CHEM 237 | Fall 2008 | Name answer key
J. Medina | Laboratory Exam | October 30th, 2008

total max points 56

Read all questions entirely and carefully before giving your answers. Be brief and use chemical equations or diagrams when appropriate.

1. (2 points) Why is it typically not recommended to use Bunsen burners for heating purposes in an organic chemistry laboratory?

The substances used in lab are or can be flammable. A Bunsen burner can induce a fire in the lab

2. (2 points) What is the proper disposal procedure for organic solvents in the chemistry laboratory?

There are two types of solvents, halogenated and non halogenated. Solvents need to be disposed of in the properly labeled waste container.

3. (2 points) Why is it necessary to maintain a well-written laboratory notebook?

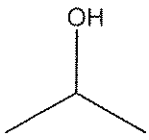
- For everyone to be able to follow the experimental procedure
- to determine possible areas of errors or improvement.
- to help analyze data and draw appropriate conclusions based on experimental data.
- to have necessary background information available

4. (2 points) What are the components of a laboratory report? Which components are considered pre-lab assignment?

pre-lab:
- date
- title
- objective
- introduction
- table of physical constants
- procedure

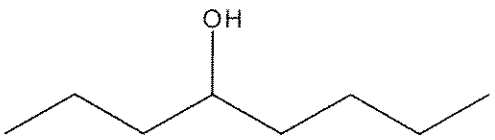
- data
- results
- post-lab questions
- conclusions

5. (4 points) Predict if the following substances are soluble, partially soluble or insoluble in water. Briefly explain your choice.



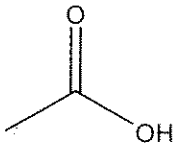
A

soluble
3 carbons
polar OH



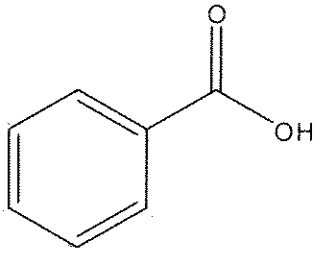
B

insoluble
8 carbons
polar OH



C

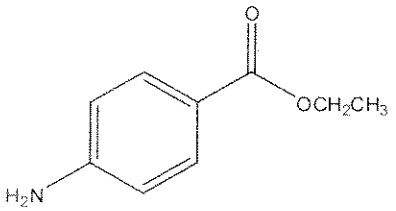
soluble
3 carbons
polar 



D

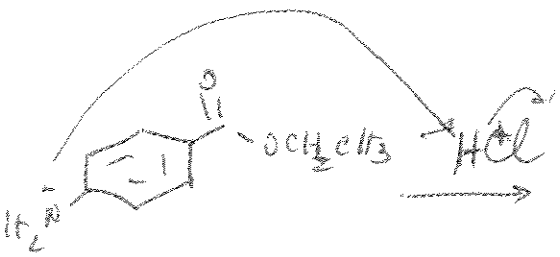
partially soluble to insoluble
7 carbons
polar 

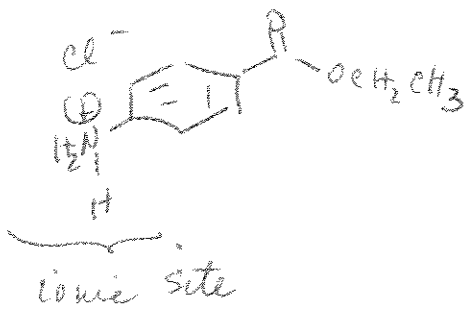
6. (6 points) Ethyl 4-aminobenzoate (structure given below) is insoluble in water. Can you predict if the solubility of this compound can be increased in either a base or an acid aqueous solution? Please include a chemical reaction to support you answer.



Ethyl 4-aminobenzoate

The solubility of this compound will increase in an acid aqueous solution due to the basic $-NH_2$ present. The acid-base reaction will produce a salt that will be soluble in water

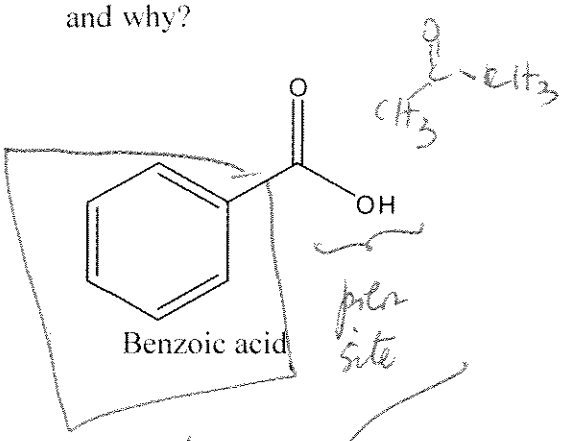




ionic site

Too polar · not polar · best match · low polarity

7. (3 points) A student was asked to dissolve benzoic acid in a suitable solvent. Solvents to choose from are water, hexane, acetone and methylene chloride. Which solvent should the student use and why?



Benzoic acid

polar site

7 carbon units

overall intermediate to low polarity

acetone is the best solvent to choose from due to similar functional group $-\overset{O}{\overset{\|}{C}}-$ and intermediate polarity.

8. (4 points) What is the difference between crystallization and precipitation? Which method is a better purification method and why?

- precipitation is a faster solidification method by putting the flask in ice after heating
- crystallization is a slower method to obtain solids from a heated solution.

crystallization is a better method since slow precipitation avoids trapping impurities in the solid

9. (2 points) How can a student experimentally check if a crystallization procedure was successful?

By determining the MP of the crystallized sample.
Solid must be completely dry
MP should be compared to literature value
- Purity is reflected by experimental value within 1-2 °C from literature value
- melting point range is 1 °C.

10. A sample of 3.25 grams of crude caffeine needs to be crystallized. The solvents to choose from are water, benzene and ethanol. Given the following solubility data:

Solubility of caffeine in selected solvents (grams/ 100 mL)

Solvent	Solubility at 25 °C	Solubility at 100 °C
Water	2.1	66.7
Benzene	1.0	4.5
Ethanol	1.5	4.5

a) (2 points) Which solvent is the most suitable for crystallization of caffeine and why?

Water because it shows the greatest difference in solubility in hot water Vs cold.

b) (2 points) What volume of the chosen solvent is required for the crystallization of the given amount of caffeine?

$$3.25\,g \times \frac{100\,mL}{66.7\,g} = 4.87\,mL$$

c) (3 points) Based on the solubility data, calculate the amount in grams of crystallized caffeine recovered from the solvent of choice.

$$4.87\,mL \times \frac{2.1\,g}{100\,mL} = 0.102\,g$$

$$g\ caffeine = (3.25 - 0.102)\,g = \boxed{3.15\,g}$$

amount of caffeine recovered will be less than 3.15g because impurities must be taken into account.

11. (4 points) Please briefly describe the process of extraction and how it is used to isolate a desired compound.

Extraction is the partitioning or distribution of a compound into two immiscible solvents according to its relative affinity for each solvent due to polarity.

- To isolate a compound, it is dissolved in a solvent and then mixed with a second immiscible solvent where the solute

12. (2 points) What is a drying agent? What are drying agents typically useful for in an extraction procedure?

A drying agent is a substance that absorbs water from an organic solvent. They are used to completely remove any remaining traces of water that might be present after extraction and can affect the purity of the solid after evaporation.

13. (4 points) What is the most fundamental principle of the chromatography technique? Diagrams can be used on explanations.

The chromatography technique is a separation of the components of a mixture based on different affinities towards the stationary phase or mobile phase. Relative affinity is due to polarity properties of the individual components of the mixture

14. (6 points) A classmate was attempting to run a TLC plate and observed the following:
- first attempt: spot did not move from the origin.
- second attempt: spot disappeared and solvent became a little colored.
- third attempt: spot moved all the way to the top.

Please explain to your classmate what is going wrong in each of the above attempts and what he/she should do to successfully run the TLC plate.

- first attempt: solvent used is not sufficiently polar to carry the analyte with it. (higher affinity of the spot for the TLC coating)

- second attempt: the spots were placed too close to the bottom edge and dipped in the solvent so the mixture dissolved in the solvent.

- third attempt: solvent used was too polar.

Student should spot the sample about 1 cm from the edge of the plate. Make sure that the spot is above the solvent level and use a solvent mixture that has a polarity in between non polar first attempt and too polar third attempt.

15. A student performing a fractional distillation to separate two liquids collects the following data:

Volume of distillate (mL)	Temperture °C
1.0	61
2.0	63
3.0	65
4.0	70
5.0	70
6.0	72
7.0	73
8.0	76
9.0	85
10.0	90
11.0	95
12.0	98
13.0	98
14.0	98
15.0	99

1st component

transition

The student looks at the boiling data given: hexane, 69 °C; cyclohexane, 81 °C; heptane, 99 °C; toluene, 111 °C; ethylbenzene, 136 °C..

Based on the experimental data, answer the following questions:

a) (2 points) Was the student successful in separating the two components of the mixture by fractional distillation? Justify your answer.

1st component was separated up to mL 6.0 after a mixture of 1st + 2nd component was obtained until mL 11.0. The last 4 mL seem to be pure 2nd component.

b) (2 points) What is the identity of the two components in the unknown mixture? Justify your answer.

1°st component is hexane temprature leveled at 70°C

2nd component is heptane 4 mL distilled at 98-99°C

c) (2 points) Discuss any possible sources of error while performing this experiment based on the experimental data.

mL 6-10 over heating

mL 1-3 heat was escaping to atmosphere.