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Section 3.2  RECRYSTALLIZATION. PART A. SOLVENT SELECTION

NAME (print): __________________________ DATE: __________

INSTRUCTOR: __________________________ LABORATORY SECTION: __________

1. By marking yes (Y) or no (N) in the space provided, specify which of the following criteria are met by a good solvent for a recrystallization.

   a. The solutes are soluble in the cold solvent. ______
   b. The solvent does not react chemically with the solutes. ______
   c. The solvent is polar rather than nonpolar. ______
   d. The boiling point of the solvent is above 100 °C (760 torr). ______
   e. The boiling point of the solvent preferably is below the melting point of the solute. ______

2. Specify the criterion applied in this experimental procedure to classify a solute as being “soluble” in a particular solvent.

3. Review the functional groups present in resorcinol, benzoic acid, naphthalene, and acetanilide. Predict whether these molecules are expected to be polar (P) or nonpolar (NP).

   a. Resorcinol ______
   b. Benzoic acid ______
   c. Naphthalene ______
   d. Acetanilide ______

4. Why is it important to

   a. avoid inhaling vapors of organic solvents?

   b. know the location and operating instructions of the nearest fire extinguisher when using 95% ethanol or petroleum ether for testing solubilities?

5. The flash points of 95% ethanol and petroleum ether (bp 60–80 °C) are, respectively, ______ and _____.

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6. Specify what you should do if each of the following gets on your skin.

   a. Benzoic acid

   b. Resorcinol

   c. Acetanilide

7. What action should you take if resorcinol gets in your eyes?
Section 3.2  RECRYSTALLIZATION. PART B. RECRYSTALLIZING IMPURE SOLIDS (Miniscale)

NAME (print): ____________________________ DATE: _________________
INSTRUCTOR: ____________________________ LABORATORY SECTION: ______________

1. By marking gravity (G) or vacuum (V) in the space provided, indicate which of the two different filtering techniques is more suitable for each of the following operations.
   a. Hot filtration. _____
   b. Removing decolorizing carbon. _____
   c. Isolating recrystallized solute from solution. _____

2. Why is flameless heating used for heating a solution in hexane or diethyl ether during a recrystallization?

3. Why should the size of crystals obtained in a recrystallization be neither too large nor too small?

4. What is the process of seeding, as it applies to recrystallization? What purpose does it serve?

5. What is meant by the term, “oiling out,” as it applies to crystallizations?
6. How is the purity of a recrystallized solid assessed?

7. Why should decolorizing carbon not be added to a solvent that is at or near its boiling point?

8. Why is it important to
   a. break (terminate) the vacuum before turning off the water aspirator pump when employing the equipment for vacuum filtration shown in Figure 2.52?
   b. avoid inhaling vapors of organic solvents?
   c. know the position and operating instructions of the nearest fire extinguisher when using methanol, 95% ethanol, or 2-propanol as a crystallization solvent?
   d. use a fluted filter paper for hot filtration?

9. What action should you take if benzoic acid gets on your skin?
Section 3.2  RECRYSTALLIZATION. PART B. RECRYSTALLIZING IMPURE SOLIDS (Microscale)

NAME (print): _____________________________ DATE: __________________

INSTRUCTOR: _________________________ LABORATORY SECTION: _____________

1. By marking gravity (G) or Craig tube (CT) in the space provided, indicate which of the two different filtering techniques is more suitable for each of the following operations.
   
   a. Hot filtration. _____
   b. Removing decolorizing carbon. _____
   c. Isolating recrystallized solute from solution. _____

2. Why is flameless heating used for heating a solution in hexane or diethyl ether during a recrystallization?

3. Why should the size of crystals obtained in a recrystallization be neither too large nor too small?

4. What is the process of seeding, as it applies to recrystallization? What purpose does it serve?

5. What is meant by the term, “oiling out,” as it applies to crystallizations?
6. How is the purity of a recrystallized solid assessed?

7. Why should decolorizing carbon not be added to a solvent that is at or near its boiling point?

8. Why is it important to
   a. ensure that the contents of the centrifuge are balanced before turning on this device.
   b. avoid inhaling vapors of organic solvents?
   c. know the position and operating instructions of the nearest fire extinguisher when using methanol, 95% ethanol, or 2-propanol as a crystallization solvent?
   d. use a pre-heated pipet for hot filtration?

9. What action should you take if benzoic acid gets on your skin?
Section 3.3  MELTING POINTS. Parts A/B. CALIBRATION OF THERMOMETER AND DETERMINING CAPILLARY-TUBE MELTING POINTS

NAME (print): ___________________________ DATE: __________________
INSTRUCTOR: __________________________ LABORATORY SECTION: _____________

1. List a convenient source of data concerning the physical constants and properties of organic compounds.

2. Indicate which of the following statements is true (T) and which is false (F).
   
   a. An impurity raises the melting point of an organic compound. _____
   b. A eutectic mixture has a sharp melting point, just as does a pure compound. _____
   c. If the rate of heating of the oil bath used in a melting-point determination is too high, the melting point that results will likely be too low. _____
   d. The sample should not be packed tightly into a capillary melting-point tube. _____
   e. A heating bath containing mineral oil should not be used to determine the melting points of solids melting above 200 °C. _____

3. On the figure below, sketch the location of the capillary melting-point tube and the sample contained in it relative to the bulb of the thermometer.

4. What is the approximate rate at which the temperature of the heating bath should be increasing at the time the sample undergoes melting?
5. What is the preferred technique for accurately determining the melting point of an unknown compound in a minimum length of time?

6. How does measuring a mixed melting point help in determining the possible identity of two solid samples?

7. Briefly describe the technique for packing a capillary melting-point tube.

8. Why is it important to calibrate a thermometer with a set of standards having a range of melting points?

9. What action should you take if salicylic acid gets in your eyes?
Section 3.3 MELTING POINTS. PART C. WHO ELSE HAS MY COMPOUND?

NAME (print): ____________________________ DATE: ____________________
INSTRUCTOR: ____________________________ LABORATORY SECTION: ____________

1. Describe how to calculate $R_f$ from a developed TLC plate.

2. How does increasing the polarity of the developing solvent affect the $R_f$ of a compound on silica gel TLC plates?

3. What is the purpose of a co-spot in TLC analysis?

4. For a pure sample, how do you know if you heated the sample too rapidly when determining a melting point?

5. What effect does impurity have on the melting point of a compound?

6. What can you infer about a compound's structure if it is soluble in 2.5 M aqueous sodium hydroxide but not in water?
7. Compound A has an \( R_f = 0.35 \) and compound B has an \( R_f = 0.64 \) when spotted on a silica gel TLC plate and developed with hexane. Which compound is more polar?

8. Why should the solvent level in the developing chamber be lower than spot on the undeveloped TLC plate?

9. What should you do if you get 2.5 \( M \) aqueous sodium hydroxide on your skin?

10. Rank the following solvents in order of increasing polarity: acetone, hexane, methanol, ethyl acetate, dichloromethane.

11. Rank the following functional groups in order of increasing polarity: carboxylic acid, ether, ketone, alkene, alcohol.

12. How could you tell if too much sample was spotted on a TLC plate?
Section 4.1  BOILING POINTS OF PURE LIQUIDS (Miniscale)

NAME (print): _____________________________ DATE: ________________

INSTRUCTOR: ______________________ LABORATORY SECTION: __________

Questions 1, 2, and 4 may be answered by marking yes (Y) or no (N) in the space provided for each part.

1. Specify whether the addition of a nonvolatile solute to a volatile liquid
   a. has no effect on the boiling point of the volatile liquid. _____
   b. lowers the boiling point of the volatile liquid. _____
   c. raises the boiling point of the volatile liquid. _____

2. Specify whether the boiling point of a pure liquid
   a. is the same in Denver, Colorado (elevation 5280 ft) as it is in San Francisco, California. _____
   b. is lower in Denver than in San Francisco. _____
   c. is usually found to be almost the same as the reported “standard boiling temperature” in San Francisco. _____

3. Explain your answers to Exercise 2.

4. Specify whether the boiling point, as determined in the miniscale boiling-point apparatus, is the temperature
   a. of the liquid at the time bubbles first emerge slowly from the liquid.
   b. at the vapor-liquid interface above the surface of the boiling liquid while a drop of liquid is suspended from the thermometer.
   c. of the liquid at the time bubbles emerge rapidly from the liquid.
   d. of the heating source at the time bubbles emerge rapidly from the liquid.
5. Why is heating a liquid in a closed system dangerous?

6. What precautions should be taken before lighting a Bunsen burner or microburner in the laboratory?

7. Why is mineral oil an inappropriate heating fluid for determining the boiling points of samples that exceed 200 °C (760 torr)?

8. What action should you take if an organic liquid used in this experiment gets on your skin?
Section 4.2  BOILING POINTS OF PURE LIQUIDS (Microscale)

NAME (print): ___________________________ DATE: __________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: _____________

Questions 1, 2, and 4 may be answered by marking yes (Y) or no (N) in the space provided for each part.

1. The addition of a nonvolatile solute to a volatile liquid
   a. has no effect on the boiling point of the volatile liquid. _____
   b. lowers the boiling point of the volatile liquid. _____
   c. raises the boiling point of the volatile liquid. _____

2. The boiling point of a pure liquid
   a. is the same in Denver, Colorado (elevation 5280 ft) as it is in San Francisco, California. _____
   b. is lower in Denver than in San Francisco. _____
   c. is usually found to be almost the same as the reported “standard boiling temperature” in San Francisco. _____

3. Explain your answers to Exercise 2.

4. The boiling point, as determined in the micro boiling-point apparatus, is the temperature at the time
   a. bubbles first emerge slowly from the inverted capillary tube.
   b. bubbles begin to emerge rapidly from the inverted capillary tube.
   c. the liquid begins to re-enter and rise in the inverted capillary tube.

5. What are the bubbles that emerge slowly from the inverted capillary tube before the boiling temperature is reached, and why does this occur?
6. Define a *closed system* as it applies to a Thiele tube and the liquids it contains.

7. Why should a closed system, as defined in Exercise 6, *not* be heated unless special apparatus is available?

8. Why is the micro boiling-point apparatus used in this procedure *not* a closed system?

9. Why is mineral oil an inappropriate heating fluid for determining the boiling points of samples that exceed 200 °C (760 torr)?

10. What precautions should be taken *before* lighting a Bunsen burner or microburner in the laboratory?

11. What action should you take if an organic liquid used in this experiment gets on your skin?
2. By marking simple (S) or fractional (F) in the space provided, indicate which of the two distillation techniques would be more suitable for the following.
   a. preparing drinking water from sea water. _____
   b. removing diethyl ether, bp 35 °C (760 torr), from a solution containing p-dichlorobenzene, bp 174 °C (760 torr). _____
   c. separating benzene, bp 80 °C (760 torr), from toluene, bp 111 °C (760 torr). _____

3. What is the purpose of the stirbar placed in the stillpot?

4. How does the composition of the liquid at the top of a fractional distillation column compare with the composition of the liquid at the bottom of a column? (Answer in terms of the relative amounts of lower-boiling and higher-boiling components.)

5. Two fractionating columns are each 40 cm in length. Column A has HETP = 2 cm and column B has HETP = 20 cm. By marking in the space provided, indicate whether column A or B would be more suitable to separate a binary mixture in which the components differ in boiling point by 10 °C?
   _____

6. Define the term, reflux ratio.
7. Why is it important to align the fractionating column as nearly vertical as possible?

8. On the figure below, sketch the correct location of the thermometer bulb during a miniscale distillation.

8. Why is it important that a drop of condensate be suspended from the thermometer during a distillation?

9. With respect to the condenser used in an apparatus for simple or fractional distillation, why should the lower rather than the upper nipple be used for the water inlet?

10. The flash point (°C) of cyclohexane is ______; that of toluene is ______.

11. List possible effects of ingesting cyclohexane or toluene.
Section 4.3 SIMPLE DISTILLATION (Microscale)

NAME (print): ___________________________ DATE: ________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. Indicate whether each of the following statements is true (T) or false (F).
   
a. Simple distillation may be used to prepare drinking water from sea water. _____
   
b. Simple distillation may be used to remove diethyl ether, bp 35 °C (760 torr), from p-

dichlorobenzene, bp 174 °C (760 torr). _____
   
d. Simple distillation may be used to separate benzene, bp 80 °C (760 torr), from toluene,
  
e. bp 111 °C (760 torr). _____

2. What is the purpose of the spinvane placed in the stillpot?

3. On the figure below, sketch the correct location of the thermometer bulb during a microscale, simple distillation.

4. Why is it important that a drop of condensate be suspended from the thermometer during a distillation?
5. With respect to the condenser used in an apparatus for simple distillation, why should the lower rather than the upper nipple be used for the water inlet?

6. The flash point (°C) of cyclohexane is ______.

7. List the possible effects of ingesting cyclohexane.

8. What action should you take if cyclohexane gets in your eyes?
Section 4.6 STEAM DISTILLATION OF CITRAL FROM LEMON GRASS OIL

Questions 1 and 3 may be answered by marking (T) or (F) in the space provided for each part to indicate whether the statement is true or false.

0. Steam distillation would be the procedure of choice for separating mixtures of
   a. methanol, bp 65 °C (760 torr), and water; methanol is completely miscible with water. _____
   b. $p$-dichlorobenzene, bp 174 °C (760 torr), and water; $p$-dichlorobenzene is insoluble in water. _____
   c. Ethylene glycol (HOCH$_2$CH$_2$OH), bp 196 °C (760 torr), and water; the glycol is miscible with water in all proportions. _____

2. Explain your answer to each part of Exercise 1.
   a. 

   b. 

   c. 

   • Indicate whether the following statement is true (T) or false (F): The boiling point during a steam distillation is always less than 100 °C.
     _____

3. Explain your answer to Exercise 3.
4. In what way are the advantages of steam distillation and vacuum distillation similar?

5. What kind of mixture may better be separated by steam distillation than by vacuum distillation?

6. Write the structural formula of the thermodynamically more stable of the two diastereomers (geometrical isomers) of citral.

7. Based on a consideration of the nature of the groups present in the diastereomers that comprise citral, why is citral insoluble in water?

8. Why is a flame not used to assist in the removal of diethyl ether from citral?

9. Why should ethereal solutions not be stored in your laboratory locker from one period to the next?

10. What action should you take if citral gets on your skin?
Section 5.3  BASE AND ACID EXTRACTIONS. PART A. ONE-BASE EXTRACTION

NAME (print): ___________________________  DATE: __________________
INSTRUCTOR: ___________________________  LABORATORY SECTION: __________

1. Write balanced equations for all chemical reactions that should occur in the extraction procedure you are to perform.

2. Devise a flow chart for purification that summarizes the separations to be performed in this experiment. Consult Figure 1.2 for an example of such a flow chart.
3. When extracting an aqueous solution with an organic solvent, if you are uncertain as to which layer is aqueous, how could you settle the issue?

4. Which layer, upper (U) or lower (L), will the following solvents usually form when used to extract dilute aqueous solutions: diethyl ether _______, dichloromethane _______, chloroform ______, hexane ______?

5. Calculate the amount of acid needed to neutralize the basic extract in the experiment you are to perform. Show your work.

6. Indicate which of the following statements is true (T) and which is false (F).
   a. Benzoic acid forms a water-soluble salt, whereas naphthalene does not. ______
   b. Carboxylic acids containing six or more carbon atoms per molecule are more soluble in diethyl ether than in water. ______
   c. Carboxylic acids containing six or more carbon atoms per molecule are more soluble in ______
   d. Carboxylic acids containing six or more carbon atoms per molecule are more soluble in ______
   e. 2.5 M sodium hydroxide than in diethyl ether. ______
   f. Naphthalene is more soluble in diethyl ether than is sodium benzoate. ______
   g. As a general rule, aqueous sodium bicarbonate is preferred to aqueous sodium hydroxide for abstracting acidic compounds from organic solutions. ______
   h. A criterion for a dry organic solution is that the solution is not cloudy. ______
   i. Drying agents need not be removed prior to removing solvents when isolating products. ______

7. Indicate the materials that are appropriate to use for extinguishing a fire involving:
   a. benzoic acid
   b. naphthalene

8. Why is frequent venting necessary when extracting the diethyl ether solution with aqueous base?
1. Write balanced equations for all chemical reactions that should occur in the extraction procedure you are to perform.

2. Devise a flow chart for purification that summarizes the separations to be performed in this experiment. Consult Figure 1.2 for an example of such a flow chart.
3. If you are uncertain as to which layer is aqueous when extracting an aqueous solution with an organic solvent, how could you settle the issue?

4. Which layer, upper (U) or lower (L), will the following solvents usually form when used to extract dilute aqueous solutions: diethyl ether _____, dichloromethane _____, chloroform _____, hexane _____?

5. Calculate the amount of acid needed to neutralize the basic extracts obtained in this experiment. Show your work.

6. Indicate which of the following statements is true (T) and which is false (F).
   a. Benzoic acid forms a water-soluble salt, whereas naphthalene does not. _____
   b. Carboxylic acids containing six or more carbon atoms per molecule are more soluble in diethyl ether than in water. _____
   c. Phenols containing six or more carbon atoms per molecule are more soluble in 2.5 \( M \) sodium hydroxide than in diethyl ether. _____
   d. Naphthalene is more soluble in diethyl ether than is sodium phenoxide. _____
   e. As a general rule, aqueous sodium bicarbonate is preferred to aqueous sodium hydroxide for abstracting acidic compounds from organic solutions. _____
   f. A criterion for a dry organic solution is that the solution is not cloudy. _____
   g. Drying agents need not be removed prior to removing solvents when isolating products. _____

7. Specify the effects of the following:
   a. Inhalation of naphthalene.
   b. Exposure of your skin to 2-naphthol.

8. Why is frequent venting necessary when extracting the diethyl ether solution with aqueous base?
Section 5.3  BASE AND ACID EXTRACTIONS. PART C. ACID AND BASE EXTRACTION

NAME (print): ___________________________ DATE: ________________
INSTRUCTOR: ___________________________ LABORATORY SECTION: _____________

1. Write equations for all chemical reactions that occur in the extraction procedure.

2. Devise a flow chart for purification that summarizes the separations performed in this experiment.
3. If you are uncertain as to which layer in the separatory funnel is aqueous when extracting an aqueous solution with an organic solvent, how could you settle the issue?

4. Which layer, upper (U) or lower (L), will each of the following solvents usually form when used to extract a dilute aqueous solution? diethyl ether _____, dichloromethane _____, chloroform _____, hexane _____.

5. With respect to the neutralization step in the procedure, calculate the amount of base required to neutralize the acidic extract and the amount of acid needed to neutralize the basic extract.
   a. Base required:
   b. Acid required:

0. Indicate whether each of the following statements is true (T) or false (F).
   a. Benzoic acid and p-nitroaniline form water-soluble salts, whereas naphthalene does not. _____
   b. Carboxylic acids and phenols containing six or more carbon atoms per molecule are more soluble in dichloromethane than in water. _____
   c. Carboxylic acids containing six or more carbon atoms per molecule are more soluble in 3 M sodium hydroxide than in dichloromethane. _____
   d. Naphthalene is more soluble in dichloromethane than is sodium benzoate. _____

1. List possible effects of inhaling excessive amounts of dichloromethane.
Section 5.4  ISOLATION OF TRIMYRISTIN FROM NUTMEG (Miniscale)

NAME (print): ________________________________ DATE: ________________

INSTRUCTOR: _______________________________ LABORATORY SECTION: _______________

1. The technique responsible for isolation of trimyristin in this experiment is an example of (check one) liquid-liquid, ____, solid-liquid, ____, gas-liquid partitioning _____.

2. Indicate which of the following statements is true (T) and which is false (F).
   a. Pure trimyristin is a liquid at room temperature. _____
   b. According to the equation defining the distribution coefficient $K$, a value of 2 for $K$ means that $A$ is more soluble in solvent $S_o$ than in solvent $S_x$. _____
      \[ K = \frac{\text{grams of } A \text{ in } S_x}{\text{grams of } A \text{ in } S_o} \times \frac{\text{mL of } S_o}{\text{mL of } S_x} \]
   c. Nutmeg contains a complex mixture of organic compounds that are soluble in diethyl ether. _____
   d. Trimyristin is more soluble in acetone than in diethyl ether. _____
   e. The functional group present in trimyristin is a carboxylic acid group. _____
   f. The term *lipid* describes a category of organic compounds that are insoluble in water. _____
   g. With respect to the technique of extraction, the term *partitioning* means physical separation of two immiscible phases by an impermeable membrane. _____

3. Explain your answer to Exercises 2e and 2f.
   e. 
   f. 
4. Why is trimyristin considered a saturated fat?

5. What is wrong with the experimental set-up shown below for extraction of nutmeg?

![Experimental Set-up](image)

6. On the figure shown in Exercise 5, indicate on the condenser the upper limit for the ring of condensate and the points at which clamps should be located.

7. Why is a flame not to be used to heat diethyl ether at reflux in this experiment?

8. What would the consequence be of not having the condenser tightly connected to the round-bottom flask during the reflux period?

9. The flash point (°C) of diethyl ether is ____; that of acetone is ____.

10. List possible effects of inhaling excessive amounts of diethyl ether.
Section 5.4  ISOLATION OF TRIMYRISTIN FROM NUTMEG (Microscale)

1. The technique responsible for isolation of trimyristin in this experiment is an example of (check one) liquid-liquid, solid-liquid, gas-liquid partitioning.

2. Indicate which of the following statements is true (T) and which is false (F).
   a. Pure trimyristin is a liquid at room temperature.  
   b. According to the equation defining the distribution coefficient $K$, a value of 2 for $K$ means that $A$ is more soluble in solvent $S_o$ than in solvent $S_x$.  
   \[ K = \frac{\text{grams of } A \text{ in } S_x}{\text{grams of } A \text{ in } S_o} \times \frac{\text{mL of } S_o}{\text{mL of } S_x} \]
   c. Nutmeg contains a complex mixture of organic compounds that are soluble in diethyl ether.  
   d. Trimyristin is more soluble in acetone than in diethyl ether.  
   e. The functional group present in trimyristin is a carboxylic acid group.  
   f. The term lipid describes a category of organic compounds that are insoluble in water.  
   g. With respect to the technique of extraction, the term partitioning means physical separation of two immiscible phases by an impermeable membrane.

7. Explain your answer to Exercises 2e and 2f.
   e.
   f.
4. Why is trimyristin considered a saturated fat?

5. What is wrong with the experimental set-up shown below for extraction of nutmeg?

![Diagram of experimental setup]

6. On the figure shown in Exercise 5, indicate on the condenser the upper limit for the ring of condensate and the point at which a clamp should be located.

7. Why is a flame not to be used to heat diethyl ether at reflux in this experiment?

8. What would the consequence be of not having the condenser tightly connected to the conical vial during the reflux period?

9. The flash point (°C) of diethyl ether is _______; that of acetone is _______.

10. List possible effects of inhaling excessive amounts of diethyl ether.
Section 6.2  THIN-LAYER CHROMATOGRAPHY. PART A. SEPARATION OF SPINACH PIGMENTS

NAME (print):  ___________________________________________________________________ DATE: __________________

INSTRUCTOR: ___________________________________ LABORATORY SECTION: _________________

1. Describe three ways in which colorless compounds can be located on a TLC plate.

2. Check TLC (thin-layer chromatography) or CC (column chromatography) as the more appropriate answer to the following questions or statements.
   a. TLC _____ CC _____ is a quicker procedure for separating components of a mixture.
   b. In TLC _____ CC _____, the solvent front moves downward.
   c. TLC _____ CC _____ is better for separating a 5-gram mixture of components.
   d. TLC _____ CC _____ is better for separating a mixture of volatile compounds.

4. Why should a TLC plate be removed from the solvent before the solvent front reaches the top of the plate?

5. What is the purpose of shaking the petroleum ether-ethanol extract of the leaves with water in a separatory funnel?

6. The separating power, or activity, of a TLC plate is increased by heating the plate in an oven at 100 °C. Why? (Hint: See Section 6.2)
7. Why should the developing chamber for a TLC plate *not* be open to the atmosphere?

8. Which of the following diagrams illustrate(s) an *improper* way of spotting a TLC plate? Tell what is wrong in each case.

a. 

b. 

c. 

d. 

Liquid level
Section 6.2  THIN-LAYER CHROMATOGRAPHY. PART B. SEPARATION OF SYN- AND ANTI-AZOBENZENES BY TLC

NAME (print): ___________________________ DATE: ________________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. Describe three ways in which colorless compounds can be located on a TLC plate.

2. Check TLC (thin-layer chromatography) or CC (column chromatography) as the more appropriate answer to the following questions or statements.
   a. TLC _____ CC _____ is a quicker procedure for separating components of a mixture.
   b. In TLC _____ CC _____, the solvent front moves downward.
   c. TLC _____ CC _____ is better for separating a 5-gram mixture of components.
   d. TLC _____ CC _____ is better for separating a mixture of volatile compounds.

3. Why should a TLC plate be removed from the solvent before the solvent front reaches the top of the plate?

4. The separating power, or activity, of a TLC plate is increased by heating the plate in an oven at 100 °C. Why? *(Hint: See Section 6.2)*

5. Why should the developing chamber for a TLC plate not be open to the atmosphere?
6. Which of the following diagrams illustrate(s) an *improper* way of spotting a TLC plate? Tell what is wrong in each case.

![Diagram of TLC spotting](image)

a.  

b.  

c.  

d.  

7. Is azobenzene considered a possible carcinogen?

8. What is the density, $d$, of azobenzene?
Section 6.3 COLUMN CHROMATOGRAPHY

1. What difficulty may result if
   a. chromatographic column is not placed in a vertical position?
   b. the liquid level of the eluent is allowed to drop below the top of the column?

2. Petroleum ether followed by dichloromethane is used to separate fluorene and 9-fluorenone. Why would reversing the order in which these solvents are used be unwise?

3. What is the best experimental procedure to use in choosing an eluting solvent for column chromatography?

4. Define:
   a. eluant
   b. eluate
   c. adsorption
5. Which type of alumina, “acidic” or “basic,” would provide for the better separation of acids?

6. How can the adsorptivity (activity) of alumina be varied?

7. Why should a mixture to be separated be introduced onto a column in a minimum amount of solvent?

8. Why should no flames be allowed on the lab bench when performing this experiment?

9. Underline the media that are appropriate for extinguishing fires involving fluorene or 9-fluorenone:
   Water  Carbon dioxide  Chemical powder  Foam

10. Is fluorene a carcinogen? a mutagen?

11. Is 9-fluorenone a mutagen?
Section 6.4  GAS-LIQUID CHROMATOGRAPHY. PART A. QUALITATIVE AND QUANTITATIVE ANALYSIS OF A MIXTURE BY GLC

NAME (print): ________________________________  DATE: __________________
INSTRUCTOR: ________________________________  LABORATORY SECTION: ______________

1. Explain how liquids can be analyzed by gas chromatography.

2. Briefly define or describe the function, in gas chromatography, of the
   a. carrier gas
   b. stationary liquid phase
   c. solid support

3. Put a checkmark below the stationary phase that should be chosen to separate a mixture of

   Carbowax 20M      SE 30
   a. alcohols.      _______      _______
   b. aromatic hydrocarbons. _______      _______

4. If ethyl acetate and n-butyl acetate are analyzed by gas chromatography, which of these esters will generally produce a peak with the shorter retention time?
5. How could you confirm your answer to Exercise 4 by experiment?

6. What operating variables determine retention time?

7. Explain why it is important to inject the sample quickly.

8. The flash points (°C) of ethylbenzene, toluene, and isopropylbenzene, respectively, are ______, ______, and ______.

9. Underline the media that are appropriate for extinguishing fires involving the hydrocarbons listed in Exercise Water Carbon dioxide Chemical powder Foam
1. Explain why it is important to mix the solutions containing two components completely before the GLC analysis.

2. Typical standards include ethyl acetate, p-xylene and ethanol. The flash points (°C), respectively, of ethyl acetate, p-xylene, ethanol are ______, ______, and ______.

3. Explain why it is important to inject the sample quickly.

4. Indicate whether the following statement is true (T) or false (F): It is acceptable for the standard and the sample whose response factor is being determined to have the same retention time under the chromatographic conditions of the measurement.

5. Explain your answer to Exercise 4.
6. Indicate whether the following statement is true (T) or false (F): In order to obtain accurate values for the GLC response factor of the unknown sample, it is essential to use equal volumes of the standard and the sample whose response factor is being determined.

   

7. Explain your answer to Exercise 6.

8. Indicate whether the following statement is true (T) or false (F): To minimize the error in determining the GLC response factor of an unknown, the peaks on the chromatogram for both the standard and the unknown should be as large as possible.

9. Explain your answer to Exercise 8.
Section 7.2  SEPARATION OF DIASTEREOMERIC 1,2-CYCLOHEXANEDIOLS

1. Indicate which of the following statements is true (T) and which is false (F).
   
   a.  trans-1,2-cyclohexanediol is a meso form.  _____
   
   b.  The cis- and trans-1,2-cyclohexanediols are enantiomers.  _____
   
   c.  The cis- and trans-1,2-cyclohexanediols cannot be separated by fractional crystallization.  _____
   
   d.  (+)-trans-cyclohexane-1,2-diol and (-)-trans-cyclohexane-1,2-diol have different chromatographic adsorption properties.  _____

2. Why should a TLC plate be removed from the solvent before the solvent front reaches the top of the plate?

3. Why should the developing chamber for a TLC plate not be open to the atmosphere?

4. What physical properties could be used to distinguish cis- from trans-1,2-cyclohexanediol?
5. Why might water *not* be an appropriate extinguishing medium for burning petroleum ether?

6. Identify any of the following diagrams that illustrate an *improperly* spotted TLC plate and explain what is wrong in each such case.

![Diagram of TLC plates](image)

a. 

b. 

c. 

d. 

7. The flash points (°C) for petroleum ether (bp 60–80 °C), acetone, and 2-propanol are, respectively, ______, ______, and ______.

8. List three of the possible effects of inhaling excessive amounts of acetone.
Section 7.3  ISOMERIZATION OF DIMETHYL MALEATE TO DIMETHYL FUMARATE

1. Write structural formulas for dimethyl maleate and dimethyl fumarate.

8. Indicate which of the following statements is true (T) and which is false (F).
   a. Dimethyl maleate and dimethyl fumarate are enantiomers. _____
   b. Dimethyl maleate and dimethyl fumarate are diastereomers. _____
   c. Dimethyl maleate and dimethyl fumarate are conformational isomers. _____
   d. Dimethyl maleate and dimethyl fumarate are constitutional isomers. _____

9. Put a check mark beside the ester that
   a. has the higher melting point, dimethyl maleate _____ dimethyl fumarate _____?
   b. has the higher boiling point, dimethyl maleate _____ dimethyl fumarate _____?
   c. is more soluble in CH₂Cl₂, dimethyl maleate _____ dimethyl fumarate _____?

5. Why should glassware containing residues of bromine not be rinsed with acetone?

6. State the recommended procedure in this experiment to be used for destroying residual bromine and write the equation for the reaction that occurs.
7. What is to be done if the melting point of the isolated dimethyl fumarate is lower than the reported melting point of this compound?

8. What is the vapor pressure of bromine at 20 °C (760 torr)?

9. List possible effects of inhaling excessive amounts of bromine.

10. What action should you take if bromine gets on your skin?

11. Provide the flash points (°C) of dichloromethane _____ and of ethanol _____.
Section 7.4  PROPERTIES OF THE ENANTIOMERS OF CARVONE

NAME (print): __________________________________________ DATE: ______________

INSTRUCTOR: _____________________________ LABORATORY SECTION: ______________

1. Put a check mark beside any of the following physical properties that would be expected to be the same for the enantiomeric carvones.
   - boiling point
   - solubility in acetone
   - the R_f-value in TLC
   - odor
   - retention time in GLC
   - rotation of plane-polarized light

2. Indicate which of the following statements is true (T) and which is false (F).
   a. (R)-(+) Carvone and limonene are diastereomers. ____
   b. (R)-(+) Carvone and (S)-(+) carvone are both volatile liquids. ____
   c. (R)-(+) Carvone and limonene are not isomers of one another. ____
   d. (R)-(+) Carvone and limonene are constitutional isomers. ____

3. What color change is expected when a sample of a carvone is treated with Br_2 in CH_2Cl_2?

4. What color change is expected when a sample of a carvone is treated with aqueous KMnO_4?

5. Answer the previous two questions for the case in which limonene rather than a carvone is used.

6. What do you expect to observe when a carvone is treated with 2,4-dinitrophenylhydrazine?
7. What is the vapor pressure of bromine at 20 °C (760 torr)?

8. Why should inhalation of the vapors of bromine be avoided?

9. What action should you take if bromine gets on your skin?

10. The flash points (°C), respectively, of ethanol, ethyl acetate, and dichloromethane are _______, _______, and _______.

11. What may occur if 2,4-dinitrophenylhydrazine is absorbed into the body?
Section 7.6  RESOLUTION OF RACEMIC 1-PHENYLETHANAMINE

NAME (print): ___________________________ DATE: ______________

INSTRUCTOR: __________________________ LABORATORY SECTION: ____________

1. Put a check mark beside any of the following physical properties that would be expected to be the same for the enantiomeric 1-phenylethanamines.

   boiling point _____, solubility in acetone _____, the R_f-value in TLC _____, odor _____, retention time in GLC _____, rotation of plane-polarized light _____.

2. Methanol is to be used as the solvent for the separation of the diastereomeric forms of 1-phenylethanamine hydrogen tartrate. Briefly explain why this solvent is better for the separation than

   a. petroleum ether (bp 60–80 °C, 760 torr).

   b. water.

3. Why is the solution of 1-phenylethanamine hydrogen tartrate not to be cooled in an ice-water bath before the crystals are collected?

4. Why cannot racemic tartaric acid be used to resolve 1-phenylethanamine?
5. Why cannot meso tartaric acid be used to resolve 1-phenylethylamine?

6. What is the crystalline form of the amine hydrogen tartrate to be isolated in this experiment?

7. What is the principle on which the resolution of the racemic 1-phenylethanesamines by formation of a tartrate salt depends?

8. Why is flameless heating specified for preparing the methanolic solution of tartaric acid in this experiment?

9. The flash points (°C), respectively, of diethyl ether, methanol, and 1-phenylethylamine are ______, ______, and ______.

10. What toxicology data, if any, are provided in the abbreviated MSDS sheet for tartaric acid?
Section 9.2 FREE-RADICAL CHLORINATION OF 1-CHLOROBUTANE (Miniscale)

1. Write the structure of the substance to be used in this experiment to initiate free radical chain chlorination, and give the balanced equation for its thermal decomposition.

2. What is the molar ratio of initiator to sulfuryl chloride to be used in the procedure? Why is so little initiator needed relative to the amount of chlorinating agent used?

3. Write the balanced equation for the reaction of sulfuryl chloride with water.

4. Why would water be an inappropriate medium for extinguishing a fire involving sulfuryl chloride?

5. Why is a “gas trap” to be used in the chlorination reaction?
6. What is the theoretical amount of weight that should be lost by the reaction mixture in the procedure you are to perform? Show your calculations.

7. Why should the separatory funnel be vented frequently when the reaction mixture is shaken with aqueous sodium carbonate? Write an equation for any chemical reaction that is responsible for the need for venting.

8. What type of distillation apparatus, fractional or simple, is to be used for isolation of the chlorinated product(s)? Why is this type selected?

9. Why are pieces of glass or of stainless steel “sponge” rather than copper “sponge” recommended as packing material for the distillation column?

10. What visual criterion is to be used to assess whether the reaction mixture is dry prior to distillation?

11. Put a check mark beside any of the following materials that evolves toxic fumes upon heating or burning: 1-chlorobutane _____, sulfuryl chloride _____, sodium chloride _____, and sodium sulfate _____.

12. List possible effects of inhaling excessive amounts of sulfuryl chloride.
Section 9.2  FREE-RADICAL CHLORINATION OF 1-CHLOROBUTANE (Microscale)

1. Write the structure of the substance to be used in this experiment to initiate free radical chain chlorination, and give the balanced equation for its thermal decomposition.

2. What is the molar ratio of initiator to sulfuryl chloride to be used in the procedure? Why is so little initiator needed relative to the amount of chlorinating agent used?

3. Write the balanced equation for the reaction of sulfuryl chloride with water.

4. Why would water be an inappropriate medium for extinguishing a fire involving sulfuryl chloride?

5. Why is a “gas trap” to be used in the chlorination reaction?
6. What is the theoretical amount of weight that should be lost by the reaction mixture in the procedure you are to perform? Show your calculations.

7. Why should the conical vial be vented frequently when the reaction mixture is shaken with aqueous sodium carbonate? Write an equation for any chemical reaction that is responsible for the need for venting.

8. What technique is to be used to hasten the drying of the reaction mixture prior to distillation?

9. What visual criterion is to be used to assess whether the reaction mixture is dry prior to distillation?

10. Put a check mark beside any of the following materials that evolves toxic fumes upon heating or burning: 1-chlorobutane _____, sulfuryl chloride _____, sodium chloride _____, and sodium sulfate _____.

11. What action should you take if bromine gets on your skin?

12. List possible effects of inhaling excessive amounts of sulfuryl chloride.
Section 9.3 RELATIVE RATES OF FREE-RADICAL CHAIN BROMINATION

1. Draw a structure containing the specified type of hydrogen atom and circle the atom.
   a. $2^\circ$ aliphatic hydrogen atom.
   b. $1^\circ$ benzylic hydrogen atom.
   c. vinylic hydrogen atom.

1. What experimental criterion is to be used to measure the rates of bromination of the hydrocarbons used in this experiment?

2. Underline the proper response in the following:
   a. The results of this experiment will allow determination of the (relative, absolute) rates of bromination of a series of hydrocarbons.
   b. The mechanism of the bromination is classified as a(n) (electrophilic addition, nucleophilic substitution, electrophilic substitution, free-radical substitution) process.
   c. Molecular bromine is a (solid, liquid, gas) at room temperature and is (corrosive, non-corrosive) to the skin.
4. Why should apparatus containing residues of bromine not be rinsed with acetone? How can such residues be chemically removed?

5. Determine the average ratio of Br₂ to hydrocarbon to be used in this experiment. To perform the calculation, assume that the hydrocarbon has a density of 0.8 and a molecular weight of 100. Show your work.

6. Why are the hydrocarbons to be used in excess in this experiment?

7. Why is the bromine that is to be added to each test tube in this experiment measured out as a solution of bromine in dichloromethane rather than as pure bromine?

8. List possible effects of inhaling excessive amounts of toluene.

f. The flash point (°C) of toluene is _______; that of methylcyclohexane is _______.

g. Underline the media that are appropriate for extinguishing fires involving toluene, tert-butylbenzene and ethylbenzene: Water Carbon dioxide Chemical powder Foam
Section 10.2 DEHYDROHALOGENATION OF ALKYL HALIDES (Miniscale)

NAME (print): ______________________________ DATE: ________________

INSTRUCTOR: ____________________________ LABORATORY SECTION: ________________

1. Calculate the molar ratio of base to 2-bromo-2-methylbutane to be used in the elimination experiment(s) you perform and specify which is "limiting reagent." Show your calculations.

2. Why is a Hempel column rather than a regular condenser to be used during the period of reflux?

3. Why is the column to be filled with a packing material during this stage of the procedure?

4. Why is the hold-up of the Hempel column when being used as a fractionating column greater when the column is packed rather than unpacked?
5. Why is the vacuum adapter of the apparatus to be fitted with a drying tube throughout the course of the reaction and the distillation?

6. Why is the receiving flask to be cooled in an ice-water bath throughout the reaction and distillation?

7. Why is it particularly important that the ground-glass joint linking the Hempel column to the reaction flask be properly lubricated for the base-promoted elimination of 2-methyl-2-bromobutane?

8. Write equations for the chemical reactions that you will use to demonstrate the presence of alkenes in your distilled product.

9. What is an appropriate extinguishing medium for fires involving potassium tert-butoxide?

10. The flash points (°C) of 2-methyl-1-butene and of 2-methyl-2-butene, respectively, are _______ and _______.

11. List possible effects if 2-methyl-1-butene and 2-methyl-2-butene get on your skin.
Section 10.2  DEHYDROHALOGENATION OF ALKYL HALIDES (Microscale)

NAME (print): ______________________________________ DATE: ______________
INSTRUCTOR: ___________________ LABORATORY SECTION: ______________

1. Calculate the molar ratio of potassium hydroxide to 2-bromo-2-methylbutane to be used in the experiment you perform and specify which is "limiting reagent." Show your calculations.

2. Why is a water-cooled rather than an air-cooled condenser specified in this procedure?

3. Why is an aqueous solution of potassium hydroxide an unsuitable medium for effecting the base-promoted elimination of this procedure?

4. What would the consequence be of not having the condenser tightly mated with the Hickman stillhead?
5. Why is necessary to stir the reaction mixture during the elimination reaction?

6. Why should the distillate be kept cold after it is removed from the Hickman stillhead?

7. Why is it particularly important that the ground-glass joint linking the Hickman stillhead to the conical vial be properly lubricated for the base-promoted elimination of 2-methyl-2-bromobutane?

8. Write equations for the chemical reactions that you will use to demonstrate the presence of alkenes in your distilled product.

9. What is an appropriate extinguishing medium for fires involving potassium tert-butoxide?

10. The flash points (°C), respectively, of 2-methyl-1-butene and of 2-methyl-2-butene are ______ and ______.

11. List possible effects if 2-methyl-1-butene and 2-methyl-2-butene come in contact with your skin.
1. What is the function of the acid catalyst in promoting the dehydration of 4-methyl-2-pentanol?

2. Why would concentrated hydrochloric acid be an inappropriate catalyst for the dehydration of 4-methyl-2-pentanol?

3. Why is the formation of a substitution product involving displacement of water by attack of bisulfate upon the protonated alcohol not a reaction of concern in the elimination reaction?

4. Why is there an upper limit to the temperature at which the alkenes are to be collected?

5. Write equations for the chemical reaction(s) that you will use to demonstrate the presence of alkenes in your distilled product.
6. Why is it important to dry the crude alkenes prior to the final distillation?

7. What visual criterion is to be used to assess whether the reaction mixture is dry prior to distillation?

8. Specify the limiting reagent in the dehydration procedure you perform. Give your reasoning.

9. Underline the media appropriate for extinguishing fires involving the isomeric methylpentenes formed in the dehydration of 4-methyl-2-pentanol: Water Carbon dioxide Chemical powder Foam

10. Specify whether the alkenes formed by the dehydration procedure you are to perform have a flash point above 25 °C? If so, give the flash point(s).

11. List possible effects of inhaling excessive amounts of 4-methyl-2-pentanol.
Section 10.3  DEHYDRATION OF ALCOHOLS. PART B. DEHYDRATION OF CYCLOHEXANOL

NAME (print): ___________________________  DATE: ________________
INSTRUCTOR: ___________________________  LABORATORY SECTION: ______________

1. What is the function of the acid catalyst in promoting the dehydration of cyclohexanol?

2. Why would concentrated hydrochloric acid be an inappropriate catalyst for the dehydration of cyclohexanol?

3. Why is the formation of a substitution product involving displacement of water by attack of bisulfate upon the protonated alcohol not a reaction of concern in the elimination reaction?

4. Why is there an upper limit to the temperature at which the cyclohexene is to be collected?

5. Write equations for the chemical reaction(s) that you will use to demonstrate the presence of cyclohexene in your distilled product.
6. Why is it important to dry the crude cyclohexene prior to the final distillation?

7. What visual criterion is to be used to assess whether the reaction mixture is dry prior to distillation?

8. Specify the limiting reagent in the dehydration procedure you perform. Give your reasoning.

9. Underline the media appropriate for extinguishing fires involving cyclohexene formed in the dehydration: Water Carbon dioxide Chemical powder Foam

10. Specify whether the cyclohexene formed by the dehydration procedure you are to perform has a flash point above 25 °C? If so, give the flash point.

11. List possible effects of inhaling excessive amounts of cyclohexanol.
Section 10.5  ADDITION OF HYDROBROMIC ACID TO 1-HEXENE

1. Why do the conditions of this experiment favor electrophilic rather than free-radical addition of HBr to 1-hexene?

2. What is the role of the quaternary ammonium salt to be used in this experiment?

3. When concentrated HBr is added to 1-hexene, do you expect to observe a homogeneous or a heterogeneous reaction mixture? Why?

4. Why is vigorous stirring or other agitation of the reaction mixture important?

5. Write equations for the chemical reaction(s) responsible for the pressure build-up when the crude reaction mixture is washed with aqueous sodium bicarbonate.
6. If you are uncertain as to which layer is which when extracting an organic solution with an aqueous solution, how might you settle the issue?

7. Write balanced equations for the chemical reactions that could be used to demonstrate that 2- rather than 1- bromohexane has been formed in this experiment.

8. What is the mechanistic basis for each of the reactions you proposed in Exercise 7 that allows differentiation of 1° from 2° alkyl halides?

9. List possible effects of inhaling excessive amounts of HBr.

10. What action should you take if methyl trioctylammonium chloride or 1-hexene gets on your skin?
1. Why should apparatus containing residues of bromine not be rinsed with acetone? How can such residues be chemically removed?

2. What should you do if any bromine comes in contact with your skin?

3. What is the vapor pressure of bromine at 20 °C?

4. List possible effects of inhaling excessive amounts of bromine.

5. What changes, if any, in the color of the reaction mixture do you anticipate as the bromination proceeds?
6. Underline the proper category of the bromination reaction: nucleophilic substitution, electrophilic substitution, nucleophilic addition, electrophilic addition, elimination.

7. Classify the addition reaction as to whether it is an oxidation, reduction, or neither. Show how you reached your conclusion.

8. List possible effects of spilling dichloromethane on your skin.

9. List possible effects of inhaling excessive amounts of dichloromethane.
Section 10.6  BROMINATION OF ALKENES. PART A. BROMINATION OF (E)-STILBENE: THE GREEN APPROACH

1. What color do you expect to see when aqueous hydrogen peroxide is added to the solution contain hydrobromic acid?

2. Why would water not be a good solvent to use in place of ethanol for the reaction?

3. Is the bromide ion of HBr oxidized or reduced to form Br₂??

4. What is the limiting reagent in the reaction?

5. What changes, if any, in the color of the mixture do you anticipate as the reaction proceeds?
6. Underline the proper category of the bromination reaction: nucleophilic substitution, electrophilic substitution, nucleophilic addition, electrophilic addition, elimination.

7. Classify the addition reaction as to whether it is an oxidation, reduction, or neither. Show how you reached your conclusion.

8. List possible effects of inhaling excessive amounts of HBr.

9. What action should you take if hydrogen peroxide gets on your skin?
Section 10.6  BROMINATION OF ALKENES. PART B. BROMINATION OF (E)-CINNAMIC ACID

NAME (print):  _______________________________  DATE:  ________________
INSTRUCTOR:  ____________________________  LABORATORY SECTION:  ____________

1. What should you do if pyridinium tribromide comes in contact with your skin?

2. What should you do if glacial acetic acid comes in contact with your skin?

3. What is the boiling point of glacial acetic acid?

4. What is the limiting reagent for the formation of the dibromide? Show your calculation.

5. How do you know if any active bromine is present in the reaction mixture or filtrate?

6. How should any excess pyridinium tribromide be neutralized?
7. Explain what "like" and "unlike" mean when describing the relative stereochemistry of a molecule.

8. Which diastereomer do you expect to form as the major product of this reaction, \( \text{l-2,3-dibromo-3-phenylpropanoic acid} \) or \( \text{u-2,3-dibromo-3-phenylpropanoic acid} \)?

9. Is molecule A, drawn below, \( \text{l-2,3-dibromo-3-phenylpropanoic acid} \) or \( \text{u-2,3-dibromo-3-phenylpropanoic acid} \)?

![Molecule A diagram]

10. What is a lachrymator, and why is it inappropriate to use acetone when cleaning apparatus that may contain residual bromine?
Section 10.7  HYDRATION OF NORBORNENE

1. Why is it important to add the concentrated sulfuric acid to water rather than the reverse?

2. What is the function of sulfuric acid in promoting the hydration of norbornene?

3. Why would concentrated hydrochloric acid be an unsuitable replacement for sulfuric acid?

4. How much potassium hydroxide is required to neutralize 2 mL of concentrated sulfuric acid? Show your calculation.

5. Write a balanced equation for the chemical reaction(s) responsible for the pressure build-up when the crude reaction mixture is washed with aqueous sodium bicarbonate.
6. What is the composition of the solid that might appear in the separatory funnel during the work-up?

7. What is the purpose of washing the ethereal layer containing the product with saturated sodium bicarbonate and sodium chloride prior to drying the solution with anhydrous sodium sulfate?

8. Why is it necessary to remove all of the diethyl ether prior to subliming the product?

9. What is the purpose of the trap between the water aspirator and the sublimation apparatus?

10. Why is it necessary to seal the capillary tube before determining the melting point of exo-norborneol?

11. What action should you take if sulfuric acid gets on your skin?

10. The flash point (°C) of norbornene is _______.

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Section 10.8 HYDROBORATION-OXIDATION OF (+)-α-PINENE

NAME (print): ___________________________ DATE: ___________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: __________

1. Determine the molar ratios of each of the reactants to be used in this procedure and specify the “limiting reagent.” Show your calculations.

2. Write the chemical equation by which THF disrupts the usual equilibrium between diborane and borane.

3. Why are the solvent and apparatus to be carefully dried prior to the reaction?

4. At the end of the reaction, water is to be added to the reaction mixture containing the borane-THF complex. Why is this addition to be slow?

5. Write a balanced equation for the reaction that occurs between water and borane-THF complex.
6. What purpose is served by adding basic hydrogen peroxide to the reaction mixture?

7. What action should you take if hydrogen peroxide gets on your skin?

8. Write equations for any chemical test(s) you might use to determine whether the desired alcohol is contaminated with \((+)-\alpha\)-pinene.

9. Underline the media appropriate for extinguishing fires involving borane/THF solutions:
   Water    Carbon dioxide    Chemical powder    Foam

10. List the possible effects of inhaling excessive amounts of tetrahydrofuran.
Section 11.2  DEHYDROBROMINATION OF MESO-STILBENE DIBROMIDE

NAME (print): ___________________________ DATE: ______________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ______________

1. Explain the purpose of placing a carborundum boiling stone in the reaction vessel prior to heating.

2. Explain why triethylene glycol is used as the reaction solvent for this reaction.

3. Calculate the molar ratio of base to the meso-stilbene dibromide used in this experiment. For the purposes of this calculation, assume that the KOH contains 15% water.

4. Explain why a sand bath is preferred to a mineral oil bath as the heating source for this experiment.

5. In this experiment, the reaction vessel is cooled to room temperature prior to adding water. Explain why this is done rather than adding water to the hot reaction mixture.
6. What is expected to precipitate from the reaction mixture during the period of heating?

7. Diphenylacetylene is only sparingly soluble in diethyl ether. Explain why is the addition of water to the reaction mixture is preferable to adding diethyl ether as a means of precipitating the product for isolation by filtration.

8. The flash point (°C) of triethylene glycol is ________.

9. Is diphenylacetylene carcinogenic?

10. What action should you take if the solution of potassium hydroxide gets on your skin?
Section 11.3  PREPARATION OF 3-HYDROXY-3-METHYL-2-BUTANONE

NAME (print): ______________________________________  DATE: ___________

INSTRUCTOR: ___________________________  LABORATORY SECTION: ___________

1. Write the structural formula for the product of the reaction of 2-methyl-3-butyn-2-ol with bromine in carbon tetrachloride.

2. In the preparation of the reagents for hydration of 2-methyl-3-butyn-2-ol, why is concentrated sulfuric acid added to water rather than water added to sulfuric acid?

3. What is the purpose of adding potassium carbonate and sodium chloride to the distillate before extracting it with dichloromethane? Why not use just potassium carbonate?

4. Calculate the molar ratio of mercuric oxide and 2-methyl-3-butyn-2-ol used in this experiment. Show your calculations.
5. Is mercuric oxide the limiting reagent? Explain.

6. Why does hydration of 2-methyl-3-butyn-2-ol give the ketone, 3-hydroxy-3-methyl-2-butanone (4), rather than the aldehyde, 3-methyl-3-hydroxybutanal (5)?

7. How can 4 and 5 be differentiated chemically? by spectroscopic methods?

8. Underline the media, if any, that are not appropriate for extinguishing fires involving 2-methyl-3-butyn-2-ol: Water  Carbon dioxide  Chemical powder  Foam

9. The flash point (°C) of 2-methyl-3-butyn-2-ol is ________.

10. What action should you take if concentrated sulfuric acid gets on your skin?
Section 11.4  FORMATION OF A SILVER ACETYLIDE AND ITS DECOMPOSITION

NAME (print): __________________________________________ DATE: ________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. Write a balanced equation for the reaction of silver ammonia complex with 2-methyl-3-butyln-2-ol.

2. Why is the silver salt of the alkyne not allowed to dry on the filter?

3. Write an equation for the reaction of dilute hydrochloric acid with the silver salt of the alkyne.

4. Put a check mark beside the reported chronic effects, if any, of silver nitrate:
   carcinogen _____, mutagen _______.

5. What action should you take if ammonium hydroxide gets on your skin?

6. List the possible effects of inhaling excessive amounts of ammonia.
Section 12.3 APPLICATIONS OF DIELS-ALDER REACTIONS. PART A. REACTION OF 1,3-BUTADIENE AND MALEIC ANHYDRIDE

NAME (print): ___________________________________________ DATE: __________________
INSTRUCTOR: ___________________________________________ LABORATORY SECTION: ______________

1. Specify the conformation required to enable a 1,3-diene to undergo the Diels-Alder reaction.

2. Which conformation of 1,3-butadiene, s-cis or s-trans, is thermodynamically preferred and why?

3. Why does 1,3-butadiene react more rapidly with maleic anhydride than with another molecule of itself?

4. Why is 3-sulfolene rather than 1,3-butadiene itself to be used as a source of the diene?

5. Define the term “in situ preparation” as it applies to this experiment.

6. Why is a gas trap required in this experiment?
7. What is the limiting reagent in the reaction between 1,3-butadiene and maleic anhydride and why do you think this reagent rather than the other was made limiting? Show your calculations.

8. Why is petroleum ether to be added to the solution of product in xylene?

9. What type of filtration is to be used to isolate the crystalline product?

10. Is either 3-sulfolene or maleic anhydride mutagenic?

11. The flash point (°C) of xylene is _______.

12. List the possible effects of inhaling excessive amounts of xylene.

13. Underline the media, if any, that are not appropriate for extinguishing fires involving xylene:
   Water  Carbon dioxide  Chemical powder  Foam
Section 12.3 APPLICATIONS OF DIELS-ALDER REACTIONS. PART B. REACTION OF 1,3-CYCLOPENTADIENE AND MALEIC ANHYDRIDE (Miniscale)

NAME (print): _______________________________ DATE: __________________
INSTRUCTOR: _______________________________ LABORATORY SECTION: ____________

1. What conformation is required to enable a 1,3-diene to undergo the Diels-Alder reaction?

2. What is the limiting reagent in the reaction between 1,3-cyclopentadiene and maleic anhydride, and why do you think this reagent rather than the other one was made limiting? Show your calculations.

3. Why does 1,3-cyclopentadiene react more rapidly with maleic anhydride than with another molecule of itself?

4. Why is a 25-mL rather than a 10-mL round-bottom flask to be used for cracking dicyclopentadiene when only 7 mL of this substance is being cracked?
5. Why is a fractional rather than a simple distillation apparatus specified for the cracking of dicyclopentadiene?

6. Why must the solution of maleic anhydride in ethyl acetate-petroleum ether be homogeneous when 1,3-cyclopentadiene is added to it?

7. Why is the solution containing the desired product to be cooled slowly?

8. What type of filtration, gravity or vacuum, is to be used for isolating the product?

9. Is dicyclopentadiene a carcinogen?

10. List the possible effects of inhaling excessive amounts of 1,3-cyclopentadiene.

11. The flash points (°C) of dicyclopentadiene and ethyl acetate are, respectively, _____ and ____. 

12. What action should you take if maleic anhydride gets in your eyes?
Section 12.3 APPLICATIONS OF DIELS-ALDER REACTIONS. PART B. REACTION OF 1,3-CYCLOPENTADIENE AND MALEIC ANHYDRIDE (Microscale)

NAME (print): ___________________________________________ DATE: ____________________
INSTRUCTOR: ______________________ LABORATORY SECTION: ___________

1. What conformation is required to enable a 1,3-diene to undergo the Diels-Alder reaction?

2. What is the limiting reagent in the reaction between 1,3-cyclopentadiene and maleic anhydride, and why do you think this reagent rather than the other one was made limiting? Show your calculations.

3. Why does 1,3-cyclopentadiene react more rapidly with maleic anhydride than with another molecule of itself?

4. Why must the solution of maleic anhydride in ethyl acetate-petroleum ether be homogeneous when 1,3-cyclopentadiene is added to it?
5. Why is the solution containing the desired product to be cooled slowly?

6. What technique is to be used to effect slow cooling of the solution of product?

7. What type of filtration, gravity or Craig tube, is to be used for isolating the product?

8. Write equations for the chemical tests you are to perform on the product.

9. Is dicyclopentadiene a carcinogen?

10. List the possible effects of inhaling excessive amounts of 1,3-cyclopentadiene.

11. The flash point (°C) of ethyl acetate is ________.

12. What action should you take if maleic anhydride gets in your eyes?
Section 12.3  APPLICATIONS OF DIELS-ALDER REACTIONS. PART C. HYDROLYSIS OF ANHYDRIDES

NAME (print): ___________________________________________ DATE: ________________

INSTRUCTOR: ________________________ LABORATORY SECTION: ________________

1. What serves as the nucleophile in the conversion of a cyclic anhydride to a dicarboxylic acid?

2. Cyclic anhydrides are often insoluble in water, whereas the dicarboxylic acids derived from them are soluble. Why is there a difference in the water-solubilities of these two classes of compounds?

3. Why should a warm aqueous solution of a solid not be cooled in an ice-water bath before crystallization has started?

4. What strategies might you use to induce crystallization if the desired product oils out rather than crystallizes?

5. What do you expect to observe when an aqueous solution of your product is tested with pHydrion paper?
6. What do you expect to observe when you treat your product with Br₂/CH₂Cl₂?

7. Write the structure of the product expected from the reaction between your product and Br₂/CH₂Cl₂.

8. What action should you take if the solution of bromine in dichloromethane gets on your skin?

9. List the possible effects of inhaling excessive amounts of bromine.
Section 13.2  FORMATION OF SEMICARBAZONES UNDER KINETIC AND THERMODYNAMIC CONTROL

NAME (print): ______________________________ DATE: ________________

INSTRUCTOR: ____________________ LABORATORY SECTION: ______________

1. Define:
   a. kinetic control of a reaction.
   b. thermodynamic control of a reaction.

2. Write a balanced equation for the reaction between semicarbazide hydrochloride and dibasic potassium phosphate.

3. The three buffer systems described in this chapter are A: CH₃CO₂H/CH₃CO₂⁻; B: H₂PO₄⁻/HPO₄²⁻; and C: H₂CO₃/HCO₃⁻. Answer the following questions by using A, B, or C to refer to the proper buffer system.
   a. Which buffer system maintains the lowest pH? ______
   b. Which buffer system generally produces the fastest rate of formation of semicarbazones of aldehydes and ketones? ______
   c. Which buffer system maintains a pH in the range 6.1–6.2? ______

4. What is the purpose of cooling each reaction mixture in an ice-water bath after reaction periods of various times and at various temperatures?
5. Typical melting ranges of crystals obtained in various parts of this experiment might be, for example, 150–162 °C, 163–165 °C, and 199–201 °C. Identify these melting ranges with the mixtures of semicarbazones of cyclohexanone (C) and 2-furaldehyde (F) listed below.
   a. 98% C/2% F
   b. 98% F/2% C
   c. 40% F/60% C

6. Why should a Thiele tube containing mineral oil not be used for determining melting points in this experiment?

7. The flash points (°C) of cyclohexanone and 2-furaldehyde are ______ and ______, respectively.

8. List the possible effects of inhaling excessive amounts of cyclohexanone.

9. List the possible effects of inhaling excessive amounts of 2-furaldehyde.

10. Is 2-furaldehyde a mutagen?

11. What action should you take if 2-furaldehyde gets in your eyes?
Section 14.4  PREPARATION OF 1-BROMOBUTANE: AN S<sub>N</sub>2 REACTION

NAME (print): __________________________________ DATE: ______________________

INSTRUCTOR: __________________________ LABORATORY SECTION: ______________

1. Describe the method by which hydrogen bromide is prepared for use in this experiment.

2. Determine the limiting reagent for the conversion of 1-butanol to 1-bromobutane and the theoretical yield of product.

3. Is this reaction an S<sub>N</sub>1 or S<sub>N</sub>2 process? How will the mechanism be confirmed experimentally?

4. Write the structures of two possible side products that might be formed in this reaction.
5. What is the purpose of the following experimental techniques, and where is each used in the preparation?
   a. heating at reflux.
   b. simple distillation.
   c. adding anhydrous sodium sulfate.

6. List the possible effects of inhaling excessive amounts of 1-butanol.

7. The flash points (°C) of 1-butanol and 1-bromobutane are ______ and ______, respectively.

8. What action should you take if 1-butanol gets on your skin?

9. Indicate whether the following statement is true (T) or false (F): Water is an appropriate medium for extinguishing fires involving 1-bromobutane.
1. Determine the limiting reagent for the preparation of 2-chloro-2-methylpropane and calculate the theoretical yield of product.

2. Why is this reaction carried out at room temperature rather than at elevated temperatures?

3. Is this reaction an $S_N1$ or an $S_N2$ process? Does the experiment contain any procedures for experimental verification of the mechanism?

4. Write the structures of any possible side products in the reaction.
5. After the initial reaction is carried out and the crude product is isolated, it is washed with saturated sodium bicarbonate solution. What is the purpose of this wash, and could dilute sodium hydroxide solution be used instead of sodium bicarbonate? Explain briefly.

6. The flash point (°C) of 2-methyl-2-butanol is _______.

7. What action should you take if hydrochloric acid gets on your skin?

8. List the media appropriate for extinguishing fires involving 2-methyl-2-butanol.
Section 14.6 CHEMICAL KINETICS: EVIDENCE FOR NUCLEOPHILIC SUBSTITUTION MECHANISMS

NAME (print): ________________________________ DATE: __________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: _____________

1. Place a checkmark beside any of the following entries that need not be listed in the table you prepare in your laboratory notebook prior to performing this kinetics experiment
   a. Temperature at which the kinetic run is performed. ______
   b. Volume(s) of the aliquots withdrawn during the kinetic run. ______
   c. Molarity of the aqueous base being used to titrate the aliquots withdrawn. ______
   d. The amount of phenolphthalein added prior to titrating the aliquots. ______
   e. The volume of aqueous base required to titrate each aliquot that is withdrawn. ______
   f. The time when titration of each aliquot is performed. ______

2. What role is served by phenolphthalein in this experiment?

3. Is this reaction expected to be an S_N1 or an S_N2 process?

4. Write the structures of the organic solvolysis products expected in the reaction.

5. How is the solvolysis reaction “quenched”, that is, stopped, so that the extent of reaction can be measured as a function of time?
6. Why is it important that you quench the aliquots you withdraw as quickly as possible?

7. Why is the flask containing the solvolysis mixture kept stoppered as much as possible during the kinetic run?

8. Why must you save some of the aqueous base being used for titrations for use in a subsequent laboratory period?

9. What action should be taken if some of the solvolysis mixture comes in contact with your skin?

10. What action should you take if 2-propanol gets in your eyes?
Section 14.7  COMPETING NUCLEOPHILES IN Sn REACTIONS

NAME (print): _______________________________ DATE: _______________________

INSTRUCTOR: ______________________________ LABORATORY SECTION: _____________

1. Why is possible to conduct the reaction with 2-methyl-2-propanol reaction at room temperature, whereas that with 1-butanol requires heating?

2. These reactions use concentrated sulfuric acid. Why would water not be a suitable solvent for these reactions?

3. What is the predicted order of nucleophilicity of the halide ions for: 1-butanol and 2-methyl-2-propanol?

4. Provide an explanation for the differences in the reactivity of the halide ions.
5. In the reaction of 2-methyl-2-propanol with the halide mixture, what is the expected ratio of products? Why do you predict that particular ratio?

6. What is the gas that is evolved during the neutralization?
   a. hydrochloric acid.
   b. carbonic acid.
   c. carbon dioxide.
   d. the alcohol.

7. The reagents that are used in this experiment contain ammonium halides. The ammonium ion is removed by
   a. simple distillation.
   b. vacuum filtration.
   c. liquid-liquid extraction.
   d. evaporation.

8. In the gas chromatographic analysis, the carrier gas is
   a. air.
   b. hydrogen.
   c. carbon dioxide.
   d. helium.

9. What is the flash point of 2-methyl-2-butanol? _______

10. What should you do if the sulfuric acid mixture gets on your skin?
Section 14.8  COMPETITION BETWEEN SUBSTITUTION AND ELIMINATION

1. The bromides, used in this experiment are primary (1-bromohexane) and secondary (2-bromohexane). What results would you expect, if a tertiary bromide were used?

2. How is the excess alcohol (used as the solvent) removed from the reaction mixture?

3. What is the purpose of adding sodium sulfate to the organic layer?

4. Why is an aqueous solution of hydroxide an unsuitable medium for these reactions?

5. The hexenes produced in this reaction are more volatile than the corresponding bromides:
   T___  F___
6. The solutions that are used in this experiment have alcohols as the solvent. The excess alcohol is removed by (circle the answer):
   a. simple distillation.
   b. vacuum filtration.
   c. liquid-liquid extraction.
   d. evaporation.

7. The flash point (°C) of methanol is ________.

8. In the gas chromatographic analysis, the carrier gas is:
   a. air.
   b. hydrogen.
   c. carbon dioxide.
   d. helium.

9. What action should you take if you get potassium tert-butoxide on your skin?
Section 15.2  FRIEDEL-CRAFTS ALKYLUATION OF *p*-XYLENE WITH 1-BROMOPROPANE

1.  
   a. Determine the limiting reagent in this experiment. Show your calculations.
   
   b. Why is this reagent chosen to be limiting?

2.  
   a. What function is served by AlCl₃ in this reaction?
   
   b. Why is so little of it required?

3. Why should the *p*-xylene and the apparatus to be used in the experimental procedure be dry?
4. What experimental technique is to be used for drying the \( p \)-xylene?

5. Do you expect the ratio of \( n \)-propyl-\( p \)-xylene to isopropyl-\( p \)-xylene produced in this experiment to be larger or smaller than the ratio of \( n \)-propylbenzene to isopropylbenzene produced by a similar alkylation of benzene by 1-bromopropane? Explain your answer.

6. Why is the 1-bromopropane to be added dropwise to the reaction mixture rather than all at once?

7. What is the reason for pouring the completed reaction mixture into a crushed ice/water mixture? Why water, and why ice?

8. What compounds are present in the aqueous solution that is to be discarded?

9. What action should you take if \( \text{AlCl}_3 \) gets on your skin?

10. The flash points (°C) of \( p \)-xylene and 1-bromopropane are ______ and ______, respectively.

11. List the media appropriate for extinguishing fires involving \( p \)-xylene and 1-bromopropane.
Section 15.3 FRIEDEL-CRAFTS ACYLATION OF ANISOLE

NAME (print): ______________________________ DATE: ______________

INSTRUCTOR: ______________________________ LABORATORY SECTION: ____________

1. Why is it important to avoid exposing an acid chloride to moisture? Write the reaction that occurs when an acid chloride and water are mixed.

2. What role does the zinc oxide play in this reaction?

3. How is the zinc oxide recovered in this procedure?

4. The solvent used for the extraction is dichloromethane. This excess solvent is removed by (circle letter):
   a. simple distillation.
   b. vacuum filtration
   c. liquid-liquid extraction.
   d. evaporation.

5. During the work-up procedure, the dichloromethane layer is washed with sodium bicarbonate solution. What is the purpose of this wash?

6. What is the gas that is evolved during the addition of sodium bicarbonate solution (circle letter)?
   a. chlorine.
   b. carbonic acid.
   c. carbon dioxide.
   d. hydrogen.
7. If you accidentally spill some zinc oxide on your skin, what symptoms might you expect and what first aid measures should you take?

8. List the possible effects if dichloromethane gets on your skin.

9. Many acid chlorides are listed as a “lachrymators.” What is a lachrymator?
Section 15.4  NITRATION OF BROMOBENZENE. PART A. NITRATION

NAME (print): ..................................................  DATE: ........................
INSTRUCTOR: ........................................ LABORATORY SECTION: ..............

1. Calculate the molar ratio of nitric acid:bromobenzene used in this experiment. (Concentrated nitric acid is 16 \( M \)) What is the limiting reagent in this experiment?

2. Why is dinitration not a significant process under the conditions to be used?

3. Compare the ratio of reactants in this experiment (Exercise 1, above) with that in the experiment of Section 15.2 (see Pre-Lab Exercise 1 for that section). Why are they so different?

4. What is the function of the concentrated sulfuric acid in this experiment?

5. Why is the reaction mixture to be stirred during the addition of bromobenzene to the mixture of acids?
6. Is the nitration reaction expected to be exothermic or endothermic?

7. What compounds are present in the aqueous solution from which the isomeric bromonitrobenzenes are to be filtered?

8. Why is the $p$-isomer of the product expected to be less soluble in ethanol than the $o$-isomer?

9. Underline the media, if any, that are not appropriate for extinguishing fires involving bromobenzene and $o$- and $p$-bromonitrobenzene: Water Carbon dioxide Chemical powder Foam

10. List the possible effects of getting bromobenzene on your skin.

11. What action should you take if sulfuric or nitric acid gets on your skin?
Section 15.4  NITRATION OF BROMOBENZENE. PART B. THIN-LAYER CHROMATOGRAPHY

1. Why should the developing chamber for a TLC plate not be open to the atmosphere?

2. Why should a TLC plate be removed from the solvent before the solvent front reaches the top of the plate?

3. Which of the following diagrams illustrate(s) an improper way of spotting a TLC plate? Tell what is wrong in each such case.

   a. 
   b. 
   c. 
   d. Liquid level

   a. 
   b. 
   c. 
   d.
4. What problem would attend failure to mark the position of the solvent front on the TLC plate immediately after developing the plate?

5. What technique are you to use to determine the location of compounds on the TLC plate once it has been developed?

6. What consequence would you predict if a more polar eluant were used for the TLC chromatography?

7. The flash points (°C) for ethyl acetate and hexane are _______ and _______, respectively.

8. List the possible effects of inhaling excessive amounts of iodine.
1. What difficulty may result if
   a. the chromatography column is not placed in a vertical position?
   b. the liquid level of the eluent is allowed to drop below the top of the packing in the column?

2. Why should there be no air bubbles in the column after it has been packed?

3. What is the purpose of the layer of sand added at the top of the adsorbent when preparing the chromatography column?
4. Why should a minimum amount of solvent be used to introduce a mixture onto the chromatography column?

5. What consequence would you predict if a more polar eluant were used for the column chromatography?

6. What technique are you to use to monitor the contents of the various fractions of eluant that you collect?

7. The flash points (°C) for ethyl acetate and hexane are ______ and ______, respectively.

8. List the possible effects of inhaling excessive amounts of ethyl acetate.
Section 15.5  SUBSTITUENT EFFECTS ON ELECTROPHILIC AROMATIC SUBSTITUTION.

PART A1. QUALITATIVE MEASUREMENTS

NAME (print): ___________________________  DATE: ______________
INSTRUCTOR: ___________________________  LABORATORY SECTION: ____________

1. Why is 15 M acetic acid an appropriate solvent in which to perform rate studies of electrophilic brominations?

2. Show that 15 M acetic acid is approximately 90% acetic acid and 10% water by weight.

3. Why is benzene itself not to be used as a substrate in this experiment?

4. Why is it important to add the solution of bromine quickly and in one portion to the test tube containing the aromatic substrate?

5. What criterion is to be used for measuring the rates of bromination in the experiment?
6. Why is it important to maintain a constant temperature in this experiment?

7. Why might it be necessary to conduct this experiment at more than one temperature?

8. The flash points (°C) of anisole, diphenyl ether, and phenol are ________, ________, and ________, respectively.

9. Underline the media, if any, that are not appropriate for extinguishing fires involving the compounds listed in Exercise 8: Water  Carbon dioxide  Chemical powder  Foam

10. What action should you take if 15 M acetic acid gets on your skin?
1. Why is benzene itself not to be used as a substrate in this experiment?

2. Confirm with calculations that 90% acetic acid is approximately 15 M.

3. Why is it important to stir the solution of bromine and substrate quickly once these two reagents have been combined and to record the time at which mixing occurs?

4. At what wavelength is the spectrometer to be set for measuring the concentration of bromine present as a function of time?

5. Why is it important that the temperature be as constant as possible throughout the course of a kinetic run?

6. Why is it inadvisable to use acetone to clean the cuvette you are using in this experiment?
7. Briefly describe how you will perform a “blank” determination.

8. Why must you clean and dry the cuvette you used for the “blank” determination before using it for the kinetic run itself?

9. Where is it recommended that the needle of the spectrophotometer dial should be when a reading on it is recorded?

10. The flash points (°C) of anisole, diphenyl ether, and acetanilide are ______, ______, and ______ respectively.

11. Underline the media, if any, that are not appropriate for extinguishing fires involving the compounds listed in Exercise 8: Water  Carbon dioxide  Chemical powder  Foam
Section 15.5  SUBSTITUENT EFFECTS ON ELECTROPHILIC AROMATIC SUBSTITUTION. PART B.
ELECTROPHILIC AROMATIC BROMINATION OF MONOSUBSTITUTED ARENES

NAME (print): ___________________________ DATE: ______________
INSTRUCTOR: ___________________________ LABORATORY SECTION: __________

1. What should you do if you get bromine solution on your skin?

2. What is a lachrymator, and why must care be used when cleaning up after reactions that involve bromine?

3. Why is it necessary to use a gas trap on the reaction apparatus?

4. Which is the upper layer when an aqueous mixture and dichloromethane are placed in a separatory funnel?

5. Circle the organic product(s) that is (are) expected to be produced from the reaction of bromine with:
   a. Anisole
      \[ \text{O} \cdot \text{CH}_3 \]
      \[ \text{Br} \]
   b. Toluene
      \[ \text{CH}_3 \]
      \[ \text{Br} \]
      \[ \text{Br} \]
      \[ \text{CH}_3 \]
c. Bromobenzene

\[ \begin{align*}
&\text{Br} & \text{Br} \\
&\text{Br} & \text{Br} \\
&\text{Br} & \text{Br}
\end{align*} \]

d. Methyl benzoate

\[ \begin{align*}
&\text{O} & \text{O-CH}_3 \\
&\text{Br} & \text{Br} \\
&\text{Br} & \text{Br} \quad & \text{Br} & \text{Br}
\end{align*} \]

6. How does a Lewis acid affect the rate of electrophilic aromatic bromination.

7. If your reaction produces two products, how can you determine the ratio of products?

8. Why is dibromination not expected to occur?

9.

a. What is the purpose of adding sodium bisulfite to the reaction mixture at the end of the reaction?

b. What is the purpose of adding sodium bicarbonate to the reaction mixture at the end of the reaction?

10. List the possible effects of inhaling bromobenzene?
Section 15.5  SUBSTITUENT EFFECTS ON ELECTROPHILIC AROMATIC SUBSTITUTION.

PART C. ELECTROPHILIC AROMATIC NITRATION OF MONOSUBSTITUTED ARENES

NAME (print): ___________________________________________________________ DATE: ______________________

INSTRUCTOR: ___________________________________________________________ LABORATORY SECTION: ____________

1. What precautions should be taken, when handling concentrated nitric and sulfuric acids?

2. Which of the two acids is the stronger?

3. The electrophile in these reactions is (circle the letter):
   a. nitrosyl ion.
   b. aryl cation.
   c. nitronium ion.
   d. nitrous oxide.

4. In the nitration of methyl benzoate, what potential side reaction can occur, which would lower the yield of the desired product?

5. What is the purpose of cooling the reaction mixtures to 0 °C?

6. All of these reactions are carried out in very strong acids. How are the acids removed from the organic products (circle the letter)?
   a. simple distillation.
   b. neutralization and extraction.
   c. recrystallization.
   d. evaporation.
7. Write the three possible regioisomers of the nitration of chlorobenzene. Would you expect them to be formed in equal amounts? Explain.

8. In the gas chromatographic analysis, the carrier gas is (circle the letter):

   a. air.
   b. hydrogen.
   c. carbon dioxide.
   d. helium.

9. What action should you take if you get these concentrated acids on your skin?
Section 15.6  AZO DYES AND THE CHEMISTRY OF DYEING FABRICS

1. Define the following terms:
   a. Direct dye
   b. Parallel synthesis
   c. Compound library
   d. \( \lambda_{\text{max}} \)
   e. Hydrogen bond

2. What functional groups are present in wool fibers?

3. What should you do if concentrated hydrochloric acid comes in contact with your skin?

4. What is the limiting reagent for the formation of the diazonium salt? Show your calculation.
5. What temperature is specified for forming the diazonium salt in the procedure?

6. What should you do if no solid is formed in the coupling reaction?

7. If three different amines and four different activated arenes are used in the procedure, how many different dye compounds will be produced for the compound library?

8. Why should the diazonium salt not be isolated and stored for later use?
Section 16.2  PREPARATION OF ALDEHYDES AND KETONES BY OXIDATION OF ALCOHOLS. PART
A. OXIDATION OF CYCLODODECANOL TO CYCLODODECANONE

NAME (print): ___________________________________ DATE: __________________
INSTRUCTOR: ________________________________ LABORATORY SECTION: ____________

1. What is the oxidizing agent in this experiment and to what is it reduced during the course of the reaction?

2. What qualitative test is performed to determine whether sufficient oxidizing agent has been used in this procedure?

3. Write an equation that expresses the chemical reaction that forms the basis of the test of Exercise 2.

4. What purpose is served by washing the ethereal extract containing the product with saturated sodium bicarbonate?

5. What purpose is served by washing the ethereal extract containing the product with saturated sodium bisulfite?

6. List the possible effects if cyclooctadecanol gets on your skin.

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7. Underline the media, if any, that are *not* appropriate for extinguishing fires involving cyclododecanol and cyclododecanone: Water  Carbon dioxide  Chemical powder  Foam
Section 16.2  PREPARATION OF ALDEHYDES AND KETONES BY OXIDATION OF ALCOHOLS. PART
B. OXIDATION OF 4-CHLOROBENZYL ALCOHOL TO 4-CHLOROBENZOIC ACID

NAME (print): ___________________________ DATE: ______________
INSTRUCTOR: __________________________ LABORATORY SECTION: ___________

1. What is the oxidizing agent in this experiment and to what is it reduced in the course of the reaction?

2. What qualitative test is performed to determine whether sufficient oxidizing agent has been used in this procedure?

3. Write an equation that expresses the chemical reaction that forms the basis of the test of Exercise 2.

4. What purpose is served by washing the ethereal extract containing the product with saturated sodium bicarbonate?

5. The flash points (°C) of acetic acid, and diethyl ether are _______, and _______, respectively.

6. What are the possible effects if excessive amounts of chlorine are inhaled?
7. What action should you take if the solution of bleach gets on your skin?

8. Underline the media, if any, that are not appropriate for extinguishing fires involving 4-chlorobenzyl alcohol and 4-chlorobenzoic acid: Water  Carbon dioxide  Chemical powder Foam
Section 16.2  PREPARATION OF ALDEHYDES AND KETONES BY OXIDATION OF ALCOHOLS. PART C. AEROBIC OXIDATION OF BENZYLIC ALCOHOLS

NAME (print): ____________________________ DATE: ____________________________

INSTRUCTOR: ____________________________ LABORATORY SECTION: ____________________________

1. Mark the following statements as true or false. If false, briefly explain why this is the case.
   a. A catalyst increases the rate of a reaction by lowering the energy of the starting materials and products, _____
   b. TEMPO is a ligand in the CuBr/TEMPO oxidation. _____
   c. A heterogenous catalyst is in a phase different from that of the reactants. _____
   d. A catalyst changes the position of the equilibrium of a reaction. _____
   e. A catalyst can, in principle, be recovered at the end of a reaction. _____
   f. Molecular oxygen is reduced to water in the aerobic oxidation reaction. _____
   g. An aldehyde can be reduced to the corresponding carboxylic acid. _____

2. What is the stoichiometric oxidizing agent in the CuBr/TEMPO-catalyzed aerobic oxidation reaction?

3. Write the balanced chemical reaction for the aerobic oxidation of a benzylic alcohol.

4. What is the percentage of oxygen in air?

5. What is the oxidation state of the copper species at the start of the aerobic oxidation reaction? At the end of the reaction?

6. The boiling points (°C) of acetone and pentane are _____ and _____, respectively.

7. The densities (g/mL) of acetone and pentane are _____, and _____, respectively.


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9. List the possible symptoms of inhaling pentane. What precautions should you take to avoid exposure?

10. List the possible symptoms of skin contact with TEMPO (7). What precautions should you take to avoid exposure?
Section 16.3  BASE-CATALYZED OXIDATION-REDUCTION OF ALDEHYDES: THE CANNIZZARO REACTION

1. Write the three half reactions that sum up to the overall equation below.

   \[ 2 \text{C}_6\text{H}_4\text{ClCHO} + \text{KOH} \rightarrow \text{C}_6\text{H}_4\text{CO}_2^- + \text{C}_6\text{H}_4 \]

2. Why is the Cannizzaro reaction limited to aldehydes having no \( \alpha \)-hydrogen atoms?

3. What is the limiting reagent in this reaction? Show how you arrived at this conclusion.

4. Why do ketones having no \( \alpha \)-hydrogen atoms not undergo the Cannizzaro reaction?
5. What is an emulsion? Why is it desirable to have an emulsion rather than two phases during the reaction between 4-chlorobenzaldehyde and aqueous base?

6. What is the solid that is formed after 4-chlorobenzaldehyde has been allowed to react with aqueous potassium hydroxide? Why does it dissolve in water?

7. List the possible effects if 4-chlorobenzaldehyde gets on your skin.

8. Does 4-chlorobenzyl alcohol present a moderate or a high fire hazard?
1. What is meant by the term catalytic hydrogenation?

2. What catalyst is used in this preparation, and how is it prepared?

3. Indicate how hydrogen gas is generated for the preparation of the catalyst; write a balanced equation to show this reaction.

4. What is the stereochemistry of catalytic hydrogenation? Cite an example that shows this.
5. Does the catalytic hydrogenation of 4-cyclohexene-<i>cis</i>-1,2-dicarboxylic acid show the stereochemistry of hydrogenation? Explain.

6. Aqueous acid is used as the solvent for the reaction. Could water at pH = 7 have been used as the solvent instead of the one that is used? Explain.

7. Does addition of <i>concentrated</i> hydrochloric acid to an aqueous solution of the reduction product of this reaction increase or decrease the water solubility of the product? Why?

8. What hazardous mineral acid is produced by decomposition of chloroplatinic acid?

9. Underline the media appropriate for extinguishing fires involving sodium borohydride:
   - Water
   - Carbon dioxide
   - Chemical powder
   - Foam

10. What abbreviated MSDS toxicological data, if any, are available for 4-cyclohexene-1,2-dicarboxylic acid?

11. What action should you take if sodium borohydride solution gets on your skin?
Section 17.2  CATALYTIC HYDROGENATION OF THE CARBON-CARBON DOUBLE BOND.
PART B. TRANSFER HYDROGENATION OF CINNAMIC ACID DERIVATIVES

1. What is meant by the term *transfer hydrogenation*?

2. What is the limiting reagent for the formation of the product? Show your calculation.

3. Why should you not add the Pd/C powder to a methanol solution of the starting materials?

4. What does it mean if a reaction is chemoselective?

5. Cinnamic acid has a molecular mass of 148.16. If you used 150 mg of cinnamic acid for the transfer hydrogenation reaction, how much ammonium formate (in grams) should you use? Show your calculation.

6. Which is the upper layer when an aqueous mixture and diethyl ether is placed in a separatory funnel?
7. How does one make a filtering pipet with the piece of glass-fiber filter?

8. How do you know when the reaction is complete?

9. What hazardous vapor is produced by the decomposition of ammonium formate?

10. What is an advantage of using transfer hydrogenation rather than just using hydrogen gas?

11. What action should you take if 1 M hydrochloric acid gets on your skin?
Section 17.3  FORMATION AND REDUCTION OF N-CINNAMYLIDENE-\textit{m}-NITROANILINE

NAME (print):  

DATE:  

INSTRUCTOR:  

LABORATORY SECTION:  

1. Draw the structure of an imine, and give the equation for its conversion into an amine as done in this experiment.  

2. What metal hydride reducing agent is used in this experiment, and what are the advantages of using it rather than hydrogen gas in the presence of a catalyst? What product would be formed if catalytic hydrogenation were used?  

3. Define the term \textit{reductive amination}.  

4. Draw the structure of the imine intermediate that is formed, and describe what experimental technique is used to maximize the yield of this compound.  

5. Why is it unnecessary to isolate and purify the intermediate imine?
6. Determine the limiting reagent in this preparation and compute the theoretical yield. Show all work.

7. List the toxic fumes evolved in a fire involving cinnamaldehyde, \( m \)-nitroaniline, and cyclohexane.

8. What action should you take if \( m \)-nitroaniline gets on your skin?

9. The flash points (°C) of cyclohexane and cinnamaldehyde are ______ and ______, respectively.

10. Underline the media appropriate for extinguishing fires involving sodium borohydride:
    Water  Carbon dioxide  Chemical powder  Foam

11. Write an equation for reaction of sodium borohydride and methanol that yields hydrogen.
Section 17.4  REDUCTION OF 9-FLUORENONE

1. Determine whether sodium borohydride is the limiting reagent in this experiment. Show your work.

2. Why is it important to avoid exposing sodium borohydride to moisture? Write the reaction that occurs when sodium borohydride and water are mixed.

3. What color change should occur as the reduction of 9-fluorenone proceeds? Provide a reason for this change.

4. In the experiment, the reducing agent is allowed to react with 9-fluorenone, and after the reaction is complete, sulfuric acid is added. What is the purpose of this addition? Why is it important to dissolve all of the solids completely?
5. Describe the advantages of using a metal hydride reduction reaction rather than catalytic hydrogenation in this procedure.

6. Write an equation for the reaction that might occur during the recrystallization of fluorenol from methanol if all of the sulfuric acid has not been removed by washing.

7. List the possible effects if 9-fluorenone gets on your skin.

8. Underline the media, if any, that are not appropriate for extinguishing fires involving a combination of 9-fluorenone, fluorenol, methanol, and sodium borohydride:
   Water  Carbon dioxide  Chemical powder  Foam
Section 17.5  REDUCTIONS: A CHIRAL ALCOHOL FROM A KETONE. PART A. TARTARIC ACID-MEDIATED REDUCTION OF METHYL ACETOACETATE

NAME (print): __________________________________________ DATE: __________________________

INSTRUCTOR: __________________________ LABORATORY SECTION: __________________________

1. Determine the molar ratio of sodium borohydride to tartaric acid that you used in the experiment. Explain why this ratio is used.

2. Why is it important to avoid exposing sodium borohydride to moisture? Write the reaction that occurs when sodium borohydride and water are mixed.

3. What is the role of tartaric acid in this procedure?

4. The solvent used for the extraction is diethyl ether. This excess solvent is removed by (circle the letter):
   a. simple distillation.
   b. vacuum filtration.
   c. liquid-liquid extraction.
   d. evaporation.

5. Underline the media, if any, that are not appropriate for extinguishing fires involving a combination of tetrahydrofuran and sodium borohydride:
   Water          Carbon dioxide          Chemical powder          Foam
6. What gas is evolved during the addition of hydrochloric acid to the reaction mixture?
   a. chlorine.
   b. carbonic acid.
   c. carbon dioxide.
   d. hydrogen.

7. What are the possible effects of ingesting methyl acetoacetate?

8. What is the flash point (°C) of tetrahydrofuran?
1. Why should the Erlenmeyer flask in which the fermentation is conducted not be stoppered tightly?

2. What is the role of the Na$_2$HPO$_4$ in the reaction?

3. What is the practical advantage of using dichloromethane as the extraction solvent rather than diethyl ether?

4. Why use filter-aid for filtering the reaction mixture?

5. Why is it important not to shake the separatory funnel vigorously during the extraction of the product with dichloromethane?
6. Although sucrose is added to the reaction, it is not necessary. Propose a reason for using sucrose, and account for the fact that it is not absolutely required for the reduction.

7. What are the possible effects of ingesting methyl acetoacetate?

8. List the possible effects if dichloromethane gets on your skin.
Section 17.6  DETERMINING OPTICAL PURITY

1. Why is it important to use a dry NMR tube for measuring the optical purity of a sample using a chiral shift reagent?

2. Why should the chiral shift reagent be stored in a desiccator?

3. What is the effect of having undissolved solid in the NMR sample?

4. Why should the sample be allowed to stand for about 20 min after dissolution of the shift reagent?
5. What is the estimated accuracy of determining the enantiomeric excess by the NMR method?

6. When determining the optical purity of an alcohol using chiral shift reagents, it is important to perform the experiment for samples of both the racemic alcohol and enantiomerically enriched alcohol. Explain.

7. The ratio of the two enantiomers is determined by comparing the peak areas for the methyl peaks on the ester group. Why is this better than trying to compare the peak areas for the methine hydrogen $\alpha$ to the hydroxyl group?

8. List the possible effects of inhaling excessive amounts of chloroform.

9. Is chloroform listed as a mutagen?
Section 18.2  THE WITTIG AND RELATED REACTIONS. PART A. SYNTHESIS OF (Z)- AND (E)-STILBENES BY A WITTIG REACTION

NAME (print): __________________________________ DATE: _____________________

INSTRUCTOR: ______________________________ LABORATORY SECTION: ______________

1. What is the limiting reagent in this experiment? Show your work.

2. Explain why it is important to stir the reaction mixture vigorously.

3. After the Wittig reaction is complete, the solution of isomeric stilbenes in dichloromethane is washed with saturated sodium bisulfite. What is the purpose of this wash?

4. Suppose the aldehyde used in a Wittig reaction is contaminated with the corresponding carboxylic acid. What complication would this cause?
5. After the irradiation of the isomeric stilbenes in dichloromethane containing iodine is complete, the solution is washed with saturated sodium bisulfite. What is the purpose of this wash?

7. List the effects of inhaling excessive amounts of benzaldehyde.

8. List the possible effects of ingesting benzyltriphenylphosphonium chloride.

9. What action should you take if 50% (by weight) sodium hydroxide solution gets on your skin?

10. List the possible effects if sodium bisulfite solution gets in your eyes.
Section 18.2  THE WITTIG AND RELATED REACTIONS. PART B. SYNTHESIS OF A STILBENE BY THE HORNER-WADSWORTH-EMMONS REACTION

1. What is the limiting reagent in this experiment? Show your work.

2. Explain why exposure of the solution of potassium tert-butoxide in N,N-dimethylformamide to the atmosphere should be minimized.

3. Suppose the aldehyde used in a Horner-Wadsworth-Emmons reaction is contaminated with the corresponding carboxylic acid. What complication would this cause?

4. Is the reaction of the potassium salt 13 of the phosphonate ester with benzaldehyde expected to be exothermic or endothermic? Give the basis for your answer.
5. How is the product of this experiment freed from residual benzaldehyde?

6. What are the possible effects of inhaling excessive amounts of \(N,N\)-dimethylformamide?

7. Is \(N,N\)-dimethylformamide listed as a potential carcinogen?

8. The flash point (°C) of \(N,N\)-dimethylformamide is _______.

9. What action should you take if potassium tert-butoxide gets on your skin?
Section 18.3 PREPARATION OF TRANS-P-ANISALACETOPHENONE

NAME (print): ___________________________ DATE: ________________

INSTRUCTOR: ______________________ LABORATORY SECTION: ______________

1. \( p \)-Anisaldehyde and acetophenone are used in equimolar amounts in this experiment. Suggest a complication that might result if two moles of ketone per mole of aldehyde were used.

2. The amount of sodium hydroxide used to promote the condensation reaction is less than an equimolar amount. What complication might result if a much larger amount of concentrated sodium hydroxide were used?

3. Why is the condensation (dehydrated) product rather than the aldol addition (hydrated) product obtained in this experiment?

4. Why does the enolate ion of an aromatic ketone react faster with an aldehyde group, producing a crossed-aldol reaction, than with the carbonyl group of another molecule of ketone?
5. List the possible effects of inhaling excessive amounts of acetophenone.

6. Is \textit{p}-anisaldehyde listed as a mutagen?

7. List the possible effects of ingesting methanol.
Section 18.4 PREPARATION OF 4,4-DIMETHYL-2-CYCLOHEXEN-1-ONE

1. Why is an intramolecular rather than an intermolecular aldol condensation favored with 2,2-dimethylhexanal-5-one (28)?

2. What is the limiting reagent in the reaction to form the Michael addition product 28? Show your work.

3. Why do you think the carbonyl-containing compound identified in Exercise 2 rather than the other was made limiting?

4. Why must water be removed from the reaction mixture during the course of the reaction?
5. 1-Propanol boils at only a slightly lower temperature than toluene, and all the reagents used in this experiment are soluble in it. Why is it an inappropriate solvent to use in this procedure?

6. This reaction occurs much faster if a sulfonic acid rather than a carboxylic acid is used as the catalyst. Why might this be?

7. What is the theoretical volume of water that would be obtained in the reaction? Show your work. How might you account for the production of greater than a theoretical volume in the experiment?

8. The flash points (°C) of toluene, 3-buten-2-one, and 2-methylpropanal, respectively, are ______, ______ and ______, respectively.

9. What action should you take if 3-buten-2-one gets on your skin?

10. List the possible effects of inhaling excessive amounts of 2-methylpropanal.

11. Is toluene listed as a carcinogen?
Section 18.5 SYNTHESIS OF ETHYL 6-METHYL-2-OXO-4-PHENYL-1,2,3,4-TETRAHYDRO-PYRIMIDINE-5-CARBOXYLATE

NAME (print): ___________________________ DATE: ________________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. What is the limiting reagent for the formation of the product? Show your calculation.

2. What is the catalyst used in this reaction?

3. How is the catalyst removed from the product?

4. Why is it important to mix the ingredients thoroughly in this procedure?

5. What is the pKₐ of ethyl acetoacetate?

6. Two of the reactants in this experiment are liquids. If they are not completely consumed in the reaction, the excess liquids are removed by (circle the letter):
   a. simple distillation.
   b. vacuum filtration.
   c. liquid-liquid extraction.
   d. evaporation.
7. What is the recommended solvent for the recrystallization (circle the letter)?
   a. water.
   b. ethyl acetate.
   c. ethanol.
   d. hexanes.

8. The flash point (°C) of ethanol is ________.

9. In the thin layer chromatographic analysis, the mobile phase is (circle the letter):
   a. ethanol
   b. the TLC plate
   c. air
   d. hexanes-ethyl acetate mixture

10. Is urea listed as a mutagen?

11. In the technique of recrystallization, it is always best to use a minimum amount of solvent:
    T____  F____

12. The reported melting point (°C) of ethyl 6-methyl-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate is ________.
Section 19.2  GRIGNARD REAGENTS: PREPARATION

NAME (print): ___________________________ DATE: _______________________
INSTRUCTOR: ___________________________ LABORATORY SECTION: __________

1. What is the limiting reagent in this reaction? Show your work.

2. Why are ethereal solvents important to the success of preparing the Grignard reagent?

3. Why must the reagents, solvents, and apparatus used for preparing the Grignard reagent be dry?

4. Why were you cautioned not to place plastic and rubber parts in the drying oven?

5. Why is it necessary to have an ice-water bath available during the preparation of the Grignard reagent?
6. What signs should you look for in determining whether the reaction initiated?

7. Why should the aryl or alkyl halide from which the Grignard reagent is to be made not be added all at once to the reaction flask?

8. Why is anhydrous diethyl ether added to the magnesium in two portions, one at the beginning of the reaction and the second after formation of the Grignard reagent has started?

9. The flash points (°C) of bromobenzene and 1-bromobutane are ______, and ______.

10. What action should you take if iodine gets on your skin?

11. Are vapors of diethyl ether heavier or lighter than air?
Section 19.4  GRIGNARD REAGENTS: REACTIONS. PART A. PREPARATION OF TRIPHENYL-METHANOL

NAME (print): __________________________________________________________ DATE: __________________________
INSTRUCTOR: ____________________________ LABORATORY SECTION: ____________________________

1. What is the limiting reagent in this reaction? Show your work.

2. Why do you think the particular reagent specified in Exercise 1 was made limiting?

3. Why would the presence of methanol in the methyl benzoate lower the yield of triphenylmethanol?

4. Why must the ester and the diethyl ether in which it is dissolved be anhydrous?

5. Why is the solution of ester added to the Grignard reagent in a dropwise fashion rather than all at once?
6. What is wrong with storing ethereal solutions in the laboratory bench from one period to the next?

7. Why is technical rather than *anhydrous* diethyl ether used in the work-up procedure?

8. Why is saturated aqueous sodium bicarbonate rather than just water used to remove residual sulfuric acid from the organic solution during the work-up procedure?

9. How are unchanged ester and biphenyl removed from the desired product by the work-up procedure used?

10. What happens to any Grignard reagent that remains in the reaction mixture after addition of the ester?

11. List the possible effects of inhaling excessive amounts of methyl benzoate.

12. The flash point (°C) of cyclohexane is ______.
1. What is the limiting reagent in this procedure? Show your work.

2. Why do you think the particular reagent specified in Exercise 1 was made limiting?

3. What would be the effect on yield of exposing the crushed Dry Ice to the atmosphere for long periods of time before use?

4. Why does pressure develop in the separatory funnel when the ethereal solution of benzoic acid is being extracted with aqueous sodium hydroxide?
5. Why is technical rather than *anhydrous* diethyl ether used in the work-up of the reaction mixture?

6. How are possible by-products such as biphenyl and benzophenone removed from the desired benzoic acid by the work-up procedure?

7. What is wrong with storing ethereal solutions in the laboratory bench from one period to the next?

8. List the possible effects of spilling benzoic acid on your skin.

9. What are possible effects if Dry Ice gets on your skin?
Section 19.4  GRIGNARD REAGENTS: REACTIONS. PART C. PREPARATION OF 2-METHYL-3-
HEPTANOL

NAME (print): ___________________________________________ DATE: ______________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ______________

1. What is the limiting reagent in this procedure? Show your work.

2. Why do you think the particular reagent specified in Exercise 1 was made limiting?

3. Why is it recommended that 2-methylpropanal be freshly distilled?

4. Why must the aldehyde and the diethyl ether in which it is dissolved be \textit{anhydrous}?

5. What is wrong with storing ethereal solutions in the laboratory bench from one period to the next?

6. Why is the solution of aldehyde added to the Grignard reagent in a dropwise fashion rather than added all at once?
7. Why is technical rather than anhydrous diethyl ether used in the work-up procedure?

8. Why is hydrolysis of the reaction mixture performed with aqueous sulfuric acid that is cold rather than at room temperature?

9. How is unchanged aldehyde removed from the desired product by the work-up procedure used?

10. What happens to any Grignard reagent that remains in the reaction mixture after addition of the aldehyde?

11. List the possible effects of inhaling excessive amounts of 2-methylpropanal.

12. What action should you take if 6 M sulfuric acid gets on your skin?
Section 19.5  PREPARATION OF 3-ETHYLHEX-5-EN-3-OL

NAME (print): ________________________________ DATE: ________________
INSTRUCTOR: ______________________________ LABORATORY SECTION: ______________

1. Why must the zinc be activated prior to introducing the allyl bromide?

2. Would you expect zinc pellets or zinc strips to work better for this reaction? Explain

3. Why must the glassware be dry before conducting the reaction?

4. Is the activation method used in this laboratory physical or chemical activation?

5. Why is a drying tube used in the experimental set up?

6. Why does the zinc metal largely dissolve during the course of the reaction?
7. What signs should you observe to know that the reaction is occurring?

8. The flash point of tetrahydrofuran is (circle the letter):
   
a. 10 °C  
b. -10 °C  
c. 70 °C  
d. -17 °C

9. What is the boiling point for 3-butanone?

10. What precautions should you take when handling allyl bromide?

11. What should you do if you get iodine on your skin?
Section 19.6  PREPARATION OF 4’-METHYL-(1,1’-BIPHENYL)-4-METHANOL

NAME (print): ___________________________ DATE: ________________

INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. Determine the limiting reactant in the reaction. Show your calculations.

2. A common way to quantify the catalyst:limiting reactant ratio in a catalytic reaction is to quote the amount of catalyst used in mol%. For example, 5 mol% catalyst means that the reaction uses 5% as much catalyst as limiting reactant. Showing all your work, calculate the mol% of Pd used in this reaction.

3. Write the balanced chemical reaction for the Suzuki-Miyaura coupling of 4-methylphenylboronic acid with 4-bromobenzyl alcohol.

4. State the oxidation state of the palladium atom at:
   a. the beginning of the Suzuki-Miyaura coupling reaction. _____
   b. following the oxidative addition step. _____
   c. following the reductive elimination step. _____

5. The boiling point (°C) of dichloromethane is ________.

6. The density (g/mL) of dichloromethane is ________.

7. The solubility (g/100 mL) of dichloromethane at 25 °C in water is ________.

8. List the possible symptoms of inhaling dichloromethane. What precautions should you take to avoid exposure?

9. List the possible symptoms of eye and skin contact with potassium hydroxide solution. What precautions should you take to avoid exposure?
Section 20.2 ESTERS AND THE FISCHER ESTERIFICATION PART A. PREPARATION OF BENZOCAINE

NAME (print): _________________________________ DATE: ________________
INSTRUCTOR: ______________________ LABORATORY SECTION: ____________

1. What is the limiting reagent in this procedure? Show your work.

2. What is the advantage of using absolute ethanol rather than 95% ethanol in this experiment?

3. Why is it important to add the sulfuric acid dropwise to the ethanolic solution of p-aminobenzoic acid?

4. Draw the structure of the solid that is formed when the concentrated sulfuric acid is added to the solution of p-aminobenzoic acid.
5. Why is it important that all of the solids dissolve during the reflux period for you to obtain a good yield of product?

6. Why is it important to neutralize the reaction mixture during the work-up?

7. Assuming it was necessary to add an additional portion of concentrated sulfuric acid, calculate about how much 10% aqueous sodium carbonate would be required to neutralize the reaction mixture.

8. What is the gas that is evolved during the neutralization?

9. The flash point (°C) of absolute ethanol is ________.

10. Is p-aminobenzoic acid listed as a mutagen?

11. List possible effects of ingesting benzocaine.
Section 20.2  ESTERS AND THE FISCHER ESTERIFICATION  PART B. IDENTIFYING UNKNOWN ESTERS PRODUCED BY FISCHER ESTERIFICATION

NAME (print):  
DATE:  
INSTRUCTOR:  
LABORATORY SECTION:  

1. The solutions you obtain from the stockroom are 1 M in benzoic acid. How many moles of benzoic acid are in your sample? Show your work.

2. Anhydrous alcohols rather than their aqueous solutions are used in the esterification because water is a product of the equilibrium in the reaction:  

   T _____  F _____

3. Why is it important to add the concentrated sulfuric acid dropwise to the solutions in the microwave option?

4. Draw the structure of the intermediate that is formed when the concentrated sulfuric acid is added to the solution of benzoic acid.

5. The solutions that are used in this experiment have alcohols as both the solvent and the reactant. The excess alcohol is removed by (circle the letter):
   
   a. simple distillation.
   b. vacuum filtration.
   c. liquid-liquid extraction.

6. Why is it important to neutralize the reaction mixture during the work-up?

7. If there is unchanged carboxylic acid present after reaction with the alcohol, how is it removed?
8. What is the gas that is evolved during the neutralization (circle the answer)?
   a. hydrochloric acid.
   b. carbonic acid.
   c. carbon dioxide.
   d. the alcohol.

9. The flash point (°C) of absolute ethanol is _______: 

10. In the gas chromatographic analysis, the carrier gas is (circle the answer):
    a. air.
    b. hydrogen.
    c. carbon dioxide.
    d. helium.

11. Is benzoic acid listed as a mutagen?

12. What is the characteristic odor of esters?
Section 20.3  PREPARATION OF N,N-DIETHYL-M-TOLUAMIDE

NAME (print): ___________________________________________ DATE: ____________________
INSTRUCTOR: _______________________________ LABORATORY SECTION: ________________

1. Explain why it is important to dry the glassware before use in this experiment.

2. Explain why it is important to have an airtight seal during this reaction.

3. What gas(es) is(are) removed by the gas trap during this experiment?

4. Why should anhydrous diethyl ether rather than technical diethyl ether be used to prepare a solution of diethylamine?

5. What is the limiting reagent in the first step of this experiment? Show your work.
6. What is the limiting reagent in the second step of this experiment? Show your work.

7. What is the purpose of adding 2.5 \( M \) sodium hydroxide solution in the work-up?

8. List the possible effects of spilling \( m \)-toluic acid on your skin.

9. List the possible effects of inhaling excessive amounts of diethylamine.

10. What action should you take if thionyl chloride gets on your skin?
Section 20.4 PREPARATION OF AND CHEMILUMINESCENCE OF LUMINOL

1. Write the acid-base reaction that occurs when 3-nitrophthalic acid and hydrazine are combined.

2. Mechanistically, the conversion of 3-nitrophthalic acid and hydrazine to 3-nitrophthalhydrazide (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

3. Why is triethylene glycol rather than ethylene glycol used as the solvent for the preparation of 5-nitrophthalhydrazide?

4. What are the reported melting points of 5-nitrophthalhydrazide and luminol?

5. Why is acetic acid added to the reaction mixture prior to the isolation of luminol?

6. In what type of environment should the chemiluminescence experiment be conducted?
7. What color do you expect to see as a result of the chemiluminescence of luminol?

8. What symptoms may occur if hydrazine is inhaled?

9. What symptoms may occur if 3-nitrophthalic acid gets into your eyes and what action(s) should be taken if it does?

10. What is recommended as a means of extinguishing a fire involving 3-nitrophthalhydrazide?

11. What action should be taken if glacial acetic acid gets on your skin?

12. What symptoms accompany ingestion of 3-aminophthalhydrazide?
Section 21.2  SULFANILAMIDE: DISCOVERY AND SYNTHESIS OF THE FIRST ANTIBIOTIC. PART A. PREPARATION OF ANILINE

NAME (print): ____________________________  DATE: ____________________
INSTRUCTOR: ____________________________  LABORATORY SECTION: ____________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Determine the limiting reagent in this reaction. Show your work.

3. Why is the reduction of nitrobenzene more efficient with tin powder that is free of surface oxides?

4. What changes in the reaction mixture do you expect to see as the reduction progresses?

5. Determine whether adding the specified amount of 12 M aqueous sodium hydroxide to the reaction mixture prior to steam distillation is sufficient to make the mixture basic. Show your calculation.
6. How is aniline to be separated from residual nitrobenzene in this procedure?

7. Why is the reaction mixture to be cooled after being saturated with salt but before extraction with diethyl ether?

8. What color is pure aniline, and why are undistilled samples of it often brown in color?

9. What are possible effects if aniline gets in your eyes?

10. Is nitrobenzene listed as a possible carcinogen?

11. Is aniline listed as a possible mutagen?
Section 21.2  SULFANILAMIDE: DISCOVERY AND SYNTHESIS OF THE FIRST ANTIBIOTIC. PART B. PREPARATION OF ACETANILIDE

NAME (print): ___________________________ DATE: ______________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ______________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. How is unchanged aniline to be separated from acetanilide in this procedure?

4. Suppose you plan to make up the specified aqueous solution of sodium acetate after you combine acetic anhydride with the aqueous solution of aniline. Why is this not an appropriate strategy?
5. Why is aqueous hydrochloric acid to be combined with aniline in this procedure?

6. Why is concentrated hydrochloric acid to be added to water rather than the reverse when you prepare the dilute acid to be used in this procedure?

7. What is the role of the sodium acetate in this reaction?

8. What strategy is to be used if the isolated acetanilide is colored?

9. Is aniline listed as a potential carcinogen?

10. What action should you take if acetic anhydride gets on your skin?

11. List the possible effects of inhaling excessive amounts of acetic anhydride.
Section 21.2 SULFANILAMIDE: DISCOVERY AND SYNTHESIS OF THE FIRST ANTIBIOTIC. PART C. PREPARATION OF 4-ACETAMIDOBENZENESULFONYL CHLORIDE

NAME (print): _____________________________ DATE: ______________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ______________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. Why is it important that the acetanilide to be used in this experiment be dry?

4. Why is ice water rather than warm water to be used for hydrolysis of the reaction mixture?
5. Given the work-up procedure that is to be used, could residual acetanilide contaminate the desired product in its crude state? Explain.

6. Why is it important to break up any lumps of the crude isolated product?

7. Why should the 4-acetamidobenzenesulfonyl chloride be combined with ammonia immediately rather than in the next laboratory period?

8. What action is to be taken if chlorosulfonic acid is spilled on your skin?

9. How are you to destroy residual amounts of chlorosulfonic acid?

10. Why is water not an appropriate medium for extinguishing fires involving chlorosulfonic acid?

11. An MSDS is currently not available for p-acetamidobenzenesulfonyl chloride. In lieu of this information, provide the following information for the analogous compound, benzenesulfonyl chloride:
   a. extinguishing media are
   b. two effects of inhaling it are
   c. it is or is not listed as a possible mutagen (underline correct answer).

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Section 21.2  SULFANILAMIDE: DISCOVERY AND SYNTHESIS OF THE FIRST ANTIBIOTIC. PART 
D. PREPARATION OF 4-ACETAMIDOBENZENESULFANILAMIDE

NAME (print): ___________________________ DATE: ________________
INSTRUCTOR: ___________________________ LABORATORY SECTION: ________________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, 
   (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic 
   substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a 
   reduction of the aromatic substrate, (h) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. Why will reaction of 4-acetamidobenzenesulfonyl chloride with ammonium hydroxide give the sulfonamide 
   rather than the sulfonic acid?

4. What would account for the exothermicity that might occur when ammonium hydroxide is first added to the 
   crude 4-acetamidobenzenesulfonyl chloride?
5. Why is 6 M sulfuric acid to be added after reaction of the sulfonyl chloride with ammonium hydroxide?

6. Why might there be a preference for the use of solid sodium carbonate instead of solid sodium hydroxide for basifying the acidic hydrolysis solution to be obtained in this experiment?

7. Provide two other names under which 4-acetamidobenzenesulfonyl chloride might be listed.

8. List some possible effects if 4-acetamidobenzenesulfonyl chloride gets on your skin.

9. List the possible effects of inhaling excessive amounts of ammonia.
Section 21.2  SULFANILAMIDE: DISCOVERY AND SYNTHESIS OF THE FIRST ANTIBIOTIC. PART E. PREPARATION OF SULFANILAMIDE

NAME (print): ____________________________ DATE: ________________

INSTRUCTOR: ____________________________ LABORATORY SECTION: ________________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Assume you are to prepare 30 mL of dilute hydrochloric acid as directed in the experimental procedure. Detail how would you do this and specify the molarity of the solution that results.

3. What is the role of hydrochloric acid in this reaction?

4. Assume 30 mL of dilute HCl is used in this procedure. How much sodium carbonate would be required to neutralize this much acid? Show your work.
5. Is sulfanilamide listed as a potential carcinogen?

6. What action should you take if concentrated hydrochloric acid gets on your skin?

7. List the possible effects of ingesting sulfanilamide.
Section 21.3  SYNTHESIS OF 1-BROMO-3-CHLORO-5-IODOBENZENE. PART B. PREPARATION OF 4-BROMOACETANILIDE

NAME (print): ___________________________________________ DATE: ___________________

INSTRUCTOR: ___________________ LABORATORY SECTION: _______________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Write the structures of the two other monobromo products that might be formed in this reaction and circle the one least likely to be formed in the reaction.

3. What is the limiting reagent in this reaction? Show your work.

4. If the crude product appears colored, how is it to be decolorized?

5. What do you expect to see when ice-cold water is added to the reaction mixture?
6. What should you do if glacial acetic acid comes in contact with your skin?

7. Why should glassware containing residues of bromine not be rinsed with acetone?

8. The flash point (°C) of glacial acetic acid is ________.

9. Is glacial acetic acid listed as a potential carcinogen?

10. What action should you take if bromine gets on your skin?
Section 21.3  SYNTHESIS OF 1-BROMO-3-CHLORO-5-IODOBENZENE. PART C. PREPARATION OF 4-BROMO-2-CHLOROACETANILIDE

NAME (print): ___________________________ DATE: ____________________
INSTRUCTOR: __________________________ LABORATORY SECTION: ____________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Write the balanced equation for formation of molecular chlorine from sodium chlorate and hydrochloric acid.

3. What is the limiting reagent in this reaction? Show your work.

4. Why is a gas trap to be used in this experiment if the reaction cannot be performed in a hood?

5. What do you expect to observe in the reaction vessel as the chlorination proceeds?
6. Should recrystallization of the product be necessary, what solvent is to be used?

7. What technique is to be used to destroy chlorine if it is present in any of the filtrates or washes obtained in this experiment?

8. What is the solubility of sodium chlorate in water?

9. List the possible effects of inhaling excessive amounts of chlorine gas.

10. What action should be taken if glacial acetic acid is spilled on your skin?

11. Underline the media appropriate for extinguishing fires involving sodium chlorate:

   Water  Carbon dioxide  Chemical powder  Foam
Section 21.3  SYNTHESIS OF 1-BROMO-3-CHLORO-5-IODOBENZENE. PART D. PREPARATION OF 4-BROMO-2-CHLOROANILINE

NAME (print):  ____________________________  DATE:  __________________
INSTRUCTOR:  ____________________________  LABORATORY SECTION:  __________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. What is the role of hydrochloric acid in promoting the hydrolysis reaction?

4. Determine whether the specified amount of aqueous sodium hydroxide that is to be used is sufficient to make the reaction mixture basic. Show your work.
5. Why is flameless heating specified for heating the reaction mixture under reflux?

6. What change in the originally homogeneous solution may you observe as the reaction proceeds?

7. If you are to recrystallize the crude product, what strategy is to be used if oiling out rather than crystallization occurs?

8. List the possible effects of inhaling excessive amounts of ethanol.

9. What action should be taken if concentrated hydrochloric acid is spilled on your skin?
Section 21.3  SYNTHESIS OF 1-BROMO-3-CHLORO-5-IODOBENZENE. PART E. PREPARATION OF 4-BROMO-2-CHLORO-6-IODOANILINE

NAME (print): __________________________________________ DATE: ______________________
INSTRUCTOR: ______________________ LABORATORY SECTION: ______________________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. Why does iodination rather than chlorination occur when 4-bromo-2-chloroaniline is treated with iodine monochloride?

4. Why is the iodination being performed on 4-bromo-2-chloroaniline rather than 4-bromo-2-chloroacetanilide?
5. List the possible effects of inhaling excessive amounts of iodine monochloride.

6. The melting point of iodine monochloride is _______; its boiling point is _______.

7. Underline the media appropriate for extinguishing fires involving sodium iodine monochloride: Water
   Carbon dioxide    Chemical powder    Foam
Section 21.3 SYNTHESIS OF 1-BROMO-3-CHLORO-5-IODOBENZENE. PART F. PREPARATION OF 1-BROMO-3-CHLORO-5-IODOBENZENE

NAME (print): ____________________________ DATE: __________________

INSTRUCTOR: _________________________ LABORATORY SECTION: ____________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) an electrophilic substitution reaction, (c) an SN₂ reaction, (d) an oxidation of the aromatic substrate, (e) a reduction of the aromatic substrate, (f) none of these.

2. What is the limiting reagent in this reaction? Show your work.

3. List the possible effects of ingesting sodium nitrite.

4. Toxicological information for 1-bromo-3-chloro-5-iodobenzene are presently not available. What compound may serve as an analog for making predictions about the toxicology of this trisubstituted benzene?
Section 21.4  LIDOCAINE: SYNTHESE OF AN ANESTHETIC AGENT. PART A. PREPARATION OF 2,6-DIMETHYLANILINE

NAME (print):  ________________________________  DATE:  __________________
INSTRUCTOR:  ________________________________  LABORATORY SECTION:  ____________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the organic substrate, (g) a reduction of the organic substrate, (h) none of these.

2. Write the balanced equation for the reduction of 2,6-dimethylnitrobenzene (35) to give 36 using stannous chloride, SnCl₂, in concentrated hydrochloric acid.

3. Based upon the equation you derived in Exercise 2, what is the limiting reagent in this reaction? Show your work.

4. What is the role of stannous chloride in this reaction?

5. Write the structure of the "damp product" collected in the first filtration.
6. Why is it necessary to treat the "damp product" with base and why is heat produced when 8 M KOH is added?

7. How is any unreacted 2,6-dimethylnitrobenzene separated from the 2,6-dimethylaniline?

8. Is 2,6-dimethylaniline listed as a possible mutagen?

9. What action should you take if 2,6-dimethylnitrobenzene gets on your skin?
Section 21.4  LIDOCAINE: SYNTHESIS OF AN ANESTHETIC AGENT. PART B. PREPARATION OF α-CHLORO-2,6-DIMETHYL-ACETANILIDE

NAME (print): ___________________________ DATE: ________________
INSTRUCTOR: __________________________ LABORATORY SECTION: ____________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the organic substrate, (g) a reduction of the organic substrate, (h) none of these.

2. What is the limiting reactant in this reaction? Show your work.

3. Why should the Erlenmeyer flask used for the first step of the reaction be dry?

4. Why is aqueous sodium acetate added to the acetic acid solution of α-chloro-2,6-dimethylacetanilide (37) as part of the procedure?

5. Why is it important that the crude product be washed until there is no residual acetic acid? (Hint: Consider the next experiment.)
6. Explain how unchanged 36 is removed from 37 in the workup procedure used.

7. What action should you take if α-chloroacetyl chloride gets on your skin?

8. List the possible effects of inhaling excessive amounts of 2,6-dimethylaniline.
Section 21.4  LIDOCAINE: SYNTHESIS OF AN ANESTHETIC AGENT. PART C. PREPARATION OF LIDOCAINE

NAME (print): .................................................. DATE: ___________________
INSTRUCTOR: ______________________ LABORATORY SECTION: ______________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the organic substrate, (g) a reduction of the organic substrate, (h) none of these.

2. What is the limiting reactant in this reaction? Show your work.

3. Which reactant is used in excess? Explain why it is used in excess.

4. Explain why it is important to dry the glassware prior to reacting diethylamine with 37.

5. Explain how unchanged 37 is removed from 25 in the workup procedure used.
6. Why is it necessary to basify the aqueous solution prior to extracting the lidocaine with diethyl ether?

7. Why is acetone added to the aqueous solution of lidocaine bisulfate?

9. Why does scratching at the air-liquid interface with a glass rod help to induce crystallization?

10. List the possible effects of getting diethylamine in your eyes.

11. List the possible effects of inhaling excessive amounts of diethylamine.
Section 22.2  PREPARATION OF POLYSTYRENE

NAME (print): ________________________________ DATE: __________________
INSTRUCTOR: ______________________________ LABORATORY SECTION: ____________

1. Write an equation for the reaction of tert-butylcatechol with sodium hydroxide solution that is responsible for removing the inhibitor from commercial styrene and explain how the extraction accomplishes the desired separation.

2. Write formulas for the products of the disproportionation reaction between two styryl radicals, CH(C₆H₅)CH₂.

3. What determines whether or not decantation rather than filtration may be used to separate a solid from a liquid?

4. Is styrene listed as a potential mutagen?

5. List the possible effects of inhaling excessive amounts of styrene.
6. The flash points (°C) of styrene, tert-butyl peroxybenzoate, and xylene are _____, _____, and _____, respectively.

7. Is styrene considered a slight, moderate, or severe fire hazard (underline correct answer)?

8. Is polystyrene considered a slight, moderate, or severe fire hazard (underline correct answer)?
Section 22.3  PREPARATION OF NYLON-6,10

1. Why is decanediyl dichloride rather than decanedoic acid used in this experiment?

2. Explain how reaction between decanediyl chloride in dichloromethane solution and 1,6-hexanediamine in aqueous solution occurs although the two immiscible solutions are not mixed.

3. Why should the tip of the separatory funnel containing the 1,6-hexanediamine solution be placed no more than 1 cm above the surface of the dichloromethane solution when making the addition?

4. Why is the aqueous solution added to the dichloromethane solution, rather than vice versa?

5. Note that decanediyl dichloride and 1,6-hexanediamine are used in equimolar amounts, whereas in many experiments one reactant is used in considerable molar excess. Why is the equimolar ratio used here?
6. Why is the formic acid solution of the polymer evaporated in the hood rather than at the laboratory bench?

7. The flash point (°C) of decanediyl dichloride is _______.

8. How may the odor of 1,6-hexanediamine be characterized?

9. Is 1,6-hexanediamine listed as a potential carcinogen?

10. List the possible effects of inhaling excessive amounts of decanediyl dichloride.
Section 23.3  DISACCHARIDES: HYDROLYSIS OF SUCROSE

1. Why is the mixture of sugars derived from hydrolysis of sucrose commonly referred to as “invert” sugar?

2. What effect would the presence of air bubbles in the polarimeter tube have on the value of the measured rotation?

3. What effect would incomplete transfer of the hydrolysis mixture from the reaction flask to the volumetric flask have on the value of the measured rotation?

4. Is sucrose considered a slight, moderate, or severe fire hazard (underline correct answer)?

5. Sucrose is a non-reducing sugar.  True  _____  False  _____

6. What is the role of hydrochloric acid in the hydrolysis of sucrose?

7. What action should you take if concentrated hydrochloric acid gets on your skin?
Section 23.4  CARBOHYDRATES: THEIR CHARACTERIZATION AND IDENTIFICATION

NAME (print): __________________________________________ DATE: ______________
INSTRUCTOR: __________________________ LABORATORY SECTION: ______________

1. Tollens's and Benedict's tests depend on the ability of carbohydrates to oxidize the test reagent.
   True _____  False _____

2. Explain why nonreducing sugars give a positive Barfoed's test more slowly than do reducing sugars.

3. What is likely to happen if Tollens's test is performed in a test tube that is not clean?

4. What do you expect to see in the case of a positive Benedict's test?

5. What do you expect to see in the case of a positive Barfoed's test?

6. Why are both glucose and sucrose recommended as carbohydrates to test for purposes of comparison when performing Benedict's test on an unknown?
7. Why is it important not to heat the test solution for longer than 5 minutes when performing Barfoed’s test?

8. What purpose is served by citrate ion in the stock solution of Benedict's reagent?

9. What hazard is associated with storing unused Tollens’s reagent?

10. What are the possible effects of inhaling excessive amounts of ammonia?
Section 23.4  FORMATION OF OSAZONES

1. What is the function of saturated aqueous sodium bisulfite solution in this procedure?

2. Would you expect osazone formation to occur faster or slower in strongly acidic media as compared to weakly acidic media? Explain your answer.

3. Why does reaction of phenylhydrazine with aldoses and ketoses stop after the first two carbon atoms in the chain have been converted to hydrazone functions?

4. Why is sodium acetate required if phenylhydrazine hydrochloride is used as the source of phenylhydrazine?
5. Why would D-glucose and D-mannose be expected to form the same osazone upon reaction with phenylhydrazine?

6. Phenylhydrazine and its hydrochloride are both listed as a potentially carcinogenic compounds.
   True _____  False _____

7. What action should be taken if phenylhydrazine comes in contact with your skin?

8. Underline the media that are appropriate for extinguishing fires involving glucose, fructose, and phenylhydrazine hydrochloride:
   Water   Carbon dioxide   Chemical powder   Foam
Section 24.3 SYNTHESIS OF THE PROTECTED DIPEPTIDE ALA-PHE-OME. PART A. PREPARATION OF N-tert-BUTOXYCARBONYL-L-ALANINE

NAME (print): ___________________________________________ DATE: __________________
INSTRUCTOR: ______________________ LABORATORY SECTION: ________________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Determine the limiting reagent in this reaction. Show your work.

3. Is the product of the reaction an acidic, basic or neutral compound?

4. Upon completion of the reaction, the aqueous solution is basic. Write the structures of all of the compounds derived from the two starting materials that you expect to be present in solution.

5. Which of the compounds in your answer to the previous question are separated from your product in the first step of the work-up in which the aqueous and organic layers are separated?
6. Why is it necessary to acidify the aqueous layer before you can isolate the product of the reaction?

7. What are possible effects if di-tert-buty carbonate gets in your eyes?

8. What are possible effects if di-tert-buty carbonate gets on your skin?

9. What action should you take if di-tert-buty carbonate gets on your skin?

10. Is di-tert-buty carbonate listed as a possible carcinogen?

11. What is the melting point of L-alanine?
Section 24.3 SYNTHESIS OF THE PROTECTED DIPEPTIDE ALA-PHE-OME. PART B. PREPARATION OF METHYL L-PHENYLALANINATE HYDROCHLORIDE

NAME (print): ________________________________ DATE: __________________

INSTRUCTOR: ____________________________ LABORATORY SECTION: __________

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Thionyl chloride reacts readily with water. Write the balanced equation for this reaction.

3. Determine the limiting reagent in this reaction. Show your work.

4. Is the product of the reaction an acidic, basic or neutral compound?

5. Is L-phenylalanine listed as a possible mutagen?

6. What are the possible effects of ingesting methanol?
7. What are the possible effects of inhaling methanol?

8. What is the melting point of L-phenylalanine?

9. What action should you take if thionyl chloride gets on your skin?

10. Underline the media that are appropriate for extinguishing fires involving methanol:

    Water   Carbon dioxide   Chemical powder   Foam
Section 24.3  SYNTHESIS OF THE PROTECTED DIPEPTIDE ALA-PHE-OME. PART C. PREPARATION OF METHYL N-tert-BUTOXYCARBONYL L-ALANYL-L-PHENYLALANINATE

NAME (print):  ..................................................  DATE:  ..........................
INSTRUCTOR:  ..................................................  LABORATORY SECTION:  .................

1.  Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction,  
(b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic  
substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a  
reduction of the aromatic substrate, (h) none of these.

2.  Determine the limiting reagent in this reaction. Show your work.

3.  Is the product of the reaction an acidic, basic or neutral compound?

4.  Why is N-methylmorpholine used both in preparing Solution A and for the coupling reaction?

5.  A step in the work-up of the reaction involves partitioning the reaction mixture between water and diethyl ether.  
Write the structures of all of the compounds derived from the three starting materials that you expect to be  
present in the aqueous and ethereal layers.
6. A step in the work-up of the reaction involves washing the ethereal layer with 1 M HCl. Write the structure(s) of the compound(s) that might be extracted into the aqueous layer in this step.

7. A step in the work-up of the reaction involves washing the ethereal layer with saturated sodium bicarbonate. Write the structure(s) of the compound(s) that might be extracted into the aqueous layer in this step.

8. What are the possible effects of inhaling isobutyl chloroformate?

9. What are the possible effects of inhaling N-methylmorpholine?

10. Is N-methylmorpholine listed as a possible mutagen?
Section 24.3 SYNTHESIS OF THE PROTECTED DIPEPTIDE ALA-PHE-OME. PART D. PREPARATION OF METHYL L-ALANYL-L-PHENYLALANINATE TRIFLUOROACETATE

NAME (print): _______________________________ DATE: ___________________  
INSTRUCTOR: ___________________ LABORATORY SECTION: ___________________  

1. Mechanistically, the reaction to be performed (underline all answers that apply) is (a) an elimination reaction, (b) a nucleophilic substitution reaction, (c) a nucleophilic addition-elimination reaction, (d) an electrophilic substitution reaction, (e) a free-radical substitution reaction, (f) an oxidation of the aromatic substrate, (g) a reduction of the aromatic substrate, (h) none of these.

2. Determine the limiting reagent in this reaction. Show your work.

3. Is the product of the reaction an acidic, basic or neutral compound?

4. What is the purpose of adding diethyl ether to the flask after concentrating the solution after completion of the reaction?

5. What are the possible effects of inhaling diethyl ether?
6. What are the possible effects of inhaling trifluoroacetic acid?

7. What are the possible effects of inhaling dichloromethane?

8. Is trifluoroacetic acid listed as a possible mutagen?