

Name: _____

Organic Chemistry Laboratory

ID #: _____

Fall 2003

EXAM #1, Friday, October 17, 2003

Before starting:

Check your exam to make sure that it is complete. Place the initials of your name on each page. There should be a total of 8 pages. Problems are on the front of each sheet.

Read each question carefully, making sure that you completely answer each question. You may use the back of a sheet for additional space if needed.

Remember: Questions answered in pencil, red ink, erasable ink, or that have white-out on them cannot be regraded.

Good luck.

Question #	Possible Points	Points Scored
1	35	_____
2	39	_____
3	38	_____
4	34	_____
5	30	_____
6	24	_____
Total:		_____

1. Multiple choice and short answer (35 pts).

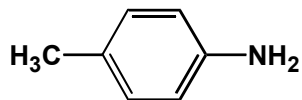
Circle the best answer to each question:

A) Which of the following statements is false?

- a) anhydrous magnesium sulfate is used as a drying reagent
- b) distillation separates molecules based on their polarity
- c) distillation under reduced pressure produces a lower boiling point
- d) GC is well suited for quantitative analysis
- e) none of the above

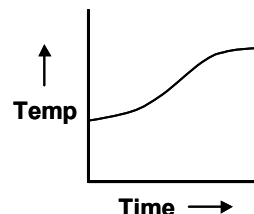
B) The organic compound shown below could be extracted into water from an organic solvent using _____ and then removed from water into an organic solvent by the addition of _____.

- a) sodium hydroxide, sodium bicarbonate
- b) ammonia, sodium hydroxide
- c) hydrochloric acid, sodium hydroxide
- d) sodium bicarbonate, sodium hydroxide
- e) none of the above



C) A student plotted a Time vs. Temperature graph during a simple distillation. The sample was collected at a constant rate of 10 drops per minute. The graph is shown below. What conclusion can be made about the sample analyzed by the student?

- a) the student was unable to get the sample to boil
- b) the sample contained one component
- c) the time of the distillation exceeded the temperature
- d) the student started the distillation at room temperature
- e) none of the above



D) For (+)-carvone and (-)-carvone, which property should not be identical?

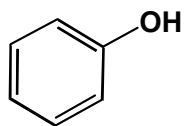
- a) solubility in diethyl ether
- b) density
- c) NMR spectrum
- d) specific rotation
- e) none of the above

- E) A student is analyzing a ^1H NMR spectrum that contains five different compounds:
i) benzene (C_6H_6), ii) ethanol ($\text{CH}_3\text{CH}_2\text{OH}$), iii) chloroform (CHCl_3), iv) deuterated dichloromethane (CD_2Cl_2), and (v) TMS ($\text{Si}(\text{CH}_3)_4$).

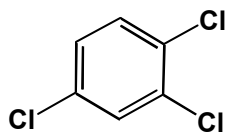
Which compound will display the highest upfield chemical shift in the spectrum?

- a) benzene
- b) ethanol
- c) chloroform
- d) deuterated dichloromethane
- e) TMS

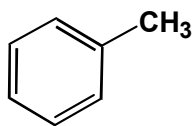
2. The questions that follow concern the separation and analysis of the following four aromatic compounds (39 pts):



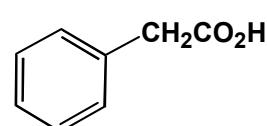
phenol
mw = 94 g/mol
bp = 182 °C



trichlorobenzene
mw = 181 g/mol
bp = 214 °C



toluene
mw = 92 g/mol
bp = 110 °C



phenylacetic acid
mw = 136.14 g/mol
bp = 265 °C

- A) What order of elution would you expect for these compounds on column chromatography (*i.e.* silica as stationary phase) using methylene chloride as the eluant?

eluting first

eluting last

- B) What order of elution would you expect for these compounds using GC analysis?

eluting first

eluting last

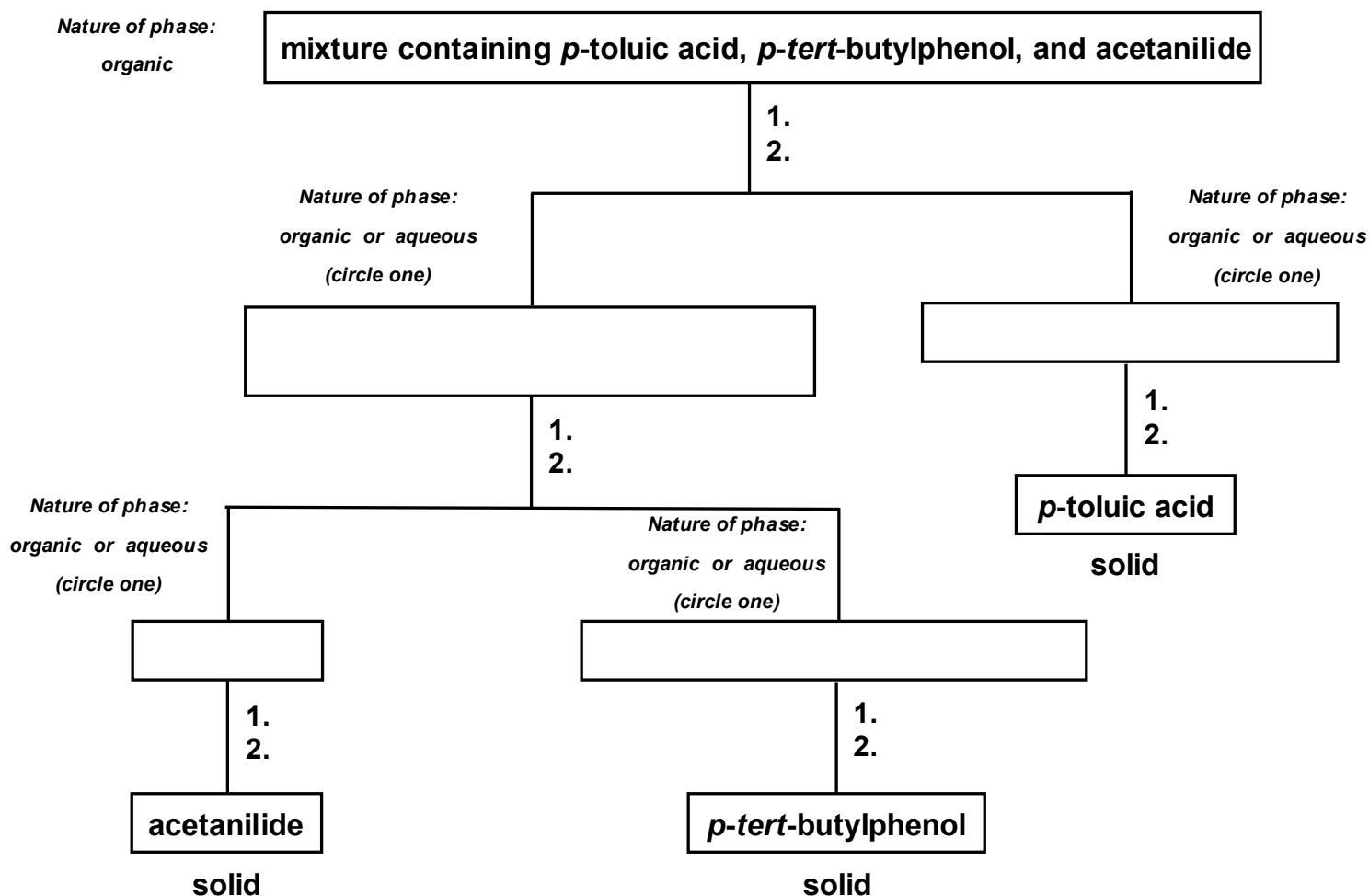
C) Which compound(s) would be extracted from an organic phase into an aqueous phase containing sodium hydroxide?

D) Which compound(s) would not be extracted from an organic phase when washed with water, hydrochloric acid, sodium hydroxide, or sodium bicarbonate?

E) Which compound(s) would not give a singlet in a ^1H NMR spectrum?

3. You are instructed to separate a mixture of three compounds from an organic phase that contains *p*-toluic acid, *p*-tert-butylphenol, and acetanilide using the flowchart below as your guide.

Fill in the missing information of the flowchart illustrating: i) the nature of each phase of your separations (circle appropriate answer), ii) the identity of each compound(s) in each phase (place answer(s) in each box), and iii) the steps taken and/or reagents used to get to each phase or the final compound (place answer next to the two numbers given between the boxes) (38 pts).



4. Consider the information below and answer the question that follows (34 pts).

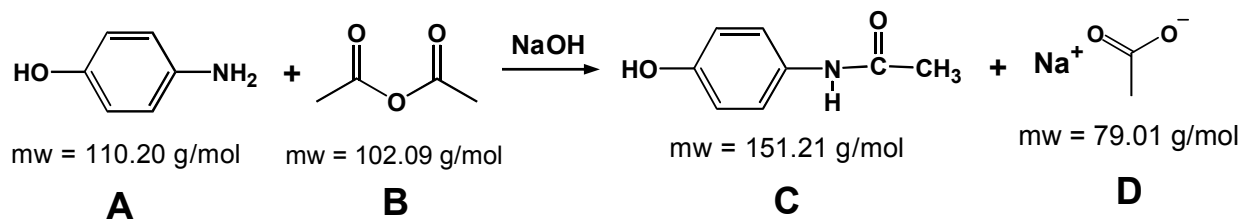
An unknown Compound A (molecular formula: $C_7H_6O_2$) is insoluble in hot water. Addition of one equivalent of NaOH to Compound A in water, however, completely dissolves Compound A. Compound A displays a strong absorption in the IR spectrum at 1689 cm^{-1} .

^1H NMR data for Compound A are as follows:

Compound A: 11.6 ppm (singlet, 1H), 7.6 ppm (multiplet, 5H)

Propose a structure for Compound A. Explain your answer using all of the information given. For the ^1H NMR spectrum, be sure to label your compound to clearly outline your peak assignments.

5. Consider the reaction below and answer the questions that follow (30 pts).



A student of organic chemistry has conducted the reaction above. To conduct the reaction, the student added 22 g of A and 10 g of B to a solution of NaOH dissolved in aqueous ethanol and heated the reaction mixture to reflux for a period of 50 minutes. The reaction produced 12 g of C. A work-up of the reaction mixture, which involved an extraction and recrystallization, produced 10 g of C.

A) Calculate the percent yield of C based on the limiting reagent (show all work).

B) Calculate the percent recovery of C based on the limiting reagent (show all work).

C) Describe how a ^1H NMR spectrum, without knowledge of the mass of C produced, can be used to determine the percent yield of C according to the reaction above.

6. Indicate the purpose of each of the following materials or equipment as described in our organic chemistry laboratory. Use complete sentences and be specific (24 pts).

A) TLC plate

B) rotary evaporator

C) NUTS

D) syringe needle