not need to know details (how much solvent was used in the extraction experiment) but you should know mechanisms of product formation, the main goal/question for each lab, the type of data necessary to answer each main question, major techniques employed in the experiment

Exam format/types of questions The exam will be out of 100 points and include short answer, interpret the data, short essay. The exam will look a lot like the quizzes and homework in style. Here are some practice questions:

1. (12pts) You are trying to purify Compound A (an acid with melting point 56 ^{o}C) that has a small impurity of Compound B (a base with melting point 79 ^{o}C). Label the following techniques as "likely effective" for purifying Compound A or "likely ineffective" for purifying compound A and explain your answer.

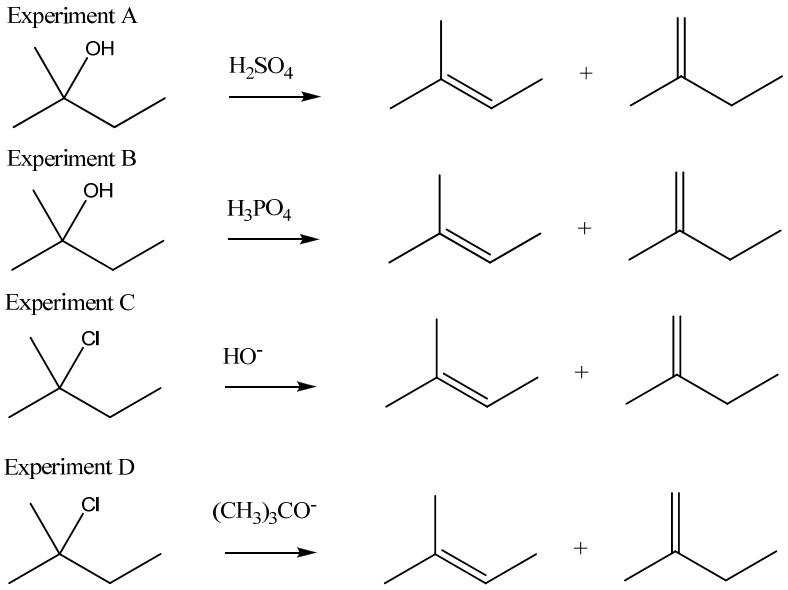
A. Extraction
B. Fractional distillation
C. TLC
D. Recrystallization

2. (8pts) You performed two dihydroxylation experiments in lab and determined whether the products were consistent with a syn or anti addition mechanism using TLC. Label the following statements related to this experiment as True or False.

_____ If a dihydroxylation reaction were to produce both of the enantiomers of trans-1,2-cyclohexanediol, we would expect to see two spots by TLC.
_____If the cis diol had an impurity in it, the R_f value of its TLC spot would have increased.
_____IR spectroscopy data could have been used to reach the same conclusions as the TLC data in this experiment.
____GC data could have been used to reach the same conclusions as the TLC data in this experiment.

Questions 3-5 are based on this experiment, similar to the elimination reaction you studied.

A team of four students carried out the following four elimination reactions which all yielded a mixture of 2-methyl-1-butene (bp 31 ^{o}C) and 2-methyl-2-butene (bp 37 ^{o}C.) The mix of alkene products was then collected by distillation and analyzed by gas chromatography. It was determined that 2-methyl-2-butene has a longer retention time on the GC.



The GC data collected was this:

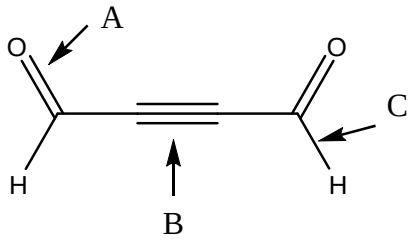
Experiment	Peak A area (2.1 min retention time)	Peak B area (3.2 min retention time)
A	300	1200
B	298	1203
C	750	751
D	1200	301

3. Does the identity of the acid affect the product distribution in acid catalyzed eliminations? Explain how the data support this answer.

4. Does the identity of the base affect the product distribution in base promoted eliminations? Explain how the data support this answer.

5. In experiment D, did the product distribution show selectivity toward the formation of 2-methyl-1-butene or 2-methyl-2-butene? Give a rationale based on physical principles for why this is the case.

6. Refer to the molecule below to answer these questions:



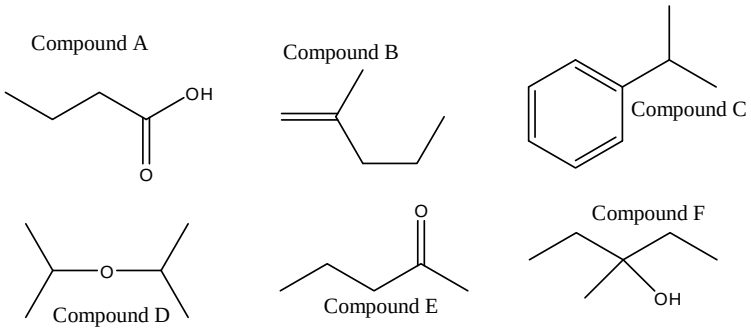
Rank the bonds according to IR frequency absorption:

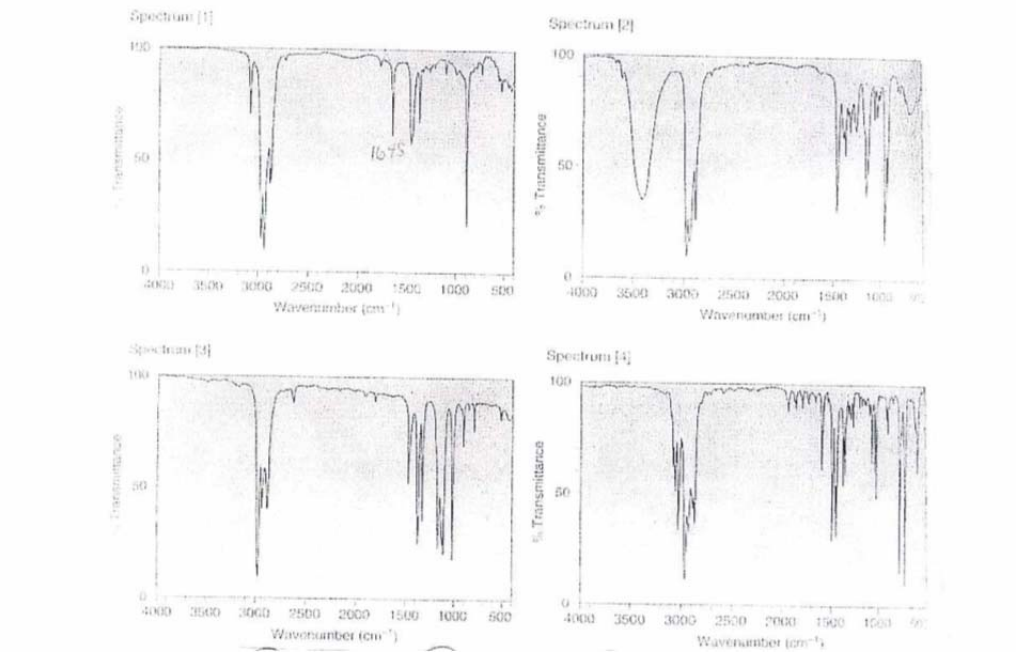
Highest frequency bond _____ > _____ > _____ lowest frequency bond

Rank the bonds according to intensity on the IR spectrum:

Highest intensity bond _____ > _____ > _____ lowest intensity bond

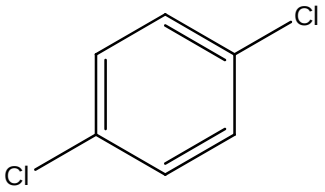
7. Match each spectrum to the appropriate compound.



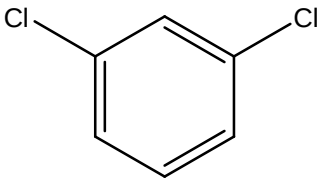


Questions 8-13 refer to this information:

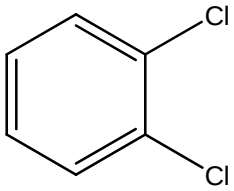
A student performed a reaction in organic laboratory that was designed to make about 2 grams of Product A. The reaction is also known to produce small quantities of Products B and C.



Product A
boiling point: 173 °C
melting point: 52 °C



Product B
boiling point: 173 °C
melting point: -25 °C



Product C
boiling point: 178 °C
melting point: -18 °C

8. Upon isolating the solid unpurified product, the student took a melting point, which was 41-49 °C. What does this data suggest about the purity of Product A? Explain the physical basis of depressed, broad melting points.

9. The student took a TLC of the crude product mix. How many spots would you expect to see if the product were purely Compound A? How many spots would you expect to see on a well resolved TLC plate if Compound A contains both Compound B and C as impurities?

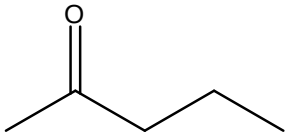
10. Would fractional distillation be an effective way to purify this material? Explain.

11. Would recrystallization be an effective way to purify this material? Explain.

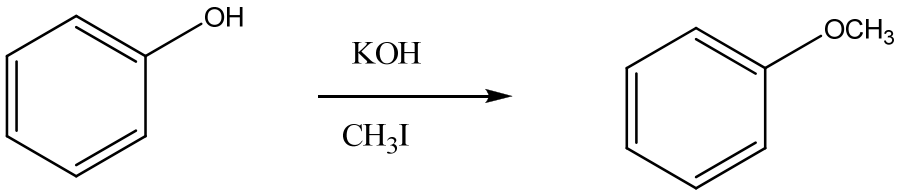
12. Would liquid/liquid extraction using a separatory funnel be an effective way to purify this material? Explain.

13. Would gas chromatography be an effective way to analyze the purity this material? Explain.

14. (18pts) A mass spectrum of the molecule below was obtained. Write a mechanism for the fragmentation that leads to MS signals at m/z = 43 and m/z = 58.



15. (24 pts) Read this procedure and answer the following questions.



In a 25 mL roundbottom flask, dissolve 10 mmol of KOH in 10 mL of ethanol. Add 2 mmol of phenol and 2 mmol of methyl iodide. Attach a reflux condenser and heat the mixture to boiling for 1 hour. Allow the reaction to cool, then pour the reaction mixture into 50 mL of 1 M NaOH. Pour the aqueous solution into a separatory funnel and add 30 mL of ether. Shake and vent. Separate the organic layer. Add sodium sulfate to the organic layer until it no longer clumps. Decant the ether layer into a roundbottom flask and evaporate the solvent to give methyl phenyl ether as an oil.

A. What is the purpose of attaching a reflux condenser?

B. Why was the reaction extracted out of 1 M NaOH? Would 1 M HCl have been more or less effective?

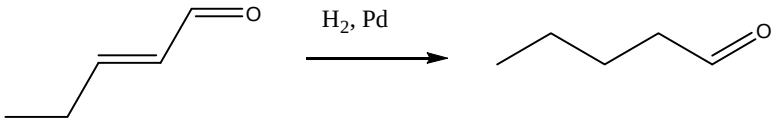
C. What is the purpose of adding sodium sulfate?

D. Which of these ways could you use as an effective means to test for the purity of your final product? Circle any that apply.

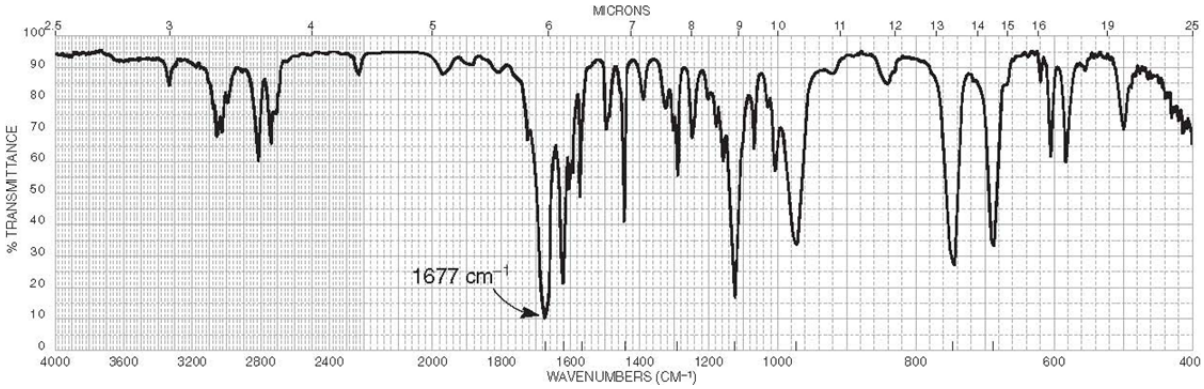
Mass spectrometry IR Spectroscopy TLC Melting point Gas Chromatgraphy

E. If 1.5 mmol of product was collected, what is the percent yield for this reaction?

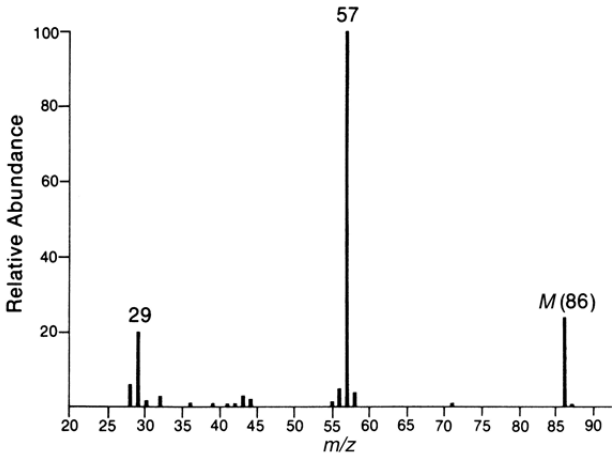
16. (6pts) How could IR be used to determine when this reaction went to completion and give the intended product? Include reference to three specific frequencies.

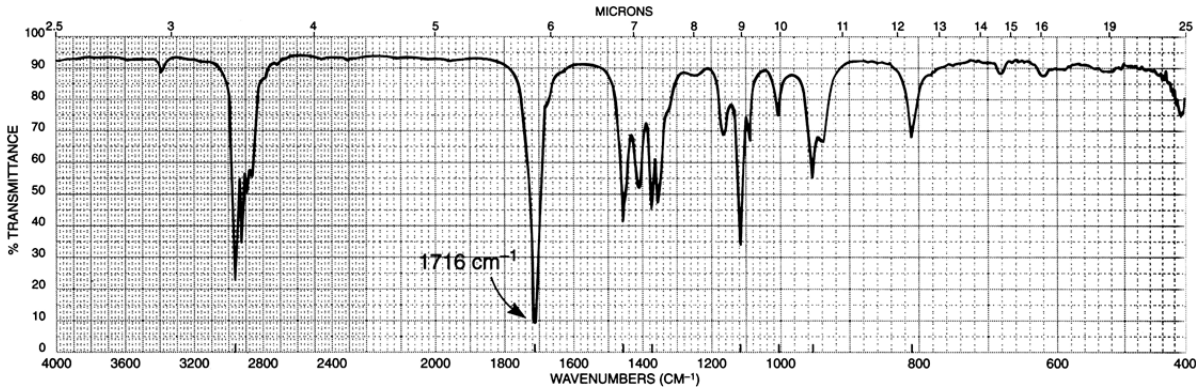


17. Provide a reasonable structure of a compound that is consistent with the molecular formula C_9H_8O and the following IR spectrum.



18. Provide the structure of a compound that is consistent with the following MS and IR data. Using your structure, propose a mechanism that accounts for the MS base peak.




MICRONS
2.5
3
4
5
6
7
8
9
10
11
12
13
14
15
16
19
25
100
90
80
70
60
50
40
30
20
10
0
% TRANSMITTANCE
1716 cm⁻¹
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
600
400
WAVENUMBERS (CM⁻¹)