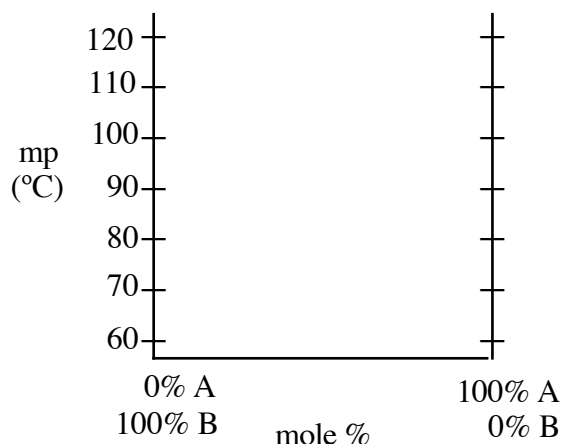


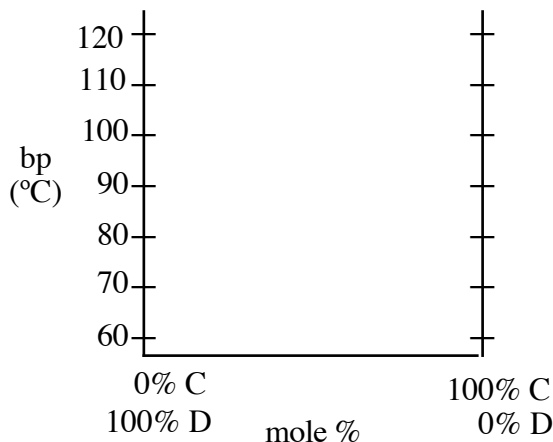
100 points

1. (6) In space a) sketch the approximate appearance of the graph of melting point versus composition for a mixture of two solids, one of which (A) melts at 80°C and the other (B) at 120°C. In space b), do the same for boiling point versus composition for an 'ideal' mixture of two liquids, one of which (C) boils at 80°C and the other (D) at 120°C.

a) mp versus composition



b) bp versus composition



2. (5) It is well known to auto mechanics that a mixture of water and antifreeze in a car radiator gives better protection from cold weather than pure antifreeze by itself. Explain why this is true, relating your answer to one of the lab experiments. (Pure water freezes and melts at 0°C, and pure antifreeze freezes and melts at -13°C.)

3. (5) You have discovered an unlabeled solid sample in your lab drawer that you think is adipic acid, but you're not sure. You find that it has mp 149-151°C. There is a bottle of pure adipic acid on the side shelf in the lab. What simple test should you perform to determine whether or not your sample is really adipic acid? What would you do, what might you see, and what would it mean?

4. (5) Simple distillation involves only a single vaporization/condensation stage, whereas fractional distillation involves multiple, successive vaporization/condensation stages. Briefly explain why these multiple stages can lead to a separation of two liquids. As an example, start with a 50/50 mixture of ethyl acetate (EA, bp 77°C) and toluene (TOL, bp 110°C).

5. (5) In the space at the right, make a simple sketch of a fractional distillation apparatus, and indicate where the multiple vaporization/condensation stages occur.

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

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

1. Pot \_\_\_\_\_

2. Half way up the column \_\_\_\_\_

3. Head \_\_\_\_\_

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

1. Pot \_\_\_\_\_

2. Half way up the column \_\_\_\_\_

3. Head \_\_\_\_\_

7. (8) In an ordinary recrystallization: a) At what point are soluble impurities removed?

b) At what point are insoluble impurities removed?

c) What would be the specific consequence of using too much solvent?

d) The recrystallized mixture is normally chilled in ice. How might chilling in ice actually lead to a less pure recrystallized sample?

8. Listed below are solubility data for an organic substance, A, in water.

a) (3) Suppose 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 just dissolve?

<u>temp (°C)</u>	<u>solubility (g/100 mL H<sub>2</sub>O)</u>
0	1.5
20	3.0
40	6.5
60	11.0
80	17.0

b) (4) If the heated solution from a) is cooled to 0°C, how many grams of A would crystallize? (Show your work.)

9. (9) You have just finished analyzing a sample of toluene using gas chromatography, and found that the retention time was 3.4 min. Indicate how the retention time of the toluene would be affected if you made the following changes in the conditions for analysis. Use the symbols A, B, C, and D as defined below.

A = no substantial change                      C = decreased retention time  
B = increased retention time                      D = could go either way

Increasing the column temperature \_\_\_\_\_

Using a different color ink in the recorder pen \_\_\_\_\_

Using a longer column \_\_\_\_\_

Decreasing the rate of gas flow \_\_\_\_\_

Increasing the chart speed on the recorder \_\_\_\_\_

Using a syringe contaminated with a little acetone \_\_\_\_\_

Using a column with a different packing material \_\_\_\_\_

Decreasing the detector sensitivity \_\_\_\_\_

Using a larger sample for injection \_\_\_\_\_

10. (5) Briefly explain what goes on inside the column of a gas chromatograph that results in the separation of a mixture.

11. (5) Modern gas chromatographs use electronic integrators for determining the relative peak areas. Assume that you have analyzed fraction # 1 from the distillation experiment, and the integrator registers the following relative areas (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.)

12. (4) In the citral experiment, you used solid anhydrous calcium chloride to remove traces of water from the ether solution. Explain how this drying agent works, including any chemical reaction.

13. a) (6) With words and diagrams, describe the steps that were involved in the isolation of pure caffeine from tea leaves. Include sketches of the equipment used.

b) (2) Why was sodium carbonate used in the caffeine experiment?

c) (2) Why was the melting point determined using a sealed capillary?

14. (20) Listed below are examples of poor lab technique. Briefly indicate what undesirable consequences might result. Be specific.

a) Forgetting to crush the crystals before determining the melting point \_\_\_\_\_

\_\_\_\_\_

b) Carrying out the hot filtration with a funnel having a 4-inch stem \_\_\_\_\_

\_\_\_\_\_

c) Forgetting to chill the recrystallized sample before suction filtration \_\_\_\_\_

\_\_\_\_\_

d) Determining the melting point of the recrystallized adipic acid 5 minutes after suction filtration \_\_\_\_\_

\_\_\_\_\_

e) Adding a boiling chip to a hot liquid \_\_\_\_\_

\_\_\_\_\_

f) In a distillation set-up, having the heating mantle resting directly on the desk top \_\_\_\_\_

\_\_\_\_\_

g) Distilling rather rapidly during the fractional distillation \_\_\_\_\_

\_\_\_\_\_

h) Heating the flask to dryness during a distillation \_\_\_\_\_

\_\_\_\_\_

i) Using acetone instead of dichloromethane in the caffeine experiment \_\_\_\_\_

\_\_\_\_\_

j) Forgetting to remove the stopper from the separatory funnel before draining the bottom layer

\_\_\_\_\_

\_\_\_\_\_