SECTION 2. CHEMICALS AND SPECIAL EQUIPMENT BY CHAPTERS

The amount provided is that required for 10 students. The equipment listed in this section is that needed in addition to the standard equipment listed in Section 1.

CH 3 Solids: Recrystallization and Melting Points.

3.2 Recrystallization

| Chemicals: | Quantity | |
|--------------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Acetanilide | | 0.60 g |
| Benzoic acid | | 0.60 g |
| Naphthalene | | 0.60 g |
| Resorcinol | | 0.60 g |
| Petroleum ether (bp 60–80 °C) | | 30 mL |
| Impure benzoic acid | 10 g | 1.0 g |
| Impure acetanilide | 10 g | 1.0 g |
| Impure naphthalene | 10 g | 1.0 g |
| Decolorizing carbon | 1–2 g | 0.5 g |
| Filter aid (Celite) | 10 g | 2.0 g |
| Methanol, 95% ethanol, or 2-propanol | 250 mL | 30 mL |

Other solvents, e.g., those listed in Table 3.1, may be needed if an unknown compound has been assigned for recrystallization. About 500 mL of each such solvent is required per *10* students.

3.3 Physical Constants: Melting Points.

Parts A/B. Melting Points.

| Chemicals (Standards for calibration): | Quantity |
|--|----------|
| 3-Phenylpropanoic acid | 0.2 g |
| Acetamide | 0.2 g |
| Acetanilide | 0.2 g |
| Benzamide | 0.2 g |
| Salicylic acid | 0.2 g |
| 4-Chloroacetanilide | 0.2 g |
| 3,5-Dinitrobenzoic acid | 0.2 g |
| Equipment: | Quantity |
| Thiele tubes | 10 |
| or electric melting point apparatus | 1 |
| Packing tubes | 10 |
| | |

Parts A/B. Melting Points. (cont.)

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Equipment: Quantity
Capillary tubes 100

Compounds for Melting Point Unknowns

| Compound | Melting point (°C) |
|---------------------------------|--------------------|
| 1,3-Dinitrobenzene | 90 |
| Acetanilide | 114 |
| Benzoic acid | 122 |
| Benzamide | 130 |
| Phthalic anhydride | 131 |
| Urea | 132 |
| trans-Cinnamic acid | 133 |
| p-Acetophenetidide (phenacetin) | 135 |
| o-Chlorobenzoic acid | 142 |
| Salicylamide | 142 |
| Benzilic acid | 150 |
| Adipic acid | 153 |
| Salicylic acid | 158 |
| Benzanilide | 163 |
| p-Bromoacetanilide | 167 |
| p-Toluic acid | 178 |
| Succinic acid | 188 |
| 3,5-Dinitrobenzoic acid | 207 |

Part C. Who Else Has My Compound?

Suggested compounds for unknowns. All compounds should be colorless, and samples should be numbered in such a way that they cannot easily be decoded. Samples should be dispensed such that there are two or three students per compound in the lab.

| | Quantity/3 students |
|----------------------------------|---------------------|
| Acetanilide | 0.6 g |
| Ethyl p-Hydroxybenzoate | 0.6 g |
| Urea | 0.6 g |
| (E)-Cinnamic acid | 0.6 g |
| Phenacetin | 0.6 g |
| Aspirin (sodium acetylsalicylate | 0.6 g |
| <i>p</i> -Phenylphenol | 0.6 g |

Part C. Who Else Has My Compound? (cont.)

| | Quantity/3 students | |
|--|---------------------|--|
| 4-Hydroxyacetanilide | 0.6 g | |
| p-Toluic Acid | 0.6 g | |
| p-Anisic Acid | 0.6 g | |
| Equipment: | Quantity | |
| Thiele tubes | 10 | |
| or electric melting point apparatus | 1 | |
| Packing tubes | 10 | |
| Melting-point capillaries | 50 | |
| TLC chambers | 10 | |
| $250~\mu m$ pre-coated silica gel TLC plates with fluorescence indicator | | |
| cut into \sim 3-cm x 10-cm strips | 60 strips | |
| Capillary pipets | 20 | |

Compounds for Melting Point Unknowns

| Compound | Melting point (°C) |
|-------------------------|---------------------------|
| Acetanilide | 113–115 |
| Ethyl p-hydroxybenzoate | 114–117 |
| Urea | 132–135 |
| trans-Cinnamic acid | 132–135 |
| Phenacetin | 133–136 (<i>dec.</i>) |
| Aspirin | 134–136 |
| <i>p</i> -Phenyphenol | 164–166 |
| 4-Hydroxyacetanilide | 168–172 |
| <i>p</i> -Toluic acid | 177–180 |
| <i>p</i> -Anisic acid | 182–185 |

CH 4 Liquids: Distillation and Boiling Points

4.2 Boiling Points of Pure Liquids

Chemicals:

Suggestions for possible boiling point unknowns are provided below.

| Equipment: | Quantity |
|--|----------|
| Thiele tubes | 10 |
| Capillary tubes for micro boiling points | 20 |
| 6- to 8-mm Tubing for samples | 10 |

4.2 Boiling Points of Pure Liquids (cont.)

Compounds for Boiling Point Knowns and Unknowns

| Compound | Boiling point (°C) |
|--|--------------------|
| Ethanol | 78 |
| 1-Chlorobutane | 78 |
| 2-Butanone (methyl ethyl ketone) | 80 |
| Cyclohexane | 81 |
| 2-Propanol | 83 |
| 2-Methyl-2-propanol (tert-butyl alcohol) | 83 |
| Methyl isobutyrate | 93 |
| Heptane | 98 |
| 2-Butanol | 100 |
| 2-Methyl-2-butanol | 102 |
| 2-Methyl-1-propanol | 108 |
| Toluene | 111 |
| 1-Butanol | 118 |
| Acetic acid | 118 |
| Tetrachloroethylene | 131 |
| Chlorobenzene | 132 |
| 4-Methyl-2-pentanol | 132 |
| Ethylbenzene | 136 |
| Isopropylbenzene | 152 |
| Cyclohexanone | 156 |
| Bromobenzene | 156 |
| Anisole | 156 |
| Cyclohexanol | 161 |
| tert-Butylbenzene | 168 |
| sec-Butylbenzene | 172 |
| Isobutylbenzene | 172 |
| 1,3-Dichlorobenzene | 179 |
| Ethyl acetoacetate | 181 |
| <i>n</i> -Butylbenzene | 183 |

4.3 and 4.4 Simple and Fractional Distillation

Chemicals: Quantity

Miniscale Microscale

Simple distillation:

Cyclohexane with non-volatile dye 100 mL 20 mL

Fractional distillation:

Cyclohexane 100 mL
Toluene 200 mL

Equipment:

Copper or stainless steel gauze, Raschig rings or other

column packings

Aluminum foil and/or glass wool (optional) for

insulating columns

4.6 Steam Distillation of Citral from Lemon Grass Oil

Chemicals: Quantity

Lemon grass oil 25 mL

Diethyl ether, *solvent grade* 300 mL

Calcium chloride, *anhydrous*, granular 5–10 g

Chemicals for unsaturation tests (see 4.7A1)

Equipment:

Apparatus for steam distillation using an internal steam source 10

4.7 Qualitative Analysis.

Part A. Tests for Unsaturation

1. Bromine in Dichloromethane

Chemicals: Quantity

Dichloromethane 25 mL

Bromine 0.01 mL

To prepare a 0.1 M solution of Br₂ in CH₂Cl₂, dissolve 0.01 mL of Br₂ in 10 mL of CH₂Cl₂; store the solution in a tightly stoppered container.

2. Potassium Permanganate

Chemicals:

Water, distilled

Potassium permanganate

Ethanol, 95%

Quantity

2 mL

0.032 g

40 mL

Dissolve 0.32 g of KMnO₄ in 20 mL of distilled water to give a 0.1 M aqueous solution.

4.7 Qualitative Analysis (cont.)

Part B. Test for Aldehyde Function

Chromic Acid

| Chemicals: | Quantity |
|-----------------------------|----------|
| Chromic anhydride | 10 g |
| Sulfuric acid, concentrated | 10 mL |
| Water, distilled | 30 mL |

To prepare chromic acid, add 1 g of chromic anhydride to 1 mL of *concentrated* H_2SO_4 and stir the mixture until a smooth paste is obtained. Then *cautiously* dilute the paste with 3 mL of distilled H_2O and stir this mixture until a clear orange solution is obtained.

CH 5 Extraction

5.3 Base and Acid Extractions

| Chemicals: | Quantity | |
|------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Benzoic acid | 22 g | 3 g |
| Naphthalene | 22 g | 3 g |
| 2-Naphthol | 7 g | 1 g |
| 4-Nitroaniline | 5 g | 1 g |
| Diethyl ether, solvent grade | 750 mL | 50 mL |
| Dichloromethane | 400 mL | 30 mL |
| Sodium bicarbonate, 1.25 M | 200 mL | 10 mL |
| Sodium hydroxide, 2.5 M | 350 mL | 10 mL |
| Sodium hydroxide, 6 M | 750 mL | 30 mL |
| Hydrochloric acid, 3 M | 250 mL | |
| Hydrochloric acid, 6 M | 750 mL | 30 mL |
| Hydrochloric acid, 12 M | | 5 mL |
| Sodium Sulfate, anhydrous | 10 g | 5 g |

5.4 Isolation of Trimyristin from Nutmeg

Acetone

| Quar | |
|-----------|----------------|
| Miniscale | Microscale |
| 40 g | 10 g |
| 150 mL | 50 mL |
| | Miniscale 40 g |

50 mL

15 mL

CH 6 Chromatography

6.2 Thin-Layer Chromatography

Part A. Separation of Spinach Pigments by TLC

| Chemicals: | Quantity |
|--|----------|
| Green leaves | 10 |
| Petroleum ether (30–60 °C) | 150 mL |
| Ethanol, absolute | 30 mL |
| Sodium sulfate, anhydrous | 10 g |
| Chloroform | 100 mL |
| Acetone | 100 mL |
| Equipment: | |
| Bottle, wide-mouth, for developing chamber | 10 |
| Eastman Type K301R2 Chromagram sheet or equivalent | 1 sheet |
| Part B. Separation of Syn- and Anti-Azobenzenes by TLC | |
| Chemicals: | Quantity |
| Azobenzene solution in toluene, 10% | 10 mL |
| Petroleum ether (30–60 °C) | 100 mL |
| Acetone | 100 mL |
| Chloroform | 100 mL |
| Equipment: | |
| Bottle, wide-mouth, for developing chamber | 10 |
| Eastman Type K301R2 Chromatogram sheet or equivalent | 1 sheet |
| Sunshine or sun lamp | |
| Column Chromatography | |

6.3 Column Chromatography

| Chemicals: | Quantity |
|----------------------------|----------|
| Alumina | 50 g |
| Sand | 10 g |
| Petroleum ether (60–80 °C) | 1 L |
| Fluorene | 1 g |
| 9-Fluorenone | 1 g |
| Dichloromethane | 125 mL |
| Equipment: | |
| 50-mL Buret | 10 |
| Glass wool or cotton | |
| Erlenmeyer flasks, 50-mL | 30 |

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6.4 Gas-Liquid Chromatography

Part A. Qualitative and Quantitative Analyses of a Mixture of Compounds by GLC

| Chemicals: | Quantity |
|-------------------|----------|
| Ethyl acetate | 10 mL |
| Ethanol, absolute | 10 mL |
| n-Butyl acetate | 10 mL |
| Ethylbenzene | 10 mL |
| Isopropylbenzene | 10 mL |
| Toluene | 10 mL |

Part B. Determining GLC Response Factors

A selection of the same chemicals required for **Part A**.

Equipment:

Gas chromatograph, equipped with column and recorder

Syringes, 1–10 μL capacity

Syringe, gas-tight

CH 7 Stereoisomers

7.2 Separation of Diastereomeric 1,2-Cyclohexanediols

| Chemicals: | Quantity |
|---|-----------------|
| 1,2-Cyclohexanediol, commercial mixture of cis- and | |
| trans-isomers | <i>ca</i> . 1 g |
| trans-1,2-Cyclohexanediol, 98% | ca. 1 g |
| Acetone | 20 mL |
| Petroleum ether, bp 60–80 °C | 75 mL |
| 2-Propanol | 25 mL |
| Iodine | 1 g |
| Equipment: | |
| Eastman Type K301R2 Chromagram sheet or equivalent | 1 sheet |
| Bottle, wide-mouth, for developing chamber | 10 |

7.3 Isomerization of Dimethyl Maleate to Dimethyl Fumarate

| Chemicals: | Quantity | |
|-----------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Dimethyl maleate | 15 mL | 5 mL |
| Bromine in dichloromethane, 0.6 M | 20 mL | |
| Bromine in dichloromethane, 0.1 M | | 10 mL |

1

7.3 Isomerization of Dimethyl Maleate to Dimethyl Fumarate (cont.)

| Chemicals: | Quantity |
|------------|----------|
|------------|----------|

| | Miniscale | Microscale |
|-----------------|-----------|------------|
| Dichloromethane | 10 mL | 5 mL |
| Ethanol, 95% | 50 mL | 10 mL |
| Cyclohexene | 10 mL | 5 mL |

Equipment:

100-watt unfrosted light bulb and socket

7.4 Properties of the Enantiomeric Carvones.

Part A. Properties of the Enantiomeric Carvones

Chemicals: Quantity

Spearmint and/or caraway seed oil 150 mL (140 g)

(Suppliers of the essential oils are listed in the Thomas

Register or in Chem Sources U.S. A. One vendor is Pfaltz

& Bauer, Inc.)

Bromine in dichloromethane, 0.1 M 10 mL

To prepare a 0.1 M solution of Br₂ in CH₂Cl₂, dissolve 0.01 mL of Br₂ in 10 mL of CH₂Cl₂;

keep the solution in a tightly stoppered container.

Equipment:

Manometer

Gas chromatograph

Polarimeter

Part B. Formation of Carvone 2,4-Dinitrophenylhydrazone

| Chemicals: | Quantity |
|-----------------------------------|----------|
| Spearmint and/or caraway seed oil | 6 mL |
| 2,4-Dinitrophenylhydrazine | 6 g |
| Sulfuric acid, concentrated | 30 mL |
| Ethanol, 95% | 350 mL |
| Ethyl acetate | 50 mL |

7.6 Resolution of Racemic 1-Phenylethanamine

| Chemicals: | Quantity |
|-----------------------------|----------|
| 1-Phenylethanamine, racemic | 125 g |
| Methanol | 3.0 L |
| (+)-Tartaric acid | 156 g |

7.6 Resolution of Racemic 1-Phenylethanamine (cont.)

| Chemicals: | Quantity |
|---------------------------|----------|
| Sodium hydroxide, 14 M | 80 mL |
| Ether, solvent grade | 1.5 L |
| Sodium chloride | 55 g |
| Sodium sulfate, anhydrous | 30 g |
| Ethanol, absolute | 300 mL |

Equipment:

Polarimeter

CH 9 Alkanes

9.2 Free-Radical Chain Chlorination of 1-Chlorobutane

| Chemicals: | Quantity | |
|---|-----------|------------|
| | Miniscale | Microscale |
| 1-Chlorobutane | 50 mL | 5 mL |
| Sulfuryl chloride | 20 mL | 2 mL |
| 1,1'-Azobis(cyclohexanenitrile) | 2.0 g | 0.2 g |
| Sodium carbonate, 0.5 M (100 g of $Na_2SO_4/4$ L of | | |
| solution) | 100 g | 10 g |
| Sodium sulfate, anhydrous | 50 g | 5 g |
| Sodium chloride solution (brine) | 300 mL | 20 mL |
| Equipment: | | |
| Glass wool, Pyrex | | |
| Gas trap | 10 | 10 |
| | | |

9.3 Relative Rates of Free-Radical Chain Bromination

| Chemicals: | Quantity |
|-----------------------------------|----------|
| Toluene | 5 mL |
| Ethylbenzene | 5 mL |
| Isopropylbenzene | 5 mL |
| tert-Butylbenzene | 5 mL |
| Cyclohexane | 5 mL |
| Methylcyclohexane | 5 mL |
| Dichloromethane | 360 mL |
| Bromine in dichloromethane, $1 M$ | 70 mL |

Equipment:

100- or 150-watt unfrosted light bulb and socket

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CH 10 Alkenes

10.2 Dehydrohalogenation of Alkyl Halides

Part A. Elimination with Alcoholic Potassium Hydroxide

| Chemicals: | Qua | Quantity | |
|------------|-----------|------------|--|
| | Miniscale | Microscale | |

Potassium hydroxide in 1-propanol, 4 M 250 mL 25 mL 2-Bromo-2-methylbutane 25 mL 10 mL

Part B. Elimination with Potassium tert-Butoxide

Chemicals: Quantity

Miniscale Microscale

Potassium tert-butoxide in anhydrous tert-butyl

alcohol, 1 N 250 mL 2-Bromo-2-methylbutane 25 mL

Qualitative Tests

Chemicals: Quantity

Cyclohexene 2 g

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

10.3 Dehydration of Alcohols

Part A. Dehydration of 4-Methyl-2-pentanol

Chemicals: Quantity

 $\begin{tabular}{lll} \it Miniscale &\it Microscale \\ \it 4-Methyl-2-pentanol &\it 40 mL \\ \it Sulfuric acid, 9 \it M (50:50 concentrated H_2SO_4:H_2O) &\it 25 mL \\ \end{tabular}$

Potassium carbonate, *anhydrous* 20 g

Part B. Dehydration of Cyclohexanol

Chemicals: Quantity

| | Miniscale | Microscale |
|---|-----------|------------|
| Cyclohexanol | 50 mL | 10 mL |
| Sulfuric acid, 9 M (50:50 concentrated H_2SO_4 : H_2O) | 25 mL | 5 mL |
| Potassium carbonate, anhydrous | 20 g | 2 g |

Qualitative Tests

Chemicals: Quantity

Cyclohexene 2 g

Bromine in dichloromethane solution (see Section 4.7A1)

Qualitative Tests (cont.)

Chemicals:

Baeyer test (see Section 4.7A2)

10.5 Addition of Hydrobromic Acid to Alkenes

Part A. Addition of Hydrogen Bromide to 1-Hexene

| Chemicals: | Quantity | |
|---|-----------|------------|
| | Miniscale | Microscale |
| 1-Hexene | 30 mL | 5 mL |
| Hydrobromic acid, concentrated | 140 mL | 20 mL |
| Methyltrioctylammonium chloride | 10 g | 1.5 g |
| Petroleum ether (30–60 °C) | 150 mL | 10 mL |
| Sodium bicarbonate, 10% (50 g of NaHCO ₃ /500 mL | | |
| of solution) | 300 mL | 10 mL |
| Sodium sulfate, anhydrous | 20 g | 2 g |

Part B. Qualitative Analysis of Alkyl Halides

1. Silver Nitrate Test

| Chemicals: | Quantity |
|----------------|----------|
| Silver nitrate | 0.4 g |
| Ethanol, 85% | 20 mL |

To prepare a 0.1 M solution of AgNO₃ in ethanol, dissolve 0.4 g of AgNO₃ in 20 mL of 95% ethanol; store the solution in a dark bottle.

2. Sodium Iodide Test

| Chemicals: | Quantity |
|---------------|----------|
| Sodium iodide | 1.5 g |
| Acetone | 10 mL |

To prepare a 1 *M* solution of NaI in ethanol, dissolve 1.5 g of NaI in 10 mL of acetone; store the solution in a dark bottle.

10.6 Bromination of Alkenes

Part A. Bromination of (E)-Stilbene

| Chemicals: | Quantity | |
|--------------------------------|-----------|------------|
| | Miniscale | Microscale |
| (E)-Stilbene | 9 g | 1.8 g |
| Dichloromethane | 125 mL | 25 mL |
| Bromine in dichloromethane 1 M | 50 mL | 10 mL |

5 g

20 g

20 g

Part B. Bromination of (E)-Stilbene: The Green Approach

| | Tart b. brommation of (E)-Stilbenc. The Orech Ap | pproach | |
|------|--|-----------|------------|
| | Chemicals: | Quantity | |
| | | Miniscale | Microscale |
| | (E)-Stilbene | 6 g | 1.5 g |
| | Hydrobromic acid, concentrated | 15 mL | 5 mL |
| | Hydrogen peroxide, 30% | 10 mL | 3 mL |
| | Ethanol, <i>95%</i> | 140 mL | 35 mL |
| | Xylene | 100 mL | 25 mL |
| | Equipment: | Quantity | |
| | | Miniscale | Microscale |
| | Pipet, 1-mL, graduated | 10 | 10 |
| | Pipet, 2 mL, graduated | 10 | |
| | Part C. Bromination of (E) -Cinnamic Acid | | |
| | Chemicals: | Quantity | |
| | | Miniscale | Microscale |
| | (E)-Cinnamic acid | 8 g | 1.5 g |
| | Acetic acid, glacial | 100 mL | 15 mL |
| | Pyridinium tribromide | 17.6 g | 3.3 g |
| | Sodium bisulfite, 10% aqueous | 50 mL | 10 mL |
| | 1:1 95% EtOH:H ₂ O | 200 mL | 40 mL |
| 10.7 | Hydration of Norbornene | | |
| | Chemicals: | Qua | ntity |
| | Sulfuric acid, concentrated | 20 | mL |
| | Norbornene | 1 | 10 g |
| | Potassium hydroxide | 1 | 15 g |
| | Diethyl ether, solvent grade | 250 | mL |
| | | | |

Sodium sulfate 10.8 Hydroboration-Oxidation of Alkenes

Sodium bicarbonate Sodium chloride

Part A. Hydroboration-Oxidation of (+)-α-Pinene

| Chemicals: | Quar | Quantity | |
|--------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Borane in THF, 1 M | 50 mL | 10 mL | |
| Tetrahydrofuran | 20 mL | 5 mL | |
| Calcium chloride | 100 g | 10 g | |

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Part A. Hydroboration-Oxidation of (+)-α-Pinene (cont.)

| Chemicals: | cals: Quantity | |
|--|----------------|------------|
| | Miniscale | Microscale |
| (+)-α-Pinene | 16 mL | 3 mL |
| Hydrogen peroxide, 30% | 15 mL | 3 mL |
| Sodium hydroxide, 3 M (120 g of NaOH/100 mL of | | |
| solution) | 15 mL | 3 mL |
| Diethyl ether, solvent grade | 200 mL | 40 mL |
| Sodium chloride | 4 g | 1 g |
| Sodium sulfate, anhydrous | 4 g | 1 g |
| Saturated brine | 200 mL | 40 mL |
| Equipment: | | |
| Rubber septum | | 10 |
| Magnetic stirrer | 10 | |
| Glass syringe | 20 | |
| Part B. Preparation of Urethanes | | |
| Chemicals: | Qua | intity |
| Phenyl isocyanate or α -naphthyl isocyanate | 5 mL | |
| Pyridine | 1 mL | |
| Petroleum ether (60–80 °C) | 50 r | nL |

CH 11 Alkynes

11.2 Dehydrobromination of meso-Stilbene Dibromide

| Chemicals: | Qua | Quantity | |
|----------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| meso-Stilbene dibromide | 8 g | 1.5 g | |
| Potassium hydroxide | 4 g | 0.8 g | |
| Triethylene glycol | 40 mL | 10 mL | |
| Boiling stone, carborundum | 10 | 10 | |
| Ethanol, <i>95%</i> | 100 mL | 20 mL | |

Qualitative Tests

Chemicals: Quantity

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Cyclohexene 2 g

11.3 Preparation of 3-Hydroxy-3-methyl-2-butanone

| Quantity |
|----------|
| 30 mL |
| 2 g |
| 36 mL |
| 30 g |
| 100 g |
| 200 mL |
| 5 g |
| 8 g |
| 50 mL |
| |

11.4 Formation of a Silver Acetylide and Its Decomposition

| Chemicals: | Quantity |
|--------------------------|----------|
| Silver nitrate, 0.1 M | 25 mL |
| Ammonium hydroxide | 50 mL |
| 2-methyl-3-butyn-2-ol | 1 mL |
| Hydrochloric acid dilute | 50 mL |

CH 12 Dienes. The Diels-Alder Reaction

12.3 Applications of Diels-Alder Reactions

Part A. Reaction of 1,3-Butadiene and Maleic Anhydride

| Chemicals: | Qua | Quantity | |
|----------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| 3-Sulfolene | 25 g | 2.5 g | |
| Maleic anhydride | 15 g | 1.5 g | |
| Xylene, anhydrous | 110 mL | 15 mL | |
| Petroleum ether (60–80 °C) | 200 mL | 20 mL | |

Qualitative Tests

Chemicals: Quantity

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Cyclohexene 6 g

Part B. Reaction of 1,3-Cyclopentadiene and Maleic Anhydride

Chemicals: Quantity

Miniscale Microscale

Dicyclopentadiene 70 mL 10 mL

Part B. Reaction of 1,3-Cyclopentadiene and Maleic Anhydride (cont.)

| Chemicals: | Qua | Quantity | |
|----------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Calcium chloride | 5 g | | |
| Maleic anhydride | 15 g | 1 g | |
| Petroleum ether (60–80 °C) | 50 mL | 4 mL | |
| Ethyl acetate | 60 mL | 4 mL | |

Qualitative Tests

Chemicals: Quantity

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Cyclohexene 6 g

Part C. Hydrolysis of Anhydrides

1. 1,4-Cyclohexene-cis-1,2-dicarboxylic Acid

Chemical: Quantity

Miniscale Microscale

4-Cyclohexene-*cis*-1,2-dicarboxylic anhydride 10 g 1g

Qualitative Tests

Chemicals: Quantity

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Cyclohexene 6 g

2. Bicyclo[2.2.1]hept-5-ene-endo-2,3-dicarboxylic Acid

Chemical: Quantity

Miniscale Microscale

Bicyclo[2.2.1]hept-5-en-endo-1,2-dicarboxylic

anhydride 10 g 1g

Qualitative Tests

Chemicals: Quantity

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Cyclohexene 2 g

25 mL

4 mL

CH 13 Kinetic and Thermodynamic Control of a Reaction

Ethanol, 95%

2-Furaldehyde

Part A. Preparation of Cyclohexanone Semicarbazone

| Quantity |
|----------|
| 5 g |
| 10 g |
| |
| 5 mL |
| 25 mL |
| |
| Quantity |
| 5 g |
| 10 g |
| |

Part C. Reactions of Semicarbazide with Cyclohexanone and 2-Furaldehyde in Phosphate Buffer Solution

| Chemicals: | Quantity |
|-----------------------------|----------|
| Semicarbazide hydrochloride | 30 g |
| Dibasic potassium phosphate | 60 g |
| Cyclohexanone | 30 g |
| 2-Furaldehyde | 30 g |
| Ethanol, 95% | 150 mL |

Part D. Reactions of Semicarbazide with Cyclohexanone and 2-Furaldehyde in Bicarbonate Buffer Solution

| Chemicals: | Quantity |
|-----------------------------|----------|
| Semicarbazide hydrochloride | 20 g |
| Sodium bicarbonate | 40 g |
| Cyclohexanone | 20 mL |
| 2-Furaldehyde | 16 mL |
| Ethanol, 95% | 100 mL |

Part E. Tests of Reversibility of Semicarbazone Formation

| Chemicals: | Quantity |
|-----------------------------|----------|
| 2-Furaldehyde | 3 mL |
| Ethanol, <i>95%</i> | 40 mL |
| Cyclohexanone semicarbazone | 3 g |
| 2-Furaldehyde semicarbazone | 3 g |

Part E. Tests of Reversibility of Semicarbazone Formation (cont.)

Chemicals: Quantity
Cyclohexanone 3 mL
Equipment:

Pipet or syringe, 1-mL, graduated 10

CH 14 Nucleophilic Aliphatic Substitution: Preparation of Alkyl Halides

14.4 Preparation of 1-Bromobutane: An S_N2 Reaction

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| Sodium bromide | 111 g | 11 g |
| Sulfuric acid, concentrated | 100 mL | 10 mL |
| Sodium hydroxide, 2 M (80 g of NaOH/L of solution) | 100 mL | 10 mL |
| Sodium chloride, saturated solution | 100 mL | 10 mL |
| Sodium sulfate, anhydrous | 10 g | 1 g |

14.5 Preparation of 2-Chloro-2-methylbutane: An S_N1 Reaction

Chemicals: Quantity Miniscale Microscale 2-Methyl-2-butanol 100 mL 10 mL Hydrochloric acid, concentrated 250 mL 25 mL Sodium chloride, saturated solution 1 L 0.1 L Sodium bicarbonate 100 g 10 g

Sodium sulfate, *anhydrous* 10 g 1 g

Qualitative Tests

Alcoholic silver nitrate classification test (see Section 10.5B1)

Sodium iodide/acetone classification test (see Section 10.5B2)

14.6 Chemical Kinetics: Evidence for Nucleophilic Substitution Mechanisms

| Chemicals: | Quantity |
|------------------------------------|----------|
| 2-Chloro-2-methylbutane | 10 g |
| Phenolphthalein indicator solution | 20 mL |
| 2-Propanol | 8 L |
| Sodium hydroxide | 60 g |
| Equipment: | Quantity |
| Buret. 50-mL | 10 |

14.7 Competing Nucleophiles in S_N Reactions

| Chemicals: | Quantity |
|---|----------|
| 1-Butanol | 20 mL |
| 2-Butanol | 20 mL |
| 2-Methyl-2-propanol | 20 mL |
| Ammonium chloride, 1.5 M in 9 M sulfuric acid | 10 mL |
| Ammonium bromide, 1.5 M in 9 M sulfuric acid | 10 mL |
| Hexanes | 50 mL |
| Sodium chloride, saturated solution | 50 mL |
| Sodium bicarbonate, saturated solution | 50 mL |
| Sodium sulfate, anhydrous | 5.0 g |

14.8 Competition between Substitution and Elimination

Chemicals: Quantity

Microscale (for 2 trials. per student)

10

| 1-Bromohexane | 16.3 g |
|---|--------|
| 2-Bromohexane | 16.3 g |
| Sodium methoxide, 1.5 M in methanol | 140 mL |
| Potassium <i>tert</i> -butoxide, 1.5 <i>M</i> in <i>tert</i> -butyl alcohol | 140 mL |
| Sodium chloride, saturated solution | 30 mL |
| Diethyl ether | 30 mL |
| Sodium sulfate, anhydrous | 5.0 g |

CH 15 Arenes. Electrophilic Aromatic Substitution

Syringe, 1-mL

15.2 Friedel-Crafts Alkylation of p-Xylene with 1-Bromopropane

Chemicals: Quantity Miniscale Microscale Aluminum chloride, anhydrous 7 g 1 g p-Xylene 150 mL 20 mL Sodium sulfate, anhydrous 50 g 5 g 85 mL 1-Bromopropane 8 mL Equipment: 10 Gas trap

15.3 Friedel-Crafts Acylation of Anisole

| Chemicals: | Qua | ntity |
|-------------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Zinc oxide | 4 g | 0.4 g |
| Benzoyl chloride | 12 mL | 1.2 mL |
| Anisole | 11 mL | 1.1 mL |
| Sodium carbonate, saturated aqueous | 50 mL | 30 mL |
| Sodium chloride, saturated aqueous | 50 mL | 30 mL |
| Sodium sulfate, anhydrous | 50 g | 5.0 g |
| Dichloromethane | 100 mL | 20 mL |
| Hexanes | 250 mL | 30 mL |
| Equipment: | | |
| Syringe, 3-mL | | 20 |
| Syringe, 1-mL | | 20 |
| Centrifuge tube, 5-mL screw cap | | 20 |

15.4 Nitration of Bromobenzene

Part A. Nitration

| Chemicals: | Quantity | |
|--|-----------|------------|
| I | Miniscale | Microscale |
| Nitric acid, concentrated | 40 mL | 5 mL |
| Sulfuric acid, concentrated | 40 mL | 5 mL |
| Bromobenzene | 45 mL | 5 mL |
| Ethanol, 95% | 160 mL | 20 mL |
| Equipment: | | |
| | Quai | ntity |
| 25- or 50-mL Buret | 10 | |
| Part B. Thin-Layer Chromatography | | |
| Chemicals: | Quai | ntity |
| Dichloromethane | 10 1 | mL |
| Hexane | 36 1 | mL |
| Iodine | 1 | 0 g |
| Ethyl acetate | 4 1 | mL |
| Equipment: | | |
| | Quai | ntity |
| Eastman Type K3012R Chromagram sheet or equivale | ent 1 sh | eet |

²⁵

Part C. Column Chromatography

| Chemicals: | Quantity |
|---------------|----------|
| Silica gel | 50 g |
| Hexane | 360 mL |
| Ethyl acetate | 40 mL |
| Equipment: | |
| 50-mL Buret | 10 |

15.5 Substituent Effects on Electrophilic Aromatic Substitution

Part A. Relative Rates of Electrophilic Aromatic Bromination

1. Qualitative Measurements

| Chemicals: | Quantity |
|-----------------------------|----------|
| Phenol | 1 g |
| Anisole | 1 g |
| Diphenyl ether | 1 g |
| Acetanilide | 1 g |
| 4-Bromophenol | 1 g |
| 1-Naphthol | 1 g |
| Acetic acid, glacial | 600 mL |
| Bromine | 5 g |
| Equipment | |
| 5-mL Pipet with graduations | 10 |
| 1-L Beaker | 10 |
| Copper wire | |
| | |

2. Quantitative Measurements

| Chen | nicals: | Quantity |
|------|----------------------|----------|
| | Anisole | 3 g |
| | Diphenyl ether | 3 g |
| | Acetanilide | 3 g |
| | Acetic acid, glacial | 300 mL |
| | Bromine | 3 g |
| Equi | ipment | |
| | Colorimeter | 5 |
| | Cuvette | 15 |

Quantity

Chemicals:

Part B. Electrophilic Aromatic Bromination of Monosubstituted Arenes

| Chemicals: | Quantity |
|--|----------|
| Anisole | 1 mL |
| Toluene | 1 mL |
| Bromobenzene | 1 mL |
| Methyl benzoate | 1 mL |
| Bromine, 1 M solution in dichloromethane | 10 mL |
| Ferric bromide | 0.3 g |
| Sodium bisulfite, 10% aqueous | 50 mL |
| Sodium bicarbonate, saturated aqueous | 50 mL |
| Deuterochloroform | 10 mL |
| Equipment | Quantity |
| Gas trap | 10 |
| NMR tube | 10 |
| | |

Part C. Electrophilic Aromatic Nitration of Monosubstituted Arenes

| | 2 |
|-------------------------------------|------------|
| | Microscale |
| Toluene | 10.0 g |
| Methyl benzoate | 10.0 g |
| Chlorobenzene | 10.0 g |
| tert-Butylbenzene | 10.0 g |
| Acetanilide | 10.0 g |
| Nitric acid, concentrated | 50 mL |
| Sulfuric acid, concentrated | 50 mL |
| Acetic acid, glacial | 10 mL |
| Sodium carbonate, saturated aqueous | 500 mL |
| Dichloromethane | 250 mL |

15.6 Azo Dyes and the Chemistry of Dyeing Fabrics

| Chemicals: | Quantity |
|-----------------------------|----------|
| 2-Aminobenzenesulfonic acid | 2 g |
| 3-Aminobenzenesulfonic acid | 2 g |
| 4-Aminobenzenesulfonic acid | 2 g |
| 1-Naphthol | 1.5 g |
| 2-Naphthol | 1.5 g |
| Salicylic acid | 1.5 g |

10 strips

15.6 Azo Dyes and the Chemistry of Dyeing Fabrics (cont.)

| Chemicals: | Quantity |
|---|----------|
| Ammonium 8-anilino-1-naphthalenesulfonate | 3.5 g |
| Sodium carbonate | 1.5 g |
| Sodium nitrite | 2 g |
| Sodium chloride | 10 g |
| Hydrochloric acid, concentrated | 6 mL |
| Sodium hydroxide, 2.5 M aqueous | 20 mL |
| Sodium chloride, saturated aqueous | 20 mL |
| Equipment: | Quantity |
| Fabric, multi-fiber, No. 43 CS1 (Kimble Chase), | |
| 1"-wide strips | 10 |

CH 16 Oxidation of Alcohols and Carbonyl Compounds

Starch-iodide paper

16.2 Preparation of Aldehydes and Ketones by Oxidation of Alcohols

Part A. Oxidation of Cyclododecanol to Cyclododecanone

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| Cyclododecanol | 5 g | |
| Acetic acid, glacial | 4 mL | |
| Acetone | 12 mL | |
| Commercial bleach (5.3% sodium hypochlorite) | 60 mL | |
| Diethyl ether, solvent grade | 100 mL | |
| Sodium bicarbonate, saturated solution | 50 mL | |
| Sodium chloride, saturated solution | 50 mL | |
| Sodium bisulfite, saturated solution | 50 mL | |
| Sodium sulfate, anhydrous | 50 g | |
| Equipment: | | |

Part B. Oxidation of 4-Chlorobenzyl Alcohol to 4-Chlorobenzoic Acid

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| Calcium hypochlorite, commercial (65%) | 26 g | 6 g |
| Acetic acid, glacial | 20 mL | 3 mL |
| 4-Chlorobenzyl alcohol | 5 g | 1 g |
| Acetonitrile | 50 mL | 10 mL |

Part B. Oxidation of 4-Chlorobenzyl Alcohol to 4-Chlorobenzoic Acid (cont.)

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| Diethyl ether, solvent grade | 300 mL | 60 mL |
| Sodium bicarbonate, saturated solution | 200 mL | 40 mL |
| Hydrochloric acid, concentrated | 250 mL | 50 mL |
| Methanol | 250 mL | 50 mL |

Equipment:

Starch-iodide paper 10 strips

Part C. Aerobic Oxidation of Benzylic Alcohols

| Chemicals: | Quantity | |
|------------------------------|-----------|------------|
| | Miniscale | Microscale |
| 4-Nitrobenzyl alcohol | 10 g | 4.0 g |
| 3-Nitrobenzyl alcohol | 10 g | 4.0 g |
| 4-Chlorobenzyl alcohol | 10 g | 4.0 g |
| Cuprous bromide | 0.9 g | 0.4 g |
| 2,2-Bipyridyl | 1 g | 0.4 g |
| TEMPO | 1 g | 0.4 g |
| N-Methylimidazole | 5 mL | 2 mL |
| Acetone, reagent grade | 250 mL | 130 mL |
| Pentane | 300 mL | 200 mL |
| Magnesium sulfate, anhydrous | 25 g | 10 g |

Part D. Preparation of Derivatives

1. Preparation of Semicarbazones

| | Chemicals: Quantity |
|-----------------------------|---------------------|
| Semicarbazide hydrochloride | 5 g |
| Sodium acetate | 8 g |

To prepare the solution for making semicarbazones, dissolve 5 g of semicarbazide hydrochloride and 8 g of sodium acetate in 50 mL of distilled $\rm H_2O$.

2. Preparation of Oximes

| | Chemicals: Quantity |
|------------------------------|---------------------|
| Hydroxylamine hydrochloride | 5 g |
| Sodium hydroxide 3 M aqueous | 8 g |

Base-Catalyzed Oxidation-Reduction of Aldehydes by the Cannizzaro Reaction 16.3

| Chemicals: | | Quantity | |
|-------------------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Potassium hydroxide | 50 g | 10 g | |
| Methanol | 25 mL | 5 mL | |
| 4-Chlorobenzaldehyde | 10 g | 2 g | |
| Dichloromethane | 160 mL | 23 mL | |
| Sodium chloride, saturated solution | 100 mL | 10 mL | |
| Sodium sulfate, anhydrous | 50 g | 10 g | |
| Hydrochloric acid, concentrated | 25 mL | 5 mL | |
| Acetone | 5 mL | 1 mL | |
| Hexane | 50 mL | 10 mL | |
| Methanol | 100 mL | 10 mL | |

CH 17 Reduction Reactions of Double Bonds; Alkenes, Carbonyl Compounds, and Imines

17.2 Catalytic Hydrogenation of the Carbon-Carbon Double Bond

Part A. Hydrogenation of 4-Cyclohexene-cis-1,2-dicarboxylic Acid

| Chemicals: | Quantity |
|---|----------|
| Chloroplatinic acid, 5% solution | 5 mL |
| Decolorizing carbon | 2 g |
| Sodium borohydride, 1 M solution | 16 mL |
| Sodium hydroxide, 1% solution | 50 mL |
| 4-Cyclohexene-cis-1,2-dicarboxylic acid (see Sec. 12.3, | |
| Part D) | 5 g |
| Hydrochloric acid, concentrated | 20 mL |
| Diethyl ether, technical | 350 mL |
| Sodium chloride | 100 g |
| Sodium sulfate, anhydrous | 10 g |
| Qualitative Tests | |
| Chemicals: | Quantity |
| Bromine in dichloromethane solution (see Section 4.7A1) | |
| Baeyer test (see Section 4.7A2) | |
| Cyclohexene | 2 g |
| Equipment: | |
| Syringe, plastic 2-mL | 10 |
| Balloons | 10 |
| Wire | 50 cm |
| 30 | |

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Part B. Transfer Hydrogenation of Cinnamic Acid Derivatives

| Chemicals: | | Quantity | | |
|---|---|-------------------|--------|--|
| | | Miniscale Microso | | |
| | 4-Fluorocinnamic acid | 0.75 g | 0.38 g | |
| | 4-Chlorocinnamic acid | 0.75 g | 0.38 g | |
| | 4-Nitrocinnamic acid | 0.75 g | 0.38 g | |
| | Benzyl cinnamate | 0.75 g | 0.38 g | |
| | Ammonium formate | 7.5 g | 3.75 g | |
| | Pd/C, 10% | 0.7 g | 0.35 g | |
| | Hydrochloric acid, 1 M | 500 mL | 45 mL | |
| | Diethyl ether, technical | 300 mL | 150 mL | |
| | Methanol | 55 mL | 25 mL | |
| Ethyl acetate:hexane, 70:30 | | 50 mL | 50 mL | |
| Equipment: | | Qua | ntity | |
| | Thiele tube | 1 | 0 | |
| | or electric melting point apparatus | | 1 | |
| Packing tube 10 Capillary tube 10 | | 10 | | |
| | | 0 | | |
| | TLC chamber | 10 | | |
| TLC plates with fluorescence indicator, 250 | | ım precoated | | |
| as 1 in x 3 in strips | | 40 strip | S | |
| Equipment: | | Quantity | | |
| | Micropipets | 1 | 0 | |
| | Whatman GF/A filter discs 2.1 cm | | 3 | |
| 17.2 Form | notion and Daduction of N Cinnamulidans and | | | |

17.3 Formation and Reduction of N-Cinnamylidene-m-nitroaniline

| Chemicals: | nemicals: Quantity | |
|------------------------|--------------------|-----------------|
| | Miniscale | Microscale |
| Cinnamaldehyde | 6 g | 1.2 g |
| <i>m</i> -Nitroaniline | 6 g | 1.2 g |
| Cyclohexane | 100 mL | $20\ \text{mL}$ |
| Sodium borohydride | 1.5 g | 300 mg |
| Methanol | 70 mL | 14 mL |
| Ethanol, 95% | 100 mL | 30 mL |
| Equipment: | | |
| Syringe, 1-mL | | 10 |

3

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17.4 Reduction of 9-Fluorenone

| Chemicals: | Qua | Quantity | | |
|--------------------|-----------|------------|--|--|
| | Miniscale | Microscale | | |
| 9-Fluorenone | 6 g | 1 g | | |
| Methanol | 100 mL | 20 mL | | |
| Sodium borohydride | 0.5 g | 0.1 g | | |
| Sulfuric acid, 3 M | 20 mL | 3.5 mL | | |

17.5 Enantioselective Reductions: A Chiral Alcohol from a Ketone

Part A. Tartaric Acid-Mediated Enantioselective Reduction of Methyl Acetoacetate

| Chemicals: | Quantity |
|--|----------|
| Sodium borohydride | 5 g |
| D-(-)-Tartaric acid | 20 g |
| L-(+)-Tartaric acid | 20 g |
| Tetrahydrofuran | 300 mL |
| Methyl acetoacetate | 4 mL |
| Diethyl ether | 250 mL |
| Hydrochloric acid, 1 M aqueous | 100 mL |
| Sodium bicarbonate, saturated solution | 200 mL |
| Sodium chloride, saturated solution | 200 mL |
| Sodium sulfate, anhydrous | 10 g |

Part B. Enzymatic Reduction of Methyl Acetoacetate

| Chemicals: | Quantity |
|---------------------------------------|----------|
| Sucrose | 400 g |
| Disodium hydrogen phosphate | 2.5 g |
| Barium hydroxide, 3% aqueous solution | 300 mL |
| Baker's yeast | 80 g |
| Methyl acetoacetate | 25 mL |
| Filter aid | 200 g |
| Sodium chloride | 190 g |
| Dichloromethane | 1 L |

Equipment:

Anaerobic fermentation apparatus

Quantity

1

Qualitative Test

| Chemicals: | Quantity |
|---|----------|
| Cyclohexene | 2 g |
| Ferric chloride, 0.2 M aqueous solution | 2 mL |

17.6 Determining Optical Purity

| Chemicals: | Quantity |
|--|----------|
| rac-Methyl 3-hydroxybutanoate | 0.3 g |
| Deuterochloroform | 7.5 mL |
| tris-[3-(Heptafluoropropylhydroxymethylene)-(+)- | |
| camphorato]europium(III) | 1 g |
| Equipment: | |
| NMR tubes | 20 |

CH 18 Reactions of Carbonyl Compounds

Chemicals:

18.2 The Wittig and Related Reactions

Part A. Preparation of (Z)- and (E)-Stilbenes by a Wittig Reaction

| | ~ | - |
|--------------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Sodium Hydroxide | 50 g | 10 g |
| Benzyltriphenylphosphonium chloride | 38 g | 7.6 g |
| Dichloromethane | 160 mL | 25 mL |
| Benzaldehyde | 10 mL | 2 mL |
| Sodium bisulfite, saturated solution | 200 mL | 30 mL |
| Sodium chloride, saturated solution | 50 mL | 10 mL |
| Iodine | 750 mg | 75 mg |
| Ethanol, 95% | 250 mL | 50 mL |
| Sodium sulfate, anhydrous | 50 g | 10 g |

Qualitative Tests

| Chemicals: | Quantity |
|---|----------|
| Cyclohexene | 2 g |
| Duraning in dishlaramethous solution (see Costion 4.74.1) | |

Bromine in dichloromethane solution (see Section 4.7A1)

Baeyer test (see Section 4.7A2)

Equipment:

Light bulb and socket

| Part B. | Preparation | of a Stilbene | by the Horner | Wadsworth- | Emmons Reaction |
|---------|-------------|---------------|---------------|------------|------------------------|
|---------|-------------|---------------|---------------|------------|------------------------|

| | Chemicals: | Quantity | |
|------|--|-----------|-------------------|
| | | Miniscale | <i>Microscale</i> |
| | Potassium <i>tert</i> -butoxide, 1 <i>M</i> in DMF | 50 mL | 10 mL |
| | Diethyl benzylphosphonate | 10 mL | 2 mL |
| | Benzaldehyde | 5 mL | 1 mL |
| | Ethanol, 95% | 100 mL | 20 mL |
| | Qualitative Tests | | |
| | Chemicals: | Qua | intity |
| | Cyclohexene | | 2 g |
| | Bromine in dichloromethane solution (see Section 4 | .7A1) | |
| | Baeyer test (see Section 4.7A2) | | |
| | Equipment: | | |
| | Rubber septum | 10 | 10 |
| | Syringe, 1-mL | 20 | 20 |
| 18.3 | Preparation of trans-p-Anisalacetophenone | | |
| | <i>p</i> -Anisaldehyde | 10 mL | 2 mL |
| | Acetophenone | 10 mL | 2 mL |
| | Ethanol, 95% | 50 mL | 10 mL |
| | Sodium hydroxide | 10 g | 5 g |
| | Methanol | 50 mL | 10 mL |
| | Qualitative Tests | | |
| | Chemicals: | Qua | entity |
| | Cyclohexene | | 2 g |
| | Bromine in dichloromethane solution (see Section 4 | .7A1) | |
| | Baeyer test (see Section 4.7A2) | | |
| | Equipment: | | |
| | Syringe, 1-mL | 20 | 20 |
| 18.4 | Preparation of 4,4-Dimethyl-2-cyclohexen-1-one | | |
| | Chemicals: | Qua | entity |
| | | Miniscale | Microscale |
| | 2-Naphthalenesulfonic acid | 1 g | 0.1 g |
| | Toluene | 250 mL | 25 mL |

35 mL

50 mL

3.5 mL

 $5 \, mL$

3-Buten-2-one

2-Methylpropanal

³⁴

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18.4 Preparation of 4,4-Dimethyl-2-cyclohexen-1-one (cont.)

| Chemicals: | Quantity | |
|------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Sodium bicarbonate | 2 g | 1 g |
| Sodium sulfate, anhydrous | 30 g | 3 g |
| Diethyl ether, solvent grade | 30 mL | |
| Qualitative Tests | | |
| Chemicals: | Oua | ntitv |

| Chemicuis. | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| 2,4-Dinitrophenylhydrazine test (see Section 7.4B) | | |
| Ethanol, 95 % | 30 mL | 10 mL |
| Equipment: | | |
| Syringe, 1-mL | | 20 |
| Syringe, 5-mL | 20 | |
| Microburner | 10 | 10 |

18.5 Synthesis of Ethyl 6-Methyl-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Chemicals: Quantity

| | Miniscale | Microscale |
|------------------------|-----------|------------|
| Benzaldehyde | 13.3 g | 2.7 g |
| Ethyl acetoacetate | 16.3 g | 3.3 g |
| Urea | 9.5 g | 1.9 g |
| p-Toluenesulfonic acid | 1.0 g | 0.2 g |
| Ethanol, anhydrous | 150 mL | 15 mL |
| Ethyl acetate | 10 mL | 10 mL |
| Hexanes | 10mL | 10 mL |

CH 19 Organometallic Chemistry

19.2 Preparation of Grignard Reagents

Phenylmagnesium Bromide

Chemicals: Quantity Miniscale Microscale Magnesium turnings 5 g 0.5 gDiethyl ether, anhydrous 150 mL 50 mL Bromobenzene 39 g 3.9 g

n-Butylmagnesium Bromide

| Chemicals: | Quantity | |
|---------------------------|-----------|------------|
| | Miniscale | Microscale |
| 1-Bromobutane | 34 g | 3.4 g |
| Magnesium turnings | 5 g | 0.5 g |
| Diethyl ether, anhydrous | 150 mL | 50 mL |
| Equipment: | | |
| Syringe, 5-mL | | 10 |
| Syringe, 1-mL | | 20 |
| Screw-cap centrifuge tube | | 10 |

19.4 Grignard Reagents: Reactions

Part A. Preparation of Triphenylmethanol

| Chemicals: | Qua | Quantity | |
|--|-----------|------------|--|
| | Miniscale | Microscale | |
| Methyl benzoate | 12 mL | 1.2 mL | |
| Diethyl ether, anhydrous | 100 mL | 20 mL | |
| Diethyl ether, solvent grade | 200 mL | 40 mL | |
| Sulfuric acid, 6 M | 150 mL | | |
| Chemicals: | Quantity | | |
| | Miniscale | Microscale | |
| Sulfuric acid, 3 M | 150 mL | 20 mL | |
| Sodium bicarbonate, saturated solution | 100 mL | 10 mL | |
| Sodium chloride, saturated solution | 50 mL | 5 mL | |
| Sodium sulfate, anhydrous | 50 g | 5 g | |
| Cyclohexane | 1.5 L | 0.15 L | |
| Equipment: | | | |
| Rubber septum | | 20 | |
| Syringe, 1-mL | | 10 | |
| Screw-cap centrifuge tube | | 20 | |
| Part R Propagation of Rangoia Acid | | | |

Part B. Preparation of Benzoic Acid

| Chemicals: | Quantity | |
|--------------------------|-----------|------------|
| | Miniscale | Microscale |
| Dry ice | 100 g | 10 g |
| Diethyl ether, anhydrous | 50 mL | 10 mL |

Part B. Preparation of Benzoic Acid (cont.)

| Chemicals: | Quantity | |
|------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Diethyl ether, solvent grade | 400 mL | 40 mL |
| Sulfuric acid, 3 M | 100 mL | 15 mL |
| Sodium hydroxide, 1 M | 200 mL | 20 mL |
| Hydrochloric acid, 6 M | 100 mL | 10 mL |
| Equipment: | | |
| Screw-cap centrifuge tube | | 20 |

Part C. Preparation of 2-Methyl-3-heptanol

| Chemicals: | Quantity | |
|------------------------------|-----------|------------|
| | Miniscale | Microscale |
| 2-Methylpropanal | 18 mL | |
| Diethyl ether, anhydrous | 50 mL | |
| Sulfuric acid, 6 M | 100 mL | |
| Diethyl ether, solvent grade | 150 mL | |
| Sodium bisulfite | 20 g | |
| Sodium chloride | 108 g | |
| Sodium bicarbonate, 1.2 M | 100 mL | |
| Sodium sulfate, anhydrous | 15 g | |

19.5 Preparation of 3-Ethylhex-5-en-3-ol

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| Zinc | 15.2 g | 3.0 g |
| Allyl Bromide | 12 mL | 2.4 mL |
| Iodine | 1.25 g | 0.25 g |
| 3-Pentanone | 12 mL | 2.5 mL |
| Tetrahydrofuran anhydrous | 130 mL | 20 mL |
| Diethyl ether | 50 mL | 5 mL |
| Hydrochloric acid, 1 M aqueous | 30 mL | 10 mL |
| Sodium chloride, saturated solution | 50 mL | 20 mL |
| Sodium bicarbonate, saturated solution | 50 mL | 20 mL |
| Sodium thiosulfate, saturated solution | 50 mL | 10 mL |
| Sodium sulfate, anhydrous | 5.0 g | 0.5 g |

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19.6 Preparation of 4'-Methyl-(1,1'-biphenyl)-4-methanol

| Chemicals: | Quantity | |
|---|-----------|-------------|
| | Miniscale | Microsccale |
| 4-Methylphenylboronic acid | 7.0 g | 1.4 g |
| 4-Bromobenzyl alcohol | 10.0 g | 2.0 g |
| Palladium, 1000 ppm aqueous solution | 10 mL | 2 mL |
| Potassium hydroxide, 1 M ethanolic solution | 100 mL | 20 mL |
| Ethanol, 95% | 120 mL | 60 mL |
| Dichloromethane | 250 mL | 100 mL |
| Magnesium sulfate, anhydrous | 10 g | 2 g |

CH 20 Carboxylic Acids and Their Derivatives

20.2 Esters and the Fischer Esterification

Part A. Preparation of Benzocaine

| Chemicals: | Quantity | |
|--|-----------|------------|
| | Miniscale | Microscale |
| p-Aminobenzoic acid | 10 g | 2 g |
| Ethanol, absolute | 130 mL | 25 mL |
| Sulfuric acid, concentrated | 15 mL | 3 mL |
| Sodium carbonate, 10% aqueous solution | 300 mL | 60 mL |
| Methanol | 100 mL | 20 mL |

Part B. Identifying Unknown Esters Produced by Fischer Esterification

| Chemicals: | Quantity |
|--|------------|
| | Microscale |
| Methanol | 30 mL |
| Ethanol (reagent grade) | 30 mL |
| 1-Propanol | 30 mL |
| 1-Butanol | 30 mL |
| Benzoic acid | 14.6 g |
| Propanoic acid | 8.8 g |
| Sulfuric acid-silica-gel | 1 g |
| Sodium carbonate, 10% aqueous solution | 30 mL |
| Sodium chloride, saturated solution | 30 mL |
| Sulfuric acid, concentrated | 3 mL |
| Diethyl ether, technical | 30 mL |
| Sodium sulfate, anhydrous | 5.0 g |
| | |

Part B. Identifying Unknown Esters Produced by Fischer Esterification (cont.) Instructor Notes

Stock solutions of the unknowns are prepared by adding 122 g of benzoic acid or 60 g of propanoic acid to 1000 mL of methanol; ethanol; 1-propanol and 1-butanol. (Stock solutions are 1 M in the carboxylic acid.) The stock solutions have a good shelf life and showed no discoloration upon storage at room temperature for up to three months.

Preparation of sulfuric acid on silica gel: Add 25 mL of *concentrated* sulfuric acid dropwise to 20 g of 60–80-mesh silica gel and dry the resulting slurry under vacuum for 24 h. Dry the resulting off-white paste further at 130 °C for 24 h.

Isothermal GC Methods:

General GC: PE Model Clarus 580 Gas Chromatograph; Restek Column (15 or 30 M) Rtx-1 Crossbonded 100% Dimethyl polysiloxane (0.32 ID; 0.25 df). Standards were run for each day's analysis and retention times are reported in min.

For propanoate esters:

Isothermal GC: Inj. Temp. 75 °C; Det. Temp: 150 °C; Air Flow: 450 mL/min; H₂ flow: 45 mL/min; Column Length: 15 M; Column 130 °C isothermal; Column Flow: 5 mL/min

TABLE 1: Isothermal Retention Times (in min) for Propanoate Esters

| Ester | RT _(min) |
|---------------------|---------------------|
| Methyl propanoate | 1.23 |
| Ethyl propanoate | 1.69 |
| 1-Propyl propanoate | 2.43 |
| 1-Butyl propanoate | 3.36 |

For benzoate esters:

Isothermal GC: Inj. Temp.: 220 °C; Det. Temp.: 220 °C; Air Flow: 450 mL/min; H₂ flow: 45 mL/min; Column Length: 30 M; Column 210 °C isothermal; Column Flow: 1 mL/min

TABLE 2: Isothermal Retention Times (in min) for Benzoate Esters

| Ester | RT _(min) |
|---------------------------|---------------------|
| Methyl benzoate | 3.65 |
| Ethyl benzoate | 3.80 |
| <i>n</i> -Propyl benzoate | 4.08 |
| n-Butyl benzoate | 4.54 |

Part B. Identifying Unknown Esters Produced by Fischer Esterification (cont.) Programmable GC Methods:

General GC: PE Model Clarus 580 Gas Chromatograph; Restek Column (15 or 30 M) Rtx-1 Crossbonded 100% Dimethyl polysiloxane (0.32 ID; 0.25 df). Standards were run for the analysis each day, and retention times are reported in min.

Three programmable GC methods may be used to separate the two families of esters. Method A is used for the rapid analysis of propanoic esters. Method B for the rapid analysis of alkyl benzoates, and Method C may be used to separate all eight compounds in a single run.

TABLE 3: RETENTION TIMES (in min) for Propanoate Esters

| Ester | METHOD A | METHOD C |
|----------------------------|----------|----------|
| Methyl propanoate | 6.55 | 3.01 |
| Ethyl propanoate | 9.96 | 4.64 |
| n-Propyl propanoate | 14.68 | 7.52 |
| <i>n</i> -Butvl propanoate | 18.88 | 9.29 |

TABLE 4: RETENTION TIMES (in min) for Benzoate Esters

| Ester | METHOD B | METHOD C |
|---------------------------|----------|----------|
| Methyl benzoate | 1.24 | 11.82 |
| Ethyl benzoate | 1.71 | 12.47 |
| <i>n</i> -Propyl benzoate | 2.24 | 13.28 |
| <i>n</i> -Butyl benzoate | 3.38 | 13.70 |

Method A: Inj. Temp.: 75 °C; Det. Temp: 150 °C; Air Flow: 450 mL/min.; H₂ flow: 45 mL/min; Column Length: 30 M; Column Program: Iso: 65 °C 8 Min.; 5 °C /Min to 150 °C; Isothermal 150 °C; 0.5 Min.; Column Flow: 2 mL/min.

Method B: Inj. Temp: 150 °C; Det. Temp: 175 °C; Air Flow: 450 mL/min; H₂ flow: 45 mL/min; Column Length: 15 M; Column Program: 85 °C iso 0.5 min. 10 °C /min to 250 °C; Iso 250 °C, 2 min; Column Flow: 5 mL/min.

Method C: Inj. Temp.: 75 °C; Det. Temp.: 150 °C; Air Flow: 450 mL/min; H₂ flow: 45 mL/min; Column Length: 30 M; Column Program: 70 °C Isothermal 6.0 min 20 °C/min to 250 °C Isothermal 250 °C 5 min; Column Flow: 5 mL/min.

20.3 Preparation of N,N-Diethyl-m-toluamide

| Chemicals: | Quantity | |
|--------------------------------------|-----------|------------|
| | Miniscale | Microscale |
| 3-Methylbenzoic acid (m-toluic acid) | 20 g | 2 g |
| Thionyl chloride | 22 mL | 2 mL |
| Diethylamine | 50 mL | 5 mL |
| Diethyl ether, anhydrous | 400 mL | 30 mL |
| Sodium hydroxide, 2.5 M | 150 mL | 15 mL |
| Hydrochloric acid, 3 M | 150 mL | 15 mL |
| Sodium sulfate, anhydrous | 20 g | 3 g |
| Alumina | 200 g | 20 g |
| Heptane | 550 mL | 55 mL |
| Equipment: | | |
| Gas traps | 10 | 10 |
| Syringe, 2-mL | | 10 |
| Screw cap centrifuge tube | | 10 |
| Chromatography columns | 10 | 10 |

20.4 Preparation and Chemiluminescence of Luminol

Part A. Preparation of Luminol

| Chemicals: | Quantity | |
|-------------------------------|-----------|------------|
| | Miniscale | Microscale |
| 3-Nitrophthalic Acid | 10 g | 2 g |
| Hydrazine, 8% | 20 mL | 4 mL |
| Triethylene glycol | 30 mL | 6 mL |
| Sodium hydroxide, 3 M | 50 mL | 10 mL |
| Sodium hydrosulfite dihydrate | 30 g | 6 g |
| Acetic acid, glacial | 20 mL | 4 mL |

Part B. Chemiluminescence

| Chemicals: | Qua | Quantity | |
|----------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Sodium hydroxide, 3 M | 20 mL | 20 mL | |
| Potassium ferricyanide, 3% | 40 mL | 40 mL | |
| Hydrogen peroxide, 3% | 40 mL | 40 mL | |

CH 21 Multistep Organic Synthesis

21.2 Sulfanilamide: Discovery and Synthesis of the First Antibiotic

Part A. Preparation of Aniline

| Chemicals: | Quantity |
|--|----------|
| Nitrobenzene | 52 mL |
| Tin powder | 131 g |
| Hydrochloric acid, concentrated | 325 mL |
| Sodium hydroxide, 12 M | 500 mL |
| Sodium chloride | 300 g |
| Diethyl ether, solvent grade | 600 mL |
| Sodium sulfate, anhydrous | 100 g |
| Equipment: | |
| Steam distillation apparatus | 10 |
| Part B. Preparation of Acetanilide | |
| Chemicals: | Quantity |
| Aniline | 36 mL |
| Hydrochloric acid, 0.4 N (33 mL of concentrated HCl/L | |
| of solution) | 1 L |
| Carbon, decolorizing | 10 g |
| Sodium acetate, trihydrate | 60 g |
| Acetic anhydride | 44 mL |
| Part C. Preparation of 4-Acetamidobenzenesulfonyl Chloride | |
| Chemicals: | Quantity |
| Acetanilide (Not required if prepared in Part B.) | 27 g |
| Chlorosulfonic acid | 80 mL |
| Dichloromethane | 100 mL |
| Equipment: | |
| Gas trap | 10 |
| Part D. Preparation of 4-Acetamidobenzenesulfonamide | |
| Chemicals: | Quantity |
| Ammonium hydroxide, concentrated | 150 mL |
| Part E. Preparation of Sulfaniliamide | |
| Chemicals: | Quantity |
| Hydrochloric acid, 6 M | 300 mL |
| Sodium carbonate | 10 g |

100 mL

25 mL

50 g

10 mL

10 mL

10 g

Solubility Tests

Chemicals: Quantity

Hydrochloric acid, 1.5 M (0.4 mL of concentrated HCl/3

mL of solution) 3 mL

Sodium hydroxide, 1.5 M (1.2 g of NaOH/20 mL of solution) 20 mL

21.3 Synthesis of 1-Bromo-3-chloro-5-iodobenzene

Part B. Preparation of 4-Bromoacetanilide

| Chemicals: | Qua | Quantity | |
|----------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Bromine | 32 mL | 1.5 mL | |
| Acetic acid, glacial | 60 mL | 33 mL | |
| Acetanilide | 81 g | 3.75 g | |
| Methanol | 100 mL | 10 mL | |
| Sodium bisulfite | 50 g | 10 g | |

Part C. Preparation of 4-Bromo-2-chloroacetanilide

| Chemicals: | Quantity | |
|---------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Hydrochloric acid, concentrated | 230 mL | 10 mL |
| Acetic acid, glacial | 280 mL | 13 mL |
| Sodium Chlorate | 28 g | 1.5 g |

Part D. Preparation of 4-Bromo-2-chloroaniline

Methanol

Methanol

Sodium bisulfite

| Chemicals: | Quantity | |
|---------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Hydrochloric acid, concentrated | 130 mL | 5 mL |
| Ethanol, 95% | 200 mL | 20 mL |
| Sodium hydroxide, 14 N | 120 mL | 50 mL |

Part E. Preparation of 4-Bromo-2-chloro-6-iodoaniline

| hemicals: Q | | Quantity | |
|--------------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Acetic acid, glacial | 750 mL | 75 mL | |
| Iodine monochloride, technical | 25 g | 2.5 g | |
| Acetic acid, 33% | 50 mL | 10 mL | |

Ouantity

Part F. Preparation of 4-Bromo-2-chloro-6-iodobenzene

| Chemicals: | | Qua | Quantity | |
|------------|------------------------------|-----------|------------|--|
| | | Miniscale | Microscale | |
| | Sulfuric acid, concentrated | 40 mL | 4 mL | |
| | Ethanol, absolute | 100 mL | 15 mL | |
| | Sodium nitrite | 7 g | 0.7 g | |
| | Dichloromethane | 300 mL | 40 mL | |
| | Methanol | 200 mL | 30 mL | |
| Equipment: | | | | |
| | Steam distillation apparatus | | 10 | |

21.4 Lidocaine: Synthesis of an Anesthetic Agent

Part A. Preparation of 2,6-Dimethylaniline

| Chemicals: | Qua | Quantity | |
|---------------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| 2,6-Dimethylnitrobenzene | 50 g | 5 g | |
| Stannous chloride dihydrate | 340 g | 34 g | |
| Hydrochloric acid, concentrated | 400 mL | 40 mL | |
| Acetic acid, glacial | 500 mL | 50 mL | |
| Diethyl ether, solvent grade | 300 mL | 30 mL | |
| Potassium hydroxide, 8 M | 500 mL | 50 mL | |
| Sodium sulfate, anhydrous | 25 g | 3 g | |

Part B. Preparation of α -Chloro-2,6-dimethylacetanilide

| | Σ | , |
|---------------------------|-----------|------------|
| | Miniscale | Microscale |
| -Chloroacetyl chloride | 28 g | 2.8 g |
| Acetic acid, glacial | 200 mL | 20 mL |
| Sodium acetate trihydrate | 43 g | 5 g |

Part C. Preparation of Lidocaine

Chemicals:

| Chemicals: | Qua | Quantity | |
|------------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| Diethylamine | 24 g | 2.2 g | |
| Toluene | 350 mL | 30 mL | |
| Hydrochloric acid, 3 M | 400 mL | 30 mL | |
| Potassium hydroxide, 8 M | 250 mL | 20 mL | |
| Diethyl ether, solvent grade | 300 mL | 30 mL | |

Part C. Preparation of Lidocaine (cont.)

| Chemicals: | Quantity | |
|---------------------------------|-----------|------------|
| | Miniscale | Microscale |
| Sodium sulfate, anhydrous | 25 g | 3 g |
| Sulfuric acid, 2.2 M in ethanol | 50 mL | 5 mL |
| Acetone | 250 mL | 25 mL |

CH 22 Polymers

22.2 Chain-Reaction Polymerization

Part A. Removal of the Inhibitor from Commercial Styrene

| Chemicals: | Quantity |
|---|----------|
| Styrene, commercial | 100 mL |
| Sodium hydroxide, 3 M (4.8 g of NaOH/40 mL of solution) | 40 mL |
| Calcium chloride | 8 g |

Part B. Polymerization of Pure Styrene

| Chemicals: | Quantity |
|---------------------------------|----------|
| Styrene, anhydrous (see Part A) | 30 mL |
| tert-Butyl peroxybenzoate | 2 mL |
| | |

Equipment:

Microburners 10

Part C. Solution Polymerization of Styrene

| Chemicals: | Quantity |
|---------------------------------------|----------|
| Styrene, anhydrous (see Part A) | 30 mL |
| Xylene, commercial mixture of isomers | 60 mL |
| tert-Butyl peroxybenzoate | 1 mL |
| Methanol | 250 mL |

22.3 Preparation of Nylon-6,10

| Chemicals: | Quantity |
|--|----------|
| Decanedioyl dichloride (sebacoyl chloride) | 20 mL |
| Dichloromethane | 1 L |
| 1,6-Hexanediamine (hexamethylenediamine) crystals or | 12 g |
| 80–95% aqueous solution | 13 mL |
| Sodium carbonate | 20 g |
| Ethanol, 50% | 2 L |
| Formic acid, 90–100% | 500 mL |

22.3 Preparation of Nylon-6,10 (cont.)

Equipment:

| Measuring pipet, 5-mL or syringe (3–5 mL) | 5 |
|--|--------|
| either Drum, made from a coffee, juice, or motor oil can | 5 |
| forceps | 5 |
| or Copper wire | 100 cm |

CH 23 Carbohydrates

23.3 Hydrolysis of Sucrose

| Chemicals: | Quantity |
|---------------------------------|----------|
| Sucrose | 75 g |
| Hydrochloric acid, concentrated | 5 mL |

23.4 Classification Tests for Carbohydrates

Tollens's Test

| Chemicals: | Quantity |
|---------------------|----------|
| Silver nitrate | 2.5 g |
| Water, distilled | 85 mL |
| Potassium hydroxide | 3 g |

To prepare the reagent, two stock solutions must be combined at the time the test is being performed. Prepare solution A is by dissolving 2.5 g of silver nitrate in 43 mL of distilled H₂O. Prepare solution B by dissolving 3 g of KOH in 42 mL of distilled H₂O.

Benedict's Test

| Chemicals: | Quantity |
|-----------------------------|----------|
| Sodium citrate, dihydrate | 26 g |
| Sodium carbonate, anhydrous | 15 g |
| Cupric sulfate | 2.6 g |
| Barfoed's Test | |

| Chemicals: | Quantity |
|----------------------|----------|
| Cupric acetate | 6 g |
| Acetic acid, glacial | 0.9 mL |

Formation of Osazones

| Chemicals: | Quantity |
|--------------------------------|-------------|
| D-Glucose, D-fructose, sucrose | 2 g of each |
| Sodium bisulfite | 10 g |
| Ethanol, 95% | 150 mL |

Quantity

Formation of Osazones (cont.)

| Chemic | eals: | Quantity |
|--------|-------------------------------|----------|
| either | Acetic acid, glacial 6 mL | |
| | Sodium acetate | 6 g |
| | Phenylhydrazine | 4 g |
| or | Sodium acetate | 6 g |
| | Phenylhydrazine hydrochloride | 6 g |

CH 24 α-Amino Acids and Peptides

Chemicals:

24.3 Synthesis of the Protected Dipeptide Ala-Phe-OMe

Part A. Preparation of N-tert-Butoxycarbonyl-L-Alanine

| Chemicals: | Qua | Quantity | |
|-------------------------------------|-----------|------------|--|
| | Miniscale | Microscale | |
| L-Alanine | 9.0 g | 2.0g | |
| Di-tert-butyl dicarbonate | 25 mL | 5 mL | |
| tert-Butyl alcohol | 50 mL | 10 mL | |
| Sodium hydroxide, 3 M | 50 mL | 10 mL | |
| Diethyl ether, technical | 500 mL | 100 mL | |
| Hydrochloric acid, 3 M | 75 mL | 15 mL | |
| Sodium chloride, saturated solution | 100 mL | 20 mL | |
| Sodium sulfate, anhydrous | 25 g | 5 g | |
| Hexanes | 500 mL | 100 mL | |
| Ethyl acetate | 50 mL | 10 mL | |

Part B. Preparation of Methyl L-Phenylalaninate Hydrochloride

| | ~ | • |
|--------------------------|-----------|------------|
| | Miniscale | Microscale |
| L-Phenylalanine | 10 g | 2 g |
| Methanol | 100 mL | 25 mL |
| Thionyl chloride | 5 mL | 1 mL |
| Diethyl ether, technical | 500 mL | 100 mL |

Part C. Preparation of Methyl N-tert-Butoxycarbonyl L-Alanyl-L-phenylalaninate

| Chemicals: | Quantity |
|------------|----------|
| | |

| | Miniscale | Microscale |
|----------------------------|-----------|------------|
| Dimethylformamide | 200 mL | 20 mL |
| <i>N</i> -Methylmorpholine | 6 mL | 1.5 mL |
| Isobutyl chloroformate | 4 mL | 1 mL |

500 mL

100 mL

Ethyl acetate

Part C. Preparation of Methyl N-tert-Butoxycarbonyl L-Alanyl-L-phenylalaninate (cont.)

| Chemicals: | Quantity | | | | |
|--|-----------|------------|--|--|--|
| | Miniscale | Microscale | | | |
| Diethyl ether, technical | 750 mL | 200 mL | | | |
| Hydrochloric acid, 1 M | 500 mL | 80 mL | | | |
| Sodium bicarbonate, saturated solution | 250 mL | 50 mL | | | |
| Sodium chloride, saturated solution | 250 mL | 50 mL | | | |
| Sodium sulfate, anhydrous | 25 g | 5 g | | | |
| Hexanes | 150 mL | 40 mL | | | |

Part D. Preparation of Methyl L-Alanyl-L-phenylalaninate Trifluoroacetate

| Chemicals: | Qua | ntity |
|--------------------------|-----------|------------|
| | Miniscale | Microscale |
| Trifluoroacetic acid | 15 mL | 2 mL |
| Dichloromethane | 60 mL | 10 mL |
| Diethyl ether, technical | 40 mL | 10 mL |

CH 25 Identifying Organic Compounds

Chemicals: This is a partial list of chemicals and solutions, with common acids, bases, and organic solvents not being included. In cases where directions are provided for preparing solutions, the amounts are to serve approximately 10 students unless otherwise noted.

Acetic anhydride

Aniline

Baeyer reagent

To prepare $0.1 M \ aqueous \ KMnO_4$, dissolve $0.32 \ g$ of potassium permanganate in $20 \ mL$ of distilled H_2O . This amount of solution should suffice for the needs of aboujt $100 \ students$.

Benzenesulfonyl chloride

Benzoyl chloride

Bromine in dichloromethane

To prepare a 0.1 M solution, dissolve 0.1 mL of Br_2 in 20 mL of CH_2Cl_2 ; keep the solution in a tightly stoppered container. This amount of solution should suffice for the needs of about 100 students.

Bromine-potassium bromide reagent

To prepare the reagent, dissolve 2 g of KBr in 12 mL of distilled water and adding 0.6 mL of Br₂.

Bromine water

To prepare the saturated solution, dissolve 11.8 mL of Br_2 in 10 mL of H_2O .

Ceric ammonium nitrate

To prepare the reagent, dissolve 2 g of ceric ammonium nitrate in 5 mL of 2 M nitric acid; the dissolution is hastened by heating.

Chromic anhydride

To prepare chromic acid, add 10 g of chromic anhydride to 10 mL of *concentrated* H_2SO_4 and stir the mixture until a smooth paste is obtained. The *cautiously* dilute the paste with 30 mL of distilled H_2O and stir this mixture until a clear orange solution is obtained.

Diethylene glycol

- 3,5-Dinitrobenzoic acid
- 3,5-Dinitrobenzoyl chloride
- 2,4-Dinitrophenylhydrazine

To prepare the solution for qualitative tests and for making 2,4-dinitrophenylhydrazones, dissolve 2 g of 2,4-dinitrophenylhydrazine in 10 mL of *concentrated* H_2SO_4 ; add this solution, with stirring, to a solution of 15 mL of distilled H_2O and 50 mL of 95% ethanol. Vigorously stir this solution and then filter it to remove any undissolved solids.

Ferric chloride

To prepare a 0.2 *M aqueous* FeCl₃ solution, dissolve 5.4 g of ferric chloride hexahydrate in 100 mL of distilled water. This amount of solution should suffice for the needs of about 100 students.

To prepare a 0.6 *M aqueous* FeCl₃ solution, dissolve 1.6 g of ferric chloride hexahydrate in 10 mL of distilled water.

To prepare a 0.5 *M methanolic* FeCl₃ solution, dissolve 1.3 g of ferric chloride hexahydrate in 10 mL of methanol.

Ferrous ammonium sulfate

To prepare a 5% solution, add 2.5 g of crystalline ferrous ammonium sulfate and 0.2 mL of *concentrated* sulfuric acid to 50 mL of recently boiled distilled water. Add a small iron nail to the solution to retard air-oxidation.

Hydrion E paper

Hydroxylamine hydrochloride

To prepare the solution for making oximes, dissolve 5 g of hydroxylamine hydrochloride in a solution of 50 mL of distilled H_2O and 30 mL of 3 M aqueous NaOH.

Iodine

To prepare the solution for the iodoform test, dissolve 10 g of iodine in a solution of 20 g of KI in 80 mL of distilled H_2O .

Lead acetate solution, 0.15 M

Lucas reagent

To prepare the reagent, dissolve 14.9 g of anhydrous zinc chloride in 10 mL of concentrated HCl.

Methyl iodide (iodomethane)

 α -Naphthol

 α -Naphthyl isocyanate

Nitric acid, fuming

Phenyl isocyanate

Picric acid

Potassium bromide (see Bromine-potassium bromide reagent)

Potassium fluoride, 5 M

Potassium iodide

Potassium permanganate (also see Baeyer reagent)

Propylene glycol

Pyridine

Ramini test (see Sodium nitroprusside)

p-Rosaniline hydrochloride

To prepare a solution for the Schiff's test, dissolve 0.1 g of p-rosaniline hydrochloride in 100 mL of distilled H_2O and then add 4 mL of saturated aqueous sodium bisulfite. Allow this solution to stand for

1h and then add 2 mL of *concentrated* HCl with stirring to complete preparation of the reagent.

Semicarbazide hydrochloride

To prepare the solution for making semicarbazones, dissolve 5 g of semicarbazide hydrochloride and 8 g of sodium acetate in 50 mL of distilled H₂O.

Silver nitrate, ethanolic solution

To prepare a 0.1 M ethanolic solution, dissolve 0.34 g of silver nitrate in 20 mL of 95% ethanol.

Silver nitrate (also see Tollens' reagent)

Simon test (see Sodium nitroprusside)

Sodium acetate

Sodium dichromate dihydrate

Sodium iodide in acetone solution

To prepare the test solution, dissolve 3 g of sodium iodide in 20 mL of acetone. Keep the solution in a dark bottle and discard it when a red-brown color appears.

Sodium-lead alloy

Sodium metal

Sodium nitroprusside

To prepare the reagent, dissolve 0.4 g of sodium nitroprusside dihydrate in 10 mL of 50% aqueous methanol.

Sulfuric acid, fuming

Thionyl chloride

Tin, granulated

Tollens's reagent

To prepare the reagent, two stock solutions must be combined at the time the test is being performed. Prepare solution A is by dissolving 2.5 g of silver nitrate in 43 mL of distilled H_2O . Prepare solution B by dissolving 3 g of KOH in 42 mL of distilled H_2O . Directions for combining these two solutions when the student is ready to do the test are provided in Section 25.7.

p-Toluidine

Zinc chloride, anhydrous (see Lucas reagent)

| | Acetamide | | | | | | | | | | | | |
|---|---|--------------------|--|---------------------------------|----------------------------------|-----------------------------|---|-----------------------------------|--|-------|-------------------|--|--|
| | | | | | C ₂ H ₅ NO | 1 | | | | | | | |
| CAS No. | PS | Color | Odor | FP | BP | MP | d | | VP | VD | Sol | | |
| 60-35-5 | Solid | Colorless | Distinct | 174 | 221 | 80–82 | 1.159 | 10 | 0 @ 105 | 2 | | | |
| Type Hazard/E | | Acut | te Hazards/ | Sympto | oms | Preve | ention | | First Aid/Fire | | | | |
| Fire | Slight fire hazard. Avoid heat, sparks, and flames. Dry cher carbon d regular f | | | | oxide, w | | | | | | | | |
| Inhalation | | and skin er | drowsiness, ruptions. Irrembranes and tract. | ritating | to | Ventilation, local exhaust. | | | Remove from exposure immediately and seek medical advice. | | | | |
| Skin | | Irritation a skin. | nd may be a | Protective gloves and clothing. | | | Remove of clothes/je thoroughl soap and medical a | ewelry, ly wash s water, ar | skin with | | | | |
| Eyes | yes Irritation, corneal damage. Safety goggles. Thoroughly with water fremoving copossible, and advice. | | | | er for sev contact | eral min, lenses if | | | | | | | |
| Ingestion Drowsiness, fatigue and skin eruptions. | | | | ausea, a | acidosis | | Do <i>not</i> eat or drink in the laboratory. | | Wash out mouth w water; if vomiting keep head lower the Seek medical advi immediately. | | occurs, han hips. | | |
| | | Τ | | | | | | | | | | | |
| Carcinogenicity Suspected carcinogen. | | | | | Mutag | genicity | | Not a kno | own muta | ıgen. | | | |

| | | | 4-Acetan | | N2O3S | | | | | | | | | |
|--|---------|---|---|----------|---------|---|---|--|------------------|--------------------------------------|----------|--|--|--|
| CAS No. | PS | Color | Odor | FP | BP | MP | d | 1 | VP | VD | Sol | | | |
| 121-61-9 | Solid | Off-white | N/A | N/A | N/A | 219 | N/A | N | N/A N/A Slightly | | | | | |
| Types of Hazard/Exposure Acute Hazards/Symptoms Prevention First | | | | | | First A | id/Fire | | | | | | | |
| Fire | | Flammable. Emits toxic fumes under fire conditions. No open flames or sparks. Water spray, card dioxide, dry cher powder or appropriam. | | | | | hemical | | | | | | | |
| Inhalation | | May be harm | May be harmful if inhaled. | | | | | Ventilation, local exhaust. Remove from exposu immediately and seek medical advice. | | | | | | |
| Skin | | May be irrita | May be irritating to the skin. | | | | Protective gloves and clothing. Remove contaminate clothes/jewelry, thoroughly wash sk soap and water, and medical advice. | | | ry, ash skin with er, and seek | | | | |
| Eyes | | Dust, vapor, eyes. | ting to the | Safety g | oggles. | | Thoroughly flush eyes with water for several removing contact lense possible, and seek med advice. | | | | | | | |
| Ingestion | | May be harm | Do <i>not</i> eat or drink in the laboratory. | | | Wash out mouth with water; if vomiting occurs, keep head lower than hips Seek medical advice immediately. | | | | | | | | |
| Carcinoge | enicity | Not a known | carcinogen | | | Muta | genicity | 7 | Not a | a known i | mutagen. | | | |

Abbreviations:

| 4-Acetamidobenzenesulfonyl Chloride C8H8ClNO3S | | | | | | | | | | | |
|--|---------|----------|---|-------------------------------|---------------------|---|--|-----|---------|----------|--|
| CAS No. | PS | Color | Odor | FP | BP | MP | d | VP | VD | Sol | |
| 121-60-8 | Solid | Tan | Acetic acid-like | N/A | N/A | 145–148 | N/A | N/A | 8.1 | Slightly | |
| Types of Hazards/Symptoms Prevention First Aid/Fire | | | | | | | | | | | |
| Fire Moderate fire hazard. No flames, no sparks, no contact with hot surfaces. Carbon dioxide, dry chemical powder, or | | | | | | | | | | | |
| Inhalation Corrosive material, chemical burns, coughing, wheezing, laryngitis, headache, and vomiting. | | | | | | | Ventilation, local exhaust. Remove from experimediately and somedical advice. | | | | |
| Skin | | Corrosiv | ve material, chemic | Protective clothing. | clot thor soa | Remove contaminated clothes/jewelry, thoroughly wash skin wi soap and water, and seek medical advice. | | | | | |
| Eyes Corrosive material, chemical burns. Safety goggles. Thoroughly flu with water for s removing conta possible, and so advice. | | | | | | r several min, tact lenses if | | | | | |
| Ingestion | | | ve material, convulses, and symptoms a gestion. | Do <i>not</i> eat the laborat | wat kee See | Wash out mouth with water; if vomiting occurs, keep head lower than hips Seek medical advice immediately. | | | | | |
| Carcinog | enicity | Not a kn | nown carcinogen. | | | Mutag | genicity | Not | a known | mutagen. | |

Abbreviations: CAS No. = Chemical Abstracts Service Registry Number; PS = physical state; FP = flash point (°C); BP = boiling point (°C) @ 760 torr unless otherwise stated; MP = melting point (°C); d = density or specific gravity (g/mL); VP = vapor pressure (torr) at specified temperature (°C); VD = vapor density relative to air (1.0); Sol = solubility in water (g/100 mL) at 25 °C unless otherwise

stated; N/A = not available or not applicable.

| Acetanilide C8H9NO | | | | | | | | | | | | | |
|--|-------|-------------|-------------------------------|--------|-----------|--|-----------|----------------------------|---|-----------------------------|--|--|--|
| CAS No. | PS | Color | Odor | FP | BP | MP | d | VP | VD | Sol | | | |
| 103-84-4 | Solid | White | Odorless | 169 | 304 | 114 | 1.219 | 1 @ 114 | @ 114 4.7 | | | | |
| Type Hazard/E | | Acu | te Hazards/S | Sympto | oms | Prev | vention | | First Aid/Fire | | | | |
| Fire Slight fire hazard. Avoid heat, sparks and flames. Dry chemical carbon dioxide regular foam. | | | | | | dioxide, | | | | | | | |
| | | | | | | Ventilation exhaust. | n, local | immed | Remove from exposure immediately and seek medical advice. | | | | |
| Skin | | | contact derm due to systen | | | Protective clothing. | gloves an | clothe thorou soap a | Remove contaminated clothes/jewelry, thoroughly wash skin wi soap and water, and seek medical advice. | | | | |
| Eyes | | Irritation. | Irritation. | | | Safety goggles. Thoroughly flush ey with water for sever removing contact let possible, and seek madvice. | | | | everal min, ct lenses if | | | |
| Ingestion Nausea, vomiting, sweating, gastric irritation, chills. Do not eat or of the laboratory | | | | | | water; keep h Seek r | | ng occurs, r than hips. | | | | | |
| Carcinog | | Muta | genicity | Possib | le mutage | en. | | | | | | | |

Abbreviations:

| | Acetic Acid (glacial) | | | | | | | | | | | | |
|---|-----------------------|-------------|---|---|--------------------------------|---|--|----------|---|--|------|--|--|
| | | | | | $C_2H_4O_2$ | | | | | | | | |
| CAS No. | PS | Color | Odor | FP | BP | MP | d | | VP | Sol | | | |
| 64-19-7 | Liquid | Colorless | Vinegary | 39 | 118 | N/A | 1.049 | 11.3 | 8 @ 20 | Soluble | | | |
| Type Hazard/E | | Acut | te Hazards/S | Pro | evention | | First Aid/Fire | | | | | | |
| Fire | | | fire hazard. \early | | | No flames, no sparks, no contact with hot surfaces. | | | carbon regular | Dry chemical powder, carbon dioxide, water, regular foam, alcohol- resistant foam | | | |
| Inhalation Irritation, pharyngeal bronchitis, coughing, breath, laryngitis, pul and hypotension. | | | | hortne | ss of | Ventilation, local exhaust. | | | Remove from exposure immediately and seek medical advice. | | | | |
| Skin | | superficial | pain, blisters destruction sorbed through | Protective gloves and clothing. | | | Remove contaminated clothes/jewelry, thoroughly wash skin w water and 5% aqueous sodium bicarbonate, and seek medical advice. | | | | | | |
| Eyes | | | | | several min, tact lenses if | | | | | | | | |
| Ingestion | | of the esop | eronecrotic l bhagus, diarr abdominal s | Do <i>not</i> eat or drink in the laboratory. | | | Seek medical advice. Give large amounts of water and allow vomiting to occur; when vomiting occurs, keep head lower than hips. | | | | | | |
| Carcinog | genicity | Not a know | wn carcinoge | en. | | Mut | tagenicity | y | Possib | le muta | gen. | | |

Abbreviations:

| Acetic Anhydride C4H6O3 | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---------------------------------|--|---|--------------------|-------------------------------|-------------------------------|
| | | | | | | | | _ | | | | |
| CAS No. | PS | Color | Odor | FP | BI | | MP | d | | VP | VD | Sol |
| 108-24-7 | Liquid | Colorless | Vinegary | 54 | 138– | 140 -73 1.0820 10 @ 36 3.52 Rea | | | | | Reacts | |
| | Types of Hazard/Exposure Acute Hazards/Symptoms Prevention First | | | | First A | id/Fire | | | | | | |
| Fire | | gases may sources, co | fire hazard. ignite at disontact with when the merate flammers. | No flames, no sparks, no contact with hot surfaces. | | | | Carbon chemic resistar | al pow | der, alcohol- | | |
| Inhalation Severe irritation, cough, choking, wheezing, chest pain, and pulmonary edema, which may be fatal. Ventilation, local exhaust. | | | | | local | | | iately a | exposure and seek e. | | | |
| Skin | | Irritation, white, wrinkled skin, blisters, and severe burns. | | | | | Protective gloves and clothing. | | | clothes thoroug | /jewelr ghly wa nd wate | ash skin with er, and seek |
| Eyes Pain, lacrimation, photophobia, and blurred vision. Corneal and conjunctival edema, iritis, corneal erosion, and opacity may be delayed effects. Safety goggles. | | | | es. | | with w removi | ater for ng con e, and | ush eyes several min, tact lenses if seek medical | | | | |
| Ingestion | Severe burns of the mouth, esophagus, and stomach with pain, difficulty swallowing, nausea, vomiting, and diarrhea. | | | ain, | Do <i>not</i> eat or drink in the laboratory. | | | | Seek medical advice. Giv large amounts of water or milk, and allow vomiting to occur; when vomiting occurs keep head lower than hips. | | | |
| Carcinog | Carcinogenicity Not a known carcinogen. | | | | | | Mutago | enicity | | Not a k | nown | mutagen. |

Abbreviations:

| | Acetone | | | | | | | | | | | | | |
|---|--|------------------------------|---|-------------------------------|----|-----|---|--------------------------|------------------------|-----------|-----|--|--|--|
| | | | | C ₃ H ₆ | 0 | | | | | | | | | |
| CAS No. | PS | Color | Odor | FP | BP | MP | d | | VP | VP VD Sol | | | | |
| 67-64-1 | Liquid | Colorless | Paint thinner- like | -17 | 56 | -94 | 0.791 | 1 | 80 @ 20 2.0 Solubl | | | | | |
| Types of Hazard/Exposure Acute Hazards/Symptoms Prevention First Aid/Fire | | | | | | | | | | | | | | |
| Fire | | | fire hazard; vapors or gases may t distant ignition sources. No flames, no sparks, no contact with hot sources. Alcohol-resistant foam, carbon dioxide, dry chemical powder, water. | | | | | | | dry | | | | |
| | | | | | | | e from exposure ately and seek I advice. | | | | | | | |
| Skin | cellular damage to the helium with edema a unts may be absorbe | and hyperemia, and clothing. | | | | es | Remove contaminated clothes/jewelry, thoroughly wash skin wi soap and water, and seek medical advice. | | | | | | | |
| Eyes | | | Irritation, corneal epithelial, conjunctival, stinging sensation, and damage to eyes. | | | | Safety goggles. Thoroughly flush eyes with water for several removing contact lens possible, and seek med advice. | | | | | | | |
| Fruity odor of the breath and mucous membrane, gastroenteric irritation, diarrhea, nausea and vomiting. Do not eat or drink in the laboratory. Wash out mou water; if vomit keep head low Seek medical a immediately. | | | | | | | vomitii d lowei lical ac | ng occurs, than hips. | | | | | | |
| Carcinog | enicity | Not a know | wn carcinogen. | | | Mut | agenicity | 7 | Possible | mutage | en. | | | |

Abbreviations:

| p-Acetophenetidide [Phenacetin] C ₁₀ H ₁₃ NO ₂ | | | | | | | | | | | | |
|---|--|----------|-------------------------------------|--------|------------|---|--------------|-----|---|--|--|--|
| CAS No. | PS | Color | Odor | FP | BP | MP | d | VP | VD | Sol | | |
| 62-44-2 | Solid | White | Odorless | N/A | Decomposes | 134–135 | N/A | N/A | N/A N/A 0.0763 | | | |
| Typ Hazard/ | nrds/Sy | mptoms | Preve | ention | | First Aid/Fire | | | | | | |
| Fire | | Slightly | / flammabl | e. | | | | | | ray, dry chemical alcohol foam, or ioxide. | | |
| Inhalation | Cyanosis, dizziness, respiratory depression. Local exhaust or breathing protection | | | | | 1 | | | | | | |
| Skin | n Possibly a mild irritant. Protective gloves and clothing. | | | | | | | | | | | |
| Eyes | | Possibl | Possibly irritating to eye tissues. | | | Safety goggles, or eye protection in combination with Thoroughly flus with water for so removing contact. | | | | aly flush eyes or for several min, contact lenses if and seek medical | | |
| Ingestion | Moderately toxic, causes cyanosis, dizziness, respiratory depression. Cardiac arrest may occur. May result in liver and kidney damage. | | | | | | | | ely and keep er than hips. lical advice | | | |
| Carcinogenicity Possible carcinogen. Mutagenicity Not a known mutagen. | | | | | | | own mutagen. | | | | | |

Abbreviations:

| Acetophenone C ₈ H ₈ O | | | | | | | | | | | | |
|---|---|-------------------------|--|--------------|---|---|--|---|--------|------------|--|--|
| CAS No. | PS | Color | Odor | FP | BP | MP | d | VP | VD | Sol | | |
| 98-86-2 | Liquid | Colorless | Floral | 82 | 203 | 19–20 | 1.030 | 1 @ 1: | 4.14 | 0.55 @ 20 | | |
| | Types of Acute Hazards/Symptoms Prevention First Aid/Fire Iazard/Exposure | | | | | | | Aid/Fire | | | | |
| Fire Moderate fire hazard. No flames, no sparks, no contact with hot surfaces. Dry chemical powd carbon dioxide, wat regular foam | | | | | | ride, water, | | | | | | |
| Inhalation | | nervous sy | coughing and stem depres dizziness, an | sion wi | ith | Ventilation exhaust. | imı | Remove from exposure immediately and seek medical advice. | | | | |
| Skin | | Irritation, r burns. | edness, pair | Protective g | clo tho soa | Remove contaminated clothes/jewelry, thoroughly wash skin wi soap and water, and seek medical advice. | | | | | | |
| epithelium. removing conta | | | | | flush eyes for several min, ontact lenses if ad seek medical | | | | | | | |
| Ingestion Sore throat, abdominal pain, nausea and central nervous system depression with headache, dizziness, and narcos | | | | epression | Do not eat the laborate | war kee See | Wash out mouth with water; if vomiting occur keep head lower than his Seek medical advice immediately. | | | | | |
| Carcinogenicity Not a known carcinogen. Mutagenicity | | | | | | | | No | a know | n mutagen. | | |

Abbreviations:

Full Download: http://downloadlink.org/product/solutions-manual-for-experimental-organic-chemistry-a-miniscale-and-microscale

| Acetylsalicylic Acid [Aspirin] C ₉ H ₈ O ₄ | | | | | | | | | | | |
|---|----------|-----------------------------------|--------------|-----|------------|---|---------|----------------------|--|--------|---------|
| CAS No. PS | | Color Odor | | FP | BP | MP | d | | VP VD | | Sol |
| 50-78-2 | Solid | White | Odorless | 250 | N/A | 134–136 | 1.340 | 3 x 10 ⁻⁶ | ⁶ @ 25 N/A 3.3 g/L | | |
| Types of Hazard/Exposure | | Acute Hazards/Symptoms | | | Prevention | | | First Aid/Fire | | | |
| Fire | | N/A | | | | N/A | | | Dry chemical powder, carbon dioxide, water, alcohol-resistant foam. | | |
| Inhalation | | May cause respiratory irritation. | | | | Ventilation, local exhaust. | | | Remove from exposure immediately and seek medical advice. | | |
| Skin | | Causes skin irritation. | | | | Protective gloves and clothing. | | | Remove contaminated clothes/jewelry. Thoroughly wash skin with soap and water, and seek medical advice | | |
| Eyes | | Causes serious eye irritation. | | | | Safety goggles. | | | Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice | | |
| Ingestion | | Harmful if swallowed. | | | | Do <i>not</i> eat or drink in the laboratory. | | | If swallowed wash out mouth with water. Seek medial advice. | | |
| Carcino | genicity | Not a kno | wn carcinoge | en. | | Mu | tagenic | ity | Not a kı | nown m | utagen. |

For more detailed information consult the Material Safety Data Sheet for this compound.

Abbreviations: