

## Chem 269. Final Exam Information.

The exam will have 20 multiple choice questions, each worth 1 pt for a total of 20 points for the exam. The exam will count as 20% of your final grade. No calculators or any electronic devices in use. Bring a # 2 pencil with you.

**\* You will submit both the answer sheet and the exam sheet itself before leaving. \***

Please arrive and be ready to be seated by exam time on the day of the exam. Wait outside of the exam room until invited in. If you arrive late, wait for us to direct you to a seat. If something comes up last minute and you are unable to come to the exam, immediately see me or contact me by email as soon as you are able.

If you have a conflict with another final exam, or if you have three exams scheduled for the same day, you must follow the procedure specified by the registrar. This includes getting a form from the registrar's office and submitting it to the course instructor.

If you need to arrange to take the exam through Disability Services, you must arrange it with them by their deadline.

How to study? Review the OWLs (as past due assignments). Review postlab questions from the experiment handouts. Look over the experiment handouts and be able to answer why each step was done and what might happen if a step was not included or was done incorrectly. If you understand what you did in lab and why you did it for each step you are in good shape.

Sample questions. These are the kinds of things you should know. This is not a complete set of possible questions. It is offered simply to give you an idea of the type of question you may be presented with. Any material from the OWL assignments or from the experiment handouts, including assigned questions, is fair game for exam questions. Answers to these sample questions will not be given. It is best for you to search them out and answer them on your own.

### Distillation.

1.) Using a boiling point – composition curve for a mixture of two liquids, starting with a mixture that is 90% higher-boiling liquid/10% lower boiling liquid (or any other combination).

- How many simple distillations would it take to produce lower boiling liquid that was at least 95% (or any %) pure?

- What would be the purity of lower-boiling liquid distilled from a fractional distillation apparatus having three (or any number of) theoretical plates?

- What would be the purity of liquid obtained from one (or any number of) simple distillation(s)?

2.) For a mixture of two compounds at room temperature, the vapor above the liquid is (enriched in lower-boiling compound, enriched in higher-boiling compound, or the same?) compared to the composition of the liquid.

- 3.) Problem 2 at the boiling point of the solution?
- 4.) Draw a temperature (y) vs volume of distillate (x) plot for a good fractional distillation of a mixture of two compounds, BPs 65° and 105° in the ratio of 1:4 (or any BPs or ratios).
- 5.) Draw the structures of the compounds which you distilled, including the unknowns, in the distillation experiment.

Gas Chromatography (GC - this is covered in the Cyclohexene handout).

- 6.) Using the distillation curve (temp vs volume) from the distillation handout, draw the GC trace for a sample (one drop) taken at the 62 drop point in the fractional distillation. Be sure to label the Time = 0 point, which peak is which (relative retention times), and the relative sizes of the peaks, but do not worry about absolute retention times. For a sample taken at 68 drops (or at any point)? (In other words, what is the % composition CYC/TOL of these samples and how is this shown on the GC?)
- 7.) For Problem 6.), how would the GC trace change if the GC column temperature were increased? - decreased? If the carrier gas flow rate were decreased? – increased?

Cyclohexene.

- 8.) What was the purpose of the phosphoric acid? - of the saturated sodium chloride? - of the calcium chloride? - of the toluene?
- 9.) What would have resulted if toluene had not been added? what would result if the distillation were carried out too rapidly?
- 10.) What is the structure of the intermediate of highest energy in the reaction? - of lowest energy?
- 11.) When mixing toluene, cyclohexene, and water, what is primarily in the upper layer? - in the lower layer?
- 12.) The GC of the final product showed a small peak with a shorter retention time than that of cyclohexene. What might this be? (hint: what other low-boiling compounds might you have inadvertently introduced?)
- 13.) Starting with 2.20 g of cyclohexanol, 1.25 g of distillate was obtained. GC analysis showed the distillate to be 89% cyclohexene. Calculate the % yield of cyclohexene. Do this for any amounts.
14. When a drop of a dilute bromine solution was added to an unknown sample, the bromine color disappeared. What could you conclude? What reaction, if any, occurred? Draw structures. What if the resulting solution was reddish in color?
- 15.) Same as 14.) but using potassium permanganate.

### Melting points.

- 16.) What two pieces of information can be obtained from a MP (range)?
- 17.) If a MP is found to be 112-116°, what can be concluded about the sample? What might the actual MP of that pure compound be?
- 18.) A sample melts at 95-96°. What can be concluded about that sample? If a different compound with the same MP is added to this sample, what observation might be expected when a MP of the mixture was taken?

### Solubilities and recrystallization.

- 19.) Using a graph show the three different solubility behaviors of solvents and note which is best for a recrystallization solvent.
- 20.) In a recrystallization how much solvent should be used? What if too much is used? What if the solution is not kept at the BP? For the rinse step what if too much solvent is used or what if the solvent is not ice cold?
- 21.) To describe the structure of a molecule what kinds of characteristics must be included?
- 22.) What does “Structure determines property.” mean? Name some properties from each of the three general types of properties.
- 23.) What are the steps in carrying out a recrystallization? What happens at each step and what can go wrong at each step?
- 24.) After dissolving the solid at the BP, why not save time and simply place the tube directly into an ice bath and stir it?
- 25.) Is it possible, if a recrystallization is done perfectly and not counting the small amount of impurities, to receive a 100% recovery of product? Why?
- 26.) Many times, even using the correct solvent and the correct amount of solvent, upon cooling no crystals form. In such a case usually if a glass stirring rod is used to gently scratch the surface of the tube beneath the surface of liquid, crystals will form. The same could be accomplished by adding a tiny crystal of solid. What’s the story?

### Extraction

- 27.) Which layer is which, what chemical species (structure) is in each layer at each stage, what acid-base reactions are going on at each step, how are solubilities manipulated, how this helps to accomplish a separation, what is the purpose of adding CaCl<sub>2</sub>, saturated salt?
- 28.) Calc’s for neutralization of acids and bases, molarity.

## Alcohols, Aldehydes, and Ketones

29.) What observations are made and what conclusions can be drawn in each test, what structural information does each test give, what are the reagents used and names of the tests, how does an impurity affect a MP and how might this affect your choice of possible compounds from the table.

30.) Simple NMR interpretation of an alc, ald, or ketone. Know basic chemical shift relationships, and simple splitting patterns.

## Trimyristin

31.) What is purpose of each step, each chemical reagent used?

32.) What reactions occur? Structures.

## Esters

33.) How to calculate mL, g from mol given density, MW (e.g., calculate the number of mL of X needed to make Y mol of ester, just as in your experiment)

34.) What is purpose of each step, each chemical reagent used, how is equilibrium driven towards product, what results if equilibrium were not driven. How are excess starting materials removed.

35.) IR of compounds involved in the ester experiment. Know basic frequencies important to this exp.

36.) Balanced reactions, calc of theoretical and actual yields.

## TLC.

38.) If you are analyzing relatively polar compounds using a silica gel plate and you find that the  $R_f$ 's are too high (e.g., 0.9), what would you do? - if they are too low (e.g., 0.1)?

39.) If you observe three spots after developing a TLC plate can you conclude that there are just three components in the mixture? Why?

40.) What would be the result if a development solvent of too low a polarity were used? Too high a polarity?

If you've gotten this far, congratulations. You should be in good shape if you understand and can answer all of these questions. *Caveat:* These questions cover a lot and show you the kinds of questions that you may see but do not necessarily cover all possible questions.