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. Give 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).

Resorcinol _____ Benzoic acid _____ Naphthalene _____ 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 ^{o}C) are, respectively, _____ and

.

6. List the effect each of the following has if it 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. 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?

e. use a *fluted* filter paper for hot filtration?

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

Sections 4.3 and 4.4 SIMPLE AND FRACTIONAL DISTILLATION (Miniscale)

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

1. 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).

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

3. 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.)

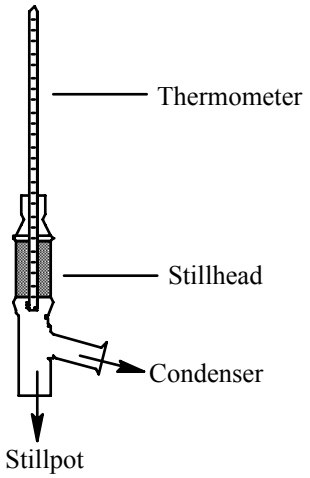
4. Two fractionating columns are each 40 cm in length. Column A has HETP = 2 cm and column B has HETP = 20 cm. By marking in the space provided, indicate whether column A or B would be more suitable to separate a binary mixture in which the components differ in boiling point by 10 °C?

_____

5. Define the term, *reflux ratio*.

6. Why is it important to align the fractionating column as nearly vertical as possible?

7. 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 4.4 SIMPLE DISTILLATION (Microscale)

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

1. Indicate whether each of the following statements is true (T) or false (F).

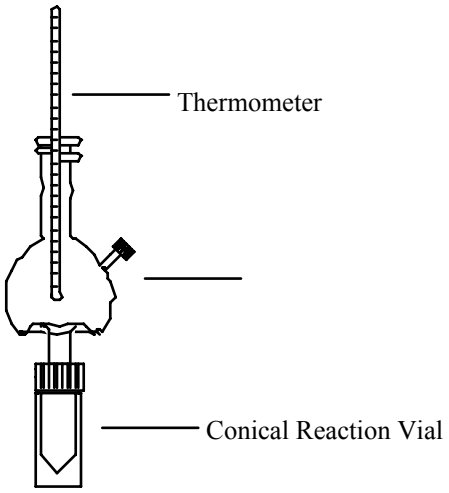
_____ **a.** Simple distillation may be used to prepare drinking water from sea water.

_____ **b.** Simple distillation may be used to remove diethyl ether, bp 35 °C (760 torr), from *p*-dichlorobenzene, bp 174 °C (760 torr).

_____ **c.** Simple distillation may be used to separate benzene, bp 80 °C (760 torr), from toluene, 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 possible effects of ingesting cyclohexane.

8. What action should you take if cyclohexane gets in your eyes?

Section 5.3 BASE EXTRACTION. 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 2.5 *M* sodium hydroxide than in diethyl ether.

_____ **d.** Naphthalene is more soluble in diethyl ether than is sodium benzoate.

_____ **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. 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?

Section 5.3 EXTRACTION. Part B. TWO-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. 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 EXTRACTION. 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:

6. 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.

7. List possible effects of inhaling excessive amounts of dichloromethane.

Section 6.3 COLUMN CHROMATOGRAPHY

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

1. What difficulty may result if

a. 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 7.3 ISOMERIZATION OF DIMETHYL MALEATE TO DIMETHYL FUMARATE

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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

2 . 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.

3. 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 CH_2Cl_2, dimethyl maleate _____ dimethyl fumarate_____?

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

5. State the recommended procedure in this experiment to be used for destroying residual bromine and write the equation for the reaction that occurs.

6. What is to be done if the melting point of the isolated dimethyl fumarate is lower than the reported melting point of this compound?

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

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

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

10. 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 9.3 BROMINATION: SELECTIVITY OF HYDROGEN ATOM ABSTRACTION

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

1. Draw a structure containing the specified type of hydrogen atom and circle the atom.

a. 2° aliphatic hydrogen atom.

b. 1° benzylic hydrogen atom.

c. vinylic hydrogen atom.

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

3. 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 Br_2 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.

9. The flash point (°C) of toluene is _____; that of methylcyclohexane is _____.

10. Underline the media that are appropriate for extinguishing fires involving toluene, *tert*-butylbenzene and ethylbenzene: Water Carbon dioxide Chemical powder Foam

Section 10.3 DEHYDRATION OF ALCOHOLS

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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

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

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

4. Why is there an upper limit to the temperature at which the alkene(s) is (are) to be collected?

5. Write equations for the chemical reaction(s) that you will use to demonstrate the presence of alkene(s) in your distilled product.

6. Why is it important to dry the crude alkene(s) 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 or the isomeric methylpentenes formed by dehydration of the corresponding alcohols: Water Carbon dioxide Chemical powder Foam

10. Specify whether the alkene or alkenes 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 the alcohol that you are to dehydrate.

Section 10.6 BROMINATION OF (*E*)-STILBENE (Discovery)

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 procedes?

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 (*E*)-STILBENE (Discovery/Green Option)

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 Br_2??

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 10.7 HYDRATION OF NORBORNENE

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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?

12. The flash point (°C) of norbornene is _____.

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. A 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 (+)-α-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 11.2 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 14.4 PREPARATION OF 1-BROMOBUTANE: AN S_N2 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_N1 or S_N2 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.

5. (cont.)

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.

_____

Section 14.5 PREPARATION OF 2-CHLORO-2-METHYLBUTANE: AN S_N1 REACTION

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 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

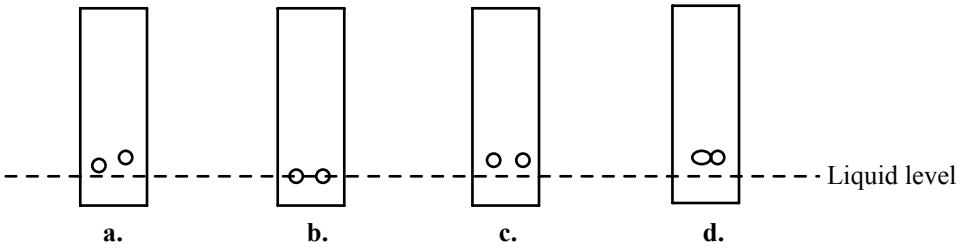
NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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.

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.

Section 15.4 NITRATION OF BROMOBENZENE. Part C. COLUMN CHROMATOGRAPHY

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 RELATIVE RATES OF ELECTROPHILIC AROMATIC SUBSTITUTION. PART A. QUALITATIVE ANALYSIS

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?

Section 15.5 RELATIVE RATES OF ELECTROPHILIC AROMATIC SUBSTITUTION. PART B. QUANTITATIVE ANALYSIS

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 17.4 REDUCTION OF 9-FLUORENONE

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 18.3 SYNTHESIS 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 *p*-anisaldehyde listed as a mutagen?

7. List the possible effects of ingesting methanol.

Section 20.2 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 stucture 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 20.4 PREPARATION AND CHEMILUMINESCENCE OF LUMINOL

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

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 SYNTHESIS OF SULFANILAMIDE. 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 SYNTHESIS OF SULFANILAMIDE. 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 SYNTHESIS OF SULFANILAMIDE.
Part C. PREPARATION OF 4-ACETAMIDO-BENZENESULFONYL 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).

Section 21.2 SYNTHESIS OF SULFANILAMIDE.
Part D. PREPARATION OF 4-ACETAMIDO-BENZENESULFANILAMIDE

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 SYNTHESIS OF SULFANILAMIDE. 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 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, $CH(C_6H_5)CH_3$.

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

NAME (print): _____ DATE: _____

INSTRUCTOR: _____ LABORATORY SECTION: _____

1. Why is decanedioyl dichloride rather than decanedoic acid used in this experiment?

2. Explain how reaction between decanedioyl 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 decanedioyl 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 decanedioyl 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 decanedioyl dichloride.