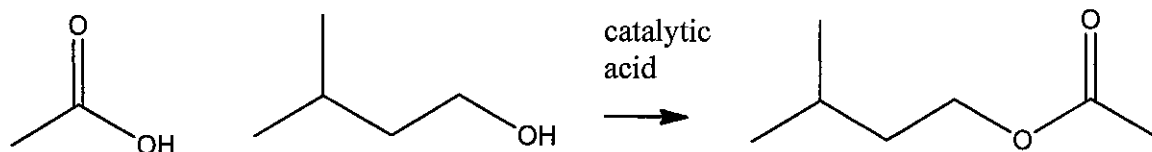




Name Key - multiple choice has version 1 and version 2  
AI (or Lab Section) \_\_\_\_\_

1. (10pts) Fred Chemist attempted to make isoamyl acetate (bp 142 °C), which is an artificial banana flavor, by mixing 10.0 g isoamyl alcohol (bp 131 °C) into excess acetic acid (bp 118 °C).



After the reaction was complete, the solution was poured into ether and washed with an aqueous solution in a separatory funnel. The ether layer was separated, dried, and evaporated. The resulting impure product was distilled, and the fractions boiling between 135-142 °C were collected. The product was analyzed by TLC (1:5 hexanes:ethyl acetate) and one spot was observed ( $R_f = 0.95$ ). In this TLC solvent, isoamyl alcohol has an  $R_f = 0.62$ .

+3

A. In the washing step, should Fred have used acid, base, or neutral water, or didn't it matter? Explain.

(+3) It doesn't matter - the impurity (H<sub>2</sub>O) is water soluble  
OR

(+2) Base - The impurity is an acid, so base extraction makes it more soluble

+3

B. What is one advantage of simple distillation? What is one advantage of fractional distillation? Which should be used in this experiment?

(+1) simple distillation - faster, easier, etc

(+1) fractional distillation - better separation

(+1) Fractional because of close b.p. OR simple because most impurities already removed

+2

C. Even though Fred only saw one spot on TLC, he was not happy with the result. Why not? What should he do to get a better analysis?

(+1)  $R_f$  is too high

(+1) use less polar TLC solvent

+2

D. Fred could have chosen to do the experiment by mixing 10 g of acetic acid with excess isoamyl alcohol, but the extraction/distillation procedure would probably not be as successful. Explain.

(+2) The extraction would not be effective in removing the excess alcohol from pdt - both are organic solvents  
CC(C)CO CC(C)CO

OR

(+1) The distillation would be harder because the excess alcohol has a bp closer to pdt (131 °C vs 142 °C)

For each of the questions below, circle the letters of ALL CORRECT answers. (2pts each)

2. If too much recrystallization solvent is used in a recrystallization:

+2

- A. Crystals will form too quickly, leading to impure product.
- B. The melting point of the purified substance will be broad and depressed.
- C. The compound will tend to "oil out."
- D. It is likely that little or no recrystallization will occur.

-1 for additional wrong answers

3. Column chromatography:

+2

- A. is highly effective in separating polar from nonpolar organic compounds.
- B. may be used to purify liquids, but not solids.
- C. is primarily used to analyze the polarity of a compound.
- D. is often used to identify an unknown compound.

-1 For additional wrong answers

4. A sample thought to contain compound A was analyzed by GC. The chromatogram showed peaks at 1, 2, and 3 minutes, with areas of 50, 60, and 90, respectively. Then authentic Compound A was mixed with the sample, and analyzed by GC. The same three peaks were observed, but this time with areas of approximately 10, 70, and 20, respectively. The percentage of compound A in the original sample is:

+2

- A. 25%
- B. 30%
- C. 50%
- D. 60%
- E. 70%

5. The retention time of a polar compound on GC can be decreased by

2

~~1~~

- A. increasing the flow rate.
- B. decreasing the sample size.
- C. changing the column material.
- D. decreasing column length.
- E. none of the above

-1 For any <sup>additional</sup> wrong answers or missing correct answers

6. The melting point of Compound A was determined to be 180-181 °C, Compound B was determined to be 165-166 °C, and Compound C was determined to be 163-181 °C. Based on these data, which of these are valid conclusions:

+2

- A. Compound A is more pure than Compound B.
- B. Compound A is more pure than Compound C.
- C. Sample C is a mixture of Compounds A and B.
- D. Compound B is an impure form of Compound A.
- E. None of the above

-1 for additional wrong answers

# Version 2

For each of the questions below, circle the letters of ALL CORRECT answers. (2pts each)

2. Column chromatography:

- +2  A. is highly effective in separating polar from nonpolar organic compounds.  
 B. may be used to purify liquids, but not solids.  
 C. is primarily used to analyze the polarity of a compound.  
 D. is often used to identify an unknown compound.

3. If too much recrystallization solvent is used in a recrystallization:

- A. Crystals will form too quickly, leading to impure product.  
 B. The melting point of the purified substance will be broad and depressed.  
 C. The compound will tend to "oil out."  
+2  D. It is likely that little or no recrystallization will occur.

4. A sample thought to contain compound A was analyzed by GC. The chromatogram showed peaks at 1, 2, and 3 minutes, with areas of 50, 60, and 90, respectively. Then authentic Compound A was mixed with the sample, and analyzed by GC. The same three peaks were observed, but this time with areas of approximately 10, 70, and 20, respectively. The percentage of compound A in the original sample is:

- A. 70%  
 B. 60%  
 C. 50%  
+2  D. 30%  
 E. 25%

5. The retention time of a polar compound on GC can be decreased by

- A. changing the column material.  
 B. decreasing column length.  
+2  C. increasing the flow rate.  
 D. decreasing the sample size.  
 E. none of the above

-1 For additional wrong answers  
or  
any missing answers

6. The melting point of Compound A was determined to be 180-181 °C, Compound B was determined to be 165-166 °C, and Compound C was determined to be 163-181 °C. Based on these data, which of these are valid conclusions:

- A. Sample C is a mixture of Compounds A and B.  
 B. Compound B is an impure form of Compound A.  
 C. Compound A is more pure than Compound B.  
+1  D. Compound A is more pure than Compound C.  
 E. None of the above

For each question, subtract one for additional wrong answers