Ch 337M - Final Exam **Name**_____
Winter, 2000 **Lab day (circle): M/W pm T/Th pm T/Th night**
115 pts

1. (24) In this course you carried out the experiments listed, and were introduced to all of the standard laboratory techniques shown below. For each technique, indicate the experiment in which it was used, and state briefly what you were trying to accomplish in using the technique.

1. Melting points
2. Recrystallization
3. Fractional distillation
4. Eugenol from cloves
5. Caffeine from tea
6. Cyclohexene
7. Grignard reaction

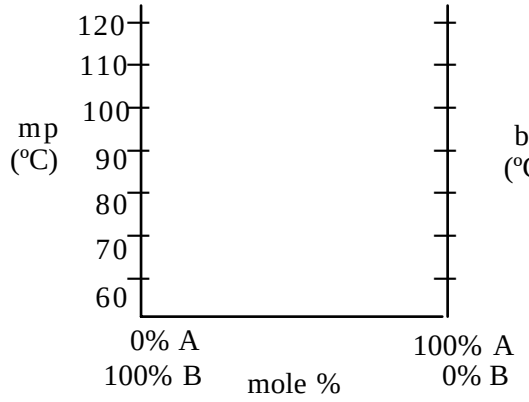
Technique	Experiment Number(s)	Purpose (brief = concise, using few words)
melting points		
mixture melting point		
recrystallization		
simple distillation		
fractional distillation		
steam distillation		
solid / liquid extraction		
liquid / liquid extraction		
chemically active extraction		
sublimation		
use of a drying agent		
gas chromatography		
refractive index		
infrared spectroscopy		
solvent evaporation		
use of a chaser solvent		

2. (6) a) Briefly explain how a drying agent, such as anhydrous $MgSO_4$, works (include appropriate equations).

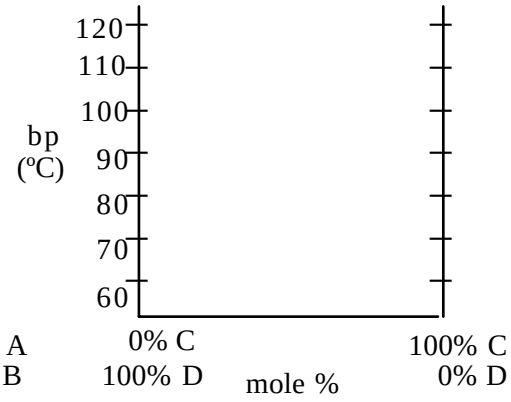
b) Students in the lab occasionally neglect to replace the lid on the bottle of $MgSO_4$. What problems might this cause?

3. (9) In space a), sketch the approximate appearance of a graph of <u>melting point</u> versus composition for a mixture of two solids, one of which (A) melts at 80ºC, and the other (B) at 120ªC. In space b), do the same for <u>boiling point</u> versus composition for an 'ideal' mixture of miscible liquids, one of which (C) boils at 80ºC, and the other (D) at 120ºC.. In space c) do the same as in b), except assume that C and D form an azeotropic mixture.

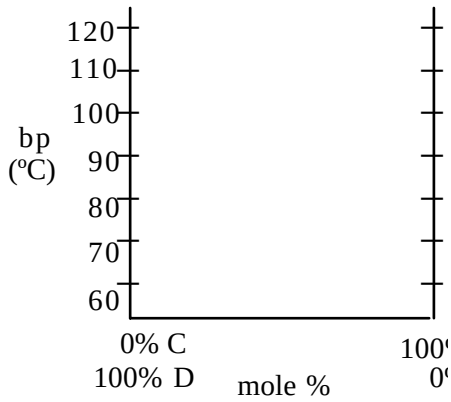
a) mp versus composition



b) bp versus composition (ideal)



b) bp versus composition (azeotrope)



4. (7) Listed below are solubility versus temperature data for an organic substance, A, in water.

a) Suppose that 2.5 g of A is mixed with 20 mL of water and slowly heated with stirring. At what temperature (approximate) would all of the sample dissolve?

<u>temp. (ºC)</u>	<u>solubility (g / 100 mL H_2O)</u>
0	1.5
20	3.0
40	6.5
60	11.0
80	17.0

b) If the heated solution from part a) is cooled to 0ºC, how many grams of A would crystallize? Show your work.

5. (12) Assume a mixture of liquids at 90ºC containing 20 mole % A and 80 mole % B. At this temperature, pure A and pure B have the following vapor pressures: P_A^o = 482 torr, P_B^o = 75 torr. In space a), calculate the indicated pressures above the liquid, assuming that A and B are miscible and exhibit ideal behavior. In space b), calculate the same quantities assuming that A and B are immiscible. Show your work.

	a) <u>miscible</u>	b) <u>immiscible</u>
Partial pressure of A	_____	_____
Partial pressure of B	_____	_____
Total pressure of A and B	_____	_____

c) Calculate the mole percent composition of the vapor above the liquid under the conditions of part a) as well as part b) Show your work.

<u>miscible</u> <u>immiscible</u>

6. (6) A mixture containing 50% benzene (bp 81ºC) and 50% ethylbenzene (bp 138ºC) is fractionally distilled.

a) Indicate the approximate temperatures at each of the following places after the first few drops of distillate have been collected.

1. Pot_____

2. Halfway up the column_____

3. Head_____

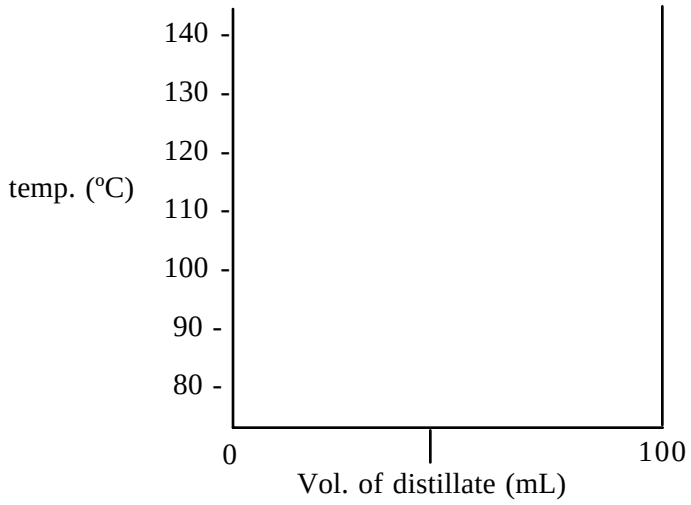
b) Indicate the approximate percent composition of the vapor at each of these places.

1. Pot_____

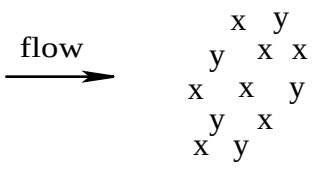
2. Halfway up the column_____

3. Head_____

7. (7) Sketch the expected distillation curve for the above fractional distillation. Assume an original sample volume of 100 mL. On the same graph, sketch the approximate pot temperature during the distillation. Indicate which curve is which.



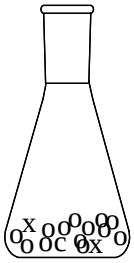
8. (7) In the space below, sketch the interior of a gas chromatograph column during analysis of a mixture of x and y, and explain briefly how a separation of mixtures takes place. Label the things that are inside the column.



gas chromatograph column

9. (5) Modern gas chromatographs use electronic integrators for determining relative areas of peaks. Assume that you have analyzed fraction # 1 from the distillation experiment, and the integrator registers the following relative values (units are arbitrary): acetone (from a contaminated; syringe) = 16, ethyl acetate = 488, toluene = 32. Calculate the percent of ethyl acetate in fraction # 1. Show your work.

10. (9) The drawing below represents an impure sample ready for recrystallization from ethanol (o = sample, x = soluble impurity, c = soluble colored impurity). Make drawings showing all of the steps in the recrystallization, including charcoal treatment, up to the point where the recrystallized sample is placed in a storage container. Indicate how you know what volume of ethanol to use, and show where the impurities end up.



11. (5) Lab instructors are generally very patient, but patience sometimes wears thin when the tenth person asks which is the aqueous layer and which is the organic layer in a separatory funnel. What simple and rapid test (less than a minute) can be used to distinguish the layers, using no more than a drop or two of each layer? Explain what you would do, observe, and conclude.

12. (5) A common laboratory reagent is concentrated nitric acid. This reagent has a density of 1.41 g / mL, and contains 71% HNO_3 by weight. The rest is water. How many milliliters of the concentrated reagent would you measure out in order to get 12.5 g of HNO_3 for a reaction? Show your work.

13. (5) In the preparation of triphenylmethanol by the Grignard method, suppose that you forgot to add the benzophenone before acidification with dilute HCl. What would happen to the Grignard reagent? Write equations for any reaction.

14. (8) Suppose that you set out to use the Grignard method for preparing 3-ethyl-3-pentanol, which is a liquid at room temperature and has a boiling point of 141ºC.

a) Write balanced equations for preparing this alcohol using the Grignard method.

b) In the container below, sketch the appearance of the reaction mixture at the beginning of the work-up stage. Describe briefly the work-up procedure as well as the final purification procedure that you would use for this synthesis. (The final purification technique appropriate for a substance having the physical property given above is _____.)