

Department of Chemistry
University of Missouri-St. Louis

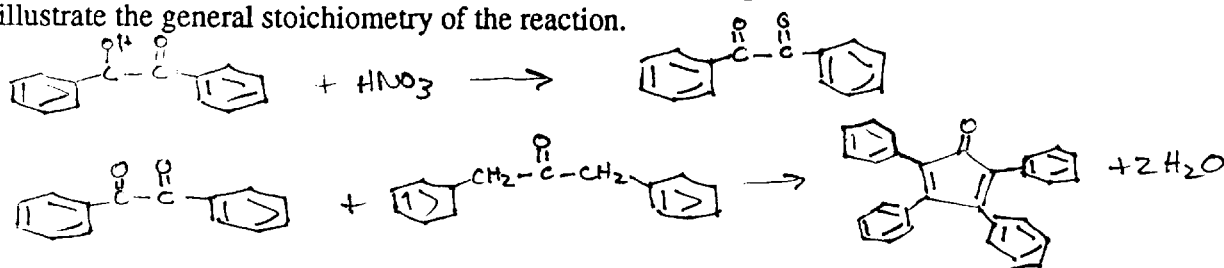
Name _____
Chem 263

Winter 1996
2nd Exam

Atomic weights: C: 12; H: 1; O: 16; N: 14

1. (20pts) A student began with 5.0 g of benzoin and using an excess of nitric acid oxidized the benzoin to benzil. A total of 4.0 g was isolated after recrystallization. A portion of the benzil (diphenylethanedione), 2.0 g, was then reacted with an equivalent molar amount of diphenylacetone in anhydrous base to produce tetraphenylcyclopentadienone. The tetraphenylcyclopentadienone was isolated in 80 % of the theoretical yield.

A. Write equations to illustrate the two reactions. The oxidation equation need not be balanced but should illustrate the general stoichiometry of the reaction.



B. How much diphenylacetone (1,3-diphenyl-2-propanone) was used in the condensation reaction?

the molecular wt of both reagents is the same 210g/mol
2g was used (diphenylacetone)

C. How much tetraphenylcyclopentadienone was isolated?

1 mole of benzil \rightarrow 1 mole \rightarrow TCP mw TCP = 384
 $2g/210 = 0.0095$
 $0.0095 \times 0.8 = 0.00762$ mol TCP isolated
 $0.00762 \times 384 g/mol = 2.92g$

D. What is the overall yield of the two reactions?

First yield = $5/212 = 0.0236$ mol benzoin \times
 0.0236 mol \times 210 = 4.95 g theoretical yield
 $4/4.95 = .808$ or 80.8 %

Overall yield = yield 1 \times yield 2 = $0.808 \times 0.8 = 0.646 =$
 64.6 %

2. (20pts) A. The separation of compounds by taking advantage of whether they are volatile or not in steam is an important commercial process. Calculate the amount of water that would be necessary to steam distill 1.0 g of o-nitrophenol at approximately 100° C if the vapor pressure of o-nitrophenol is 2.5 mm at this temperature. Assume that the contribution of o-nitrophenol to the total vapor pressure is negligible with regards to the boiling point of the mixture.

$$\begin{aligned} \text{vp pressure of H}_2\text{O} &\approx 760 \text{ mm} \\ \text{vp of o-nitrophenol} &= 2.5 \text{ mm} \end{aligned}$$

$$\frac{760}{2.5} = \frac{\frac{\text{wt}}{18}}{\frac{2}{139}}$$

$$\frac{P_{\text{H}_2\text{O}}}{P_{\text{ONP}}} = \frac{n_{\text{H}_2\text{O}}}{n_{\text{ONP}}}$$

$$m\text{w of ONP} = 139$$

$$\text{wt} = 18 \left(\frac{760}{2.5} \right) \cdot \frac{2}{139}$$

$$\text{wt} = 78.7 \text{ g} \quad \text{or } \approx 79 \text{ mL}$$

B. The vapor pressure of p-nitrophenol at 100° C is 0.02 mm. If p-nitrophenol is present with the ortho isomer during the steam distillation, how much of the p-isomer would be carried along with the ortho isomer during the collection of the x mL of water you just calculated. Assume that the ortho and para isomers are immiscible with each other. On the basis of this calculation and your assumptions, is it possible to achieve a reasonable separation of the ortho and para isomers if both are originally present in comparable amounts?

$$\frac{760}{0.02} = \frac{\frac{78.7}{18}}{\frac{\text{wt PNP}}{139}}$$

$$\begin{aligned} \text{wt PNP} &= \frac{78.7}{18} \cdot \frac{0.02}{760} \cdot 139 \\ &= 0.016 \text{ g} \end{aligned}$$

$$\frac{P_{\text{H}_2\text{O}}}{P_{\text{PNP}}} = \frac{n_{\text{H}_2\text{O}}}{n_{\text{PNP}}}$$

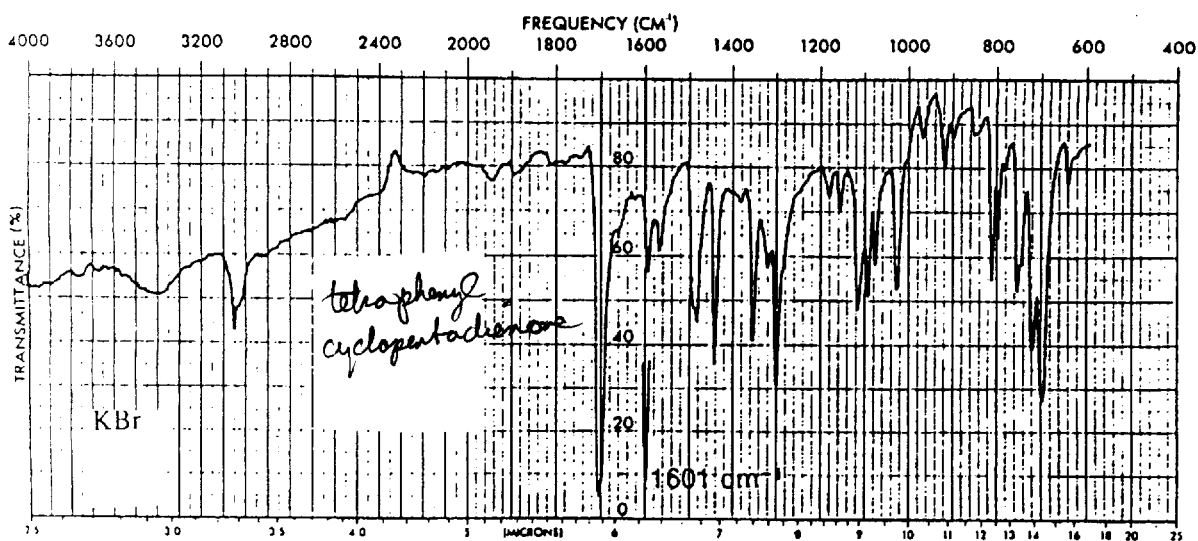
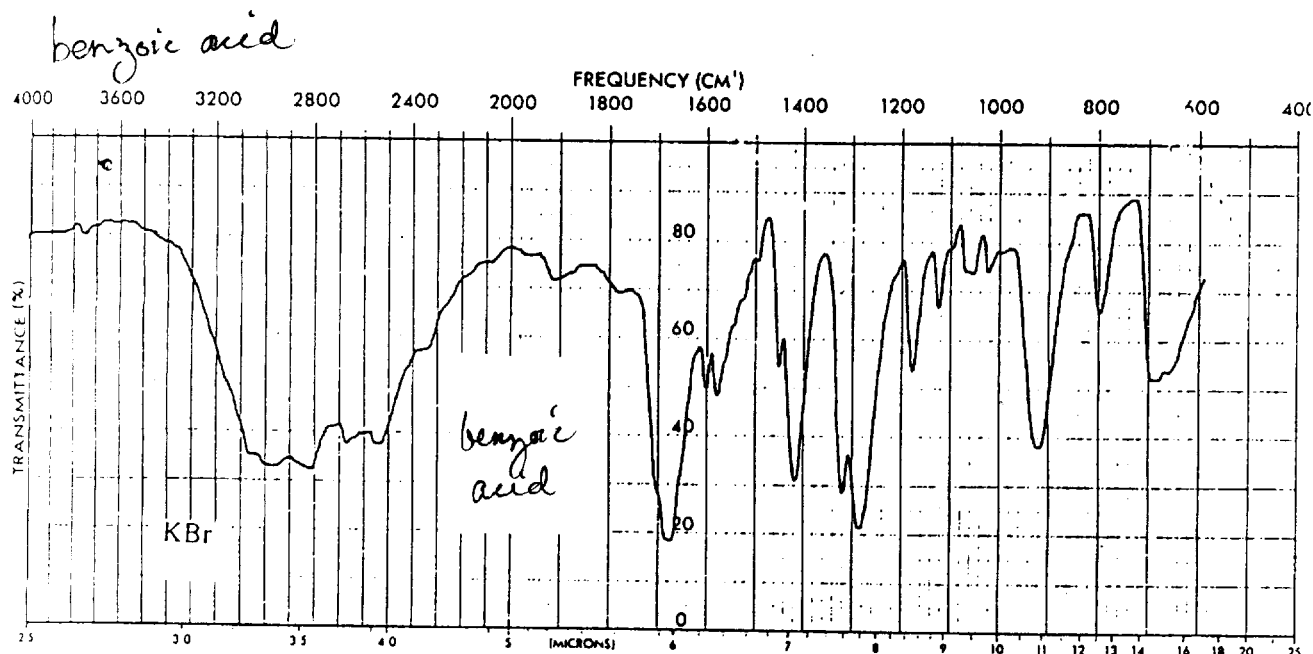
3. (15 pts) Suppose you are asked to determine whether a material isolated from a hazardous waste site was identical to a sample you have in a jar in the laboratory. How would you confirm this materials identity. List as many criteria as you can using the different techniques you have used this semester.

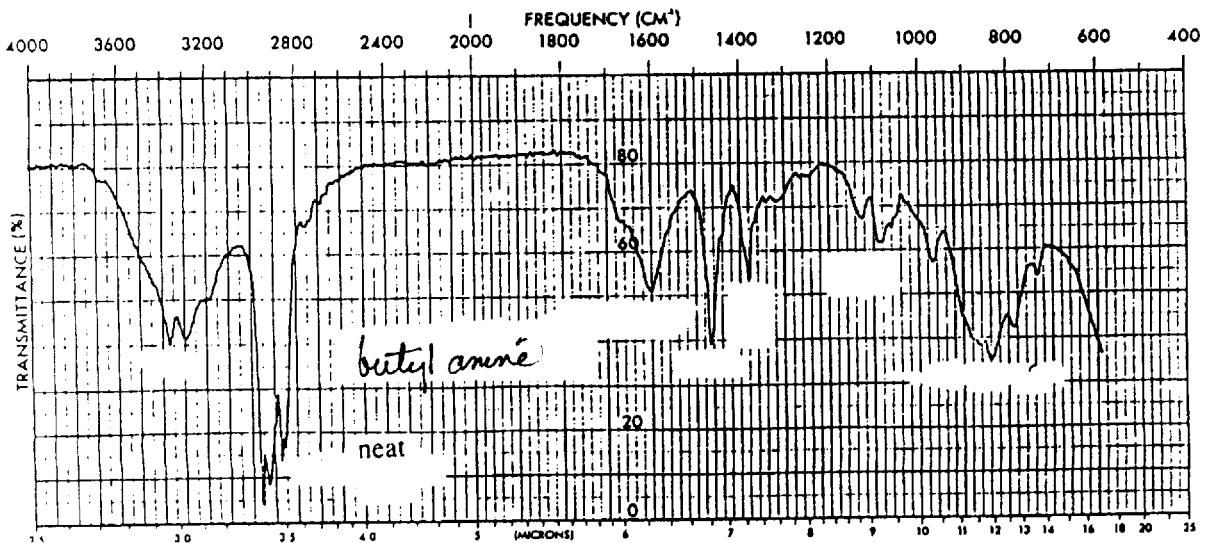
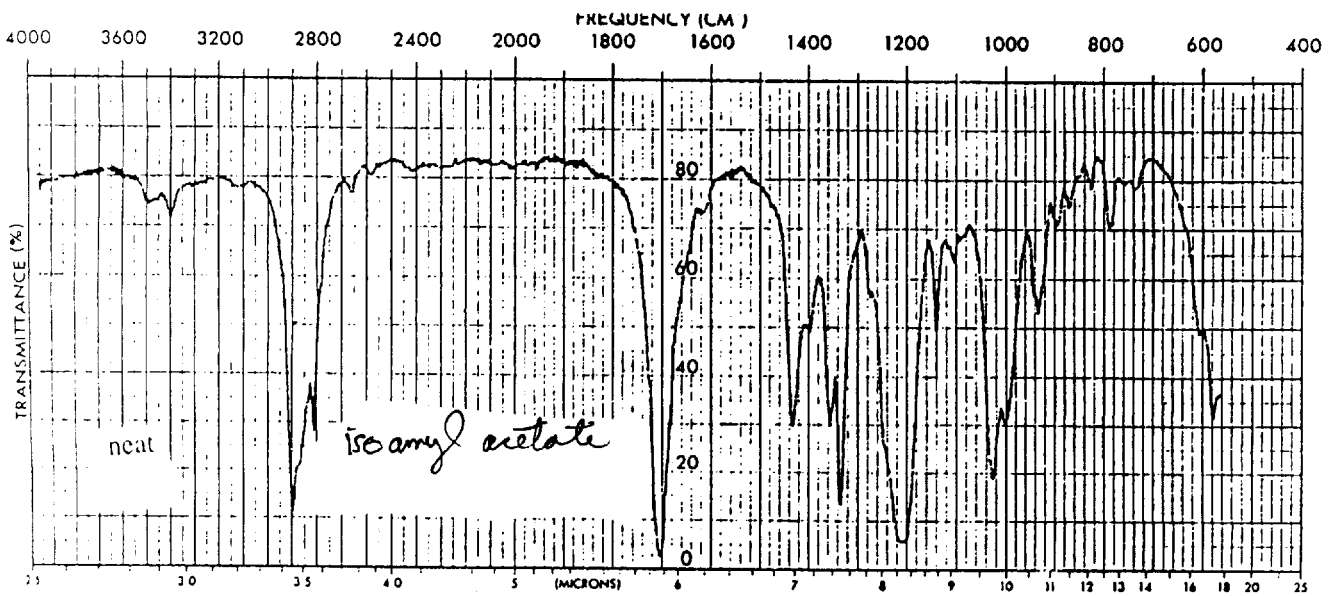
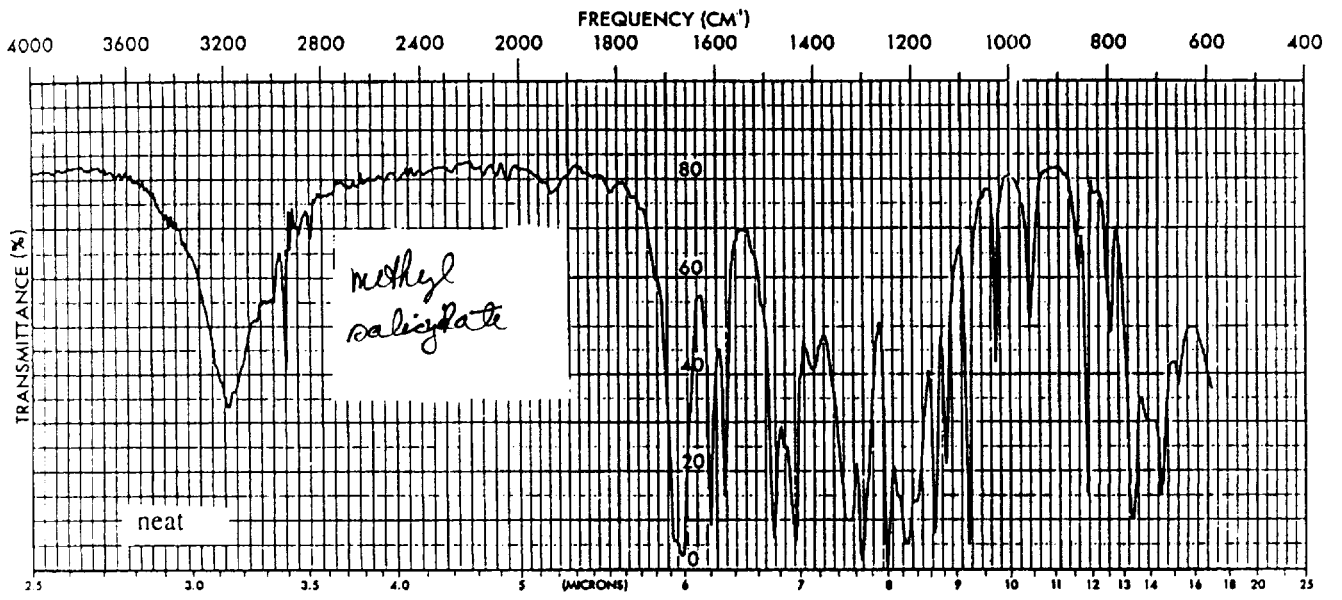
Run GC if volatile

TLC if not volatile

if pure run bp or if solid run mp

4. A student in Chem 263 was in a rush, it seems, for the entire semester. Although all the experiments were completed in a timely fashion, on examining the infrared spectra that were taken, it was realized that the spectra were not associated with a structure or any other identifying notation except perhaps for a few colored smudge marks. Help a fellow colleague out by associating the correct structure with the following spectra. The compounds prepared during the semester that did not have a properly labeled spectrum include: methyl salicylate, isoamyl acetate, tetraphenylcyclopentadienone and the students solid and liquid unknowns, which were properly identified as benzoic acid and n-butyl amine. Draw the structure on each spectrum.





5. (20pts) a. Explain how fractional distillation differs from a simple distillation.

*a fractional distillation contains many more theoretical plates
(i.e. the number of times liquid is in equilibrium with its vapor)*

- b. Describe in simple terms how a gas chromatograph works.

a sample is vaporized, separated on a column according to its volatility and detected as it elutes from the column.

- c. What are the advantages and disadvantages of using Nujol over KBr as a medium for infrared analysis.

NOT covered this semester

*NUJOL has an IR spectrum that obscures some peaks
KBr is hygroscopic - absorbs H₂O and CO₂*

- d. Explain what is meant by a theoretical plate

see 5 above