Solutions for Experimental Organic Chemistry A Miniscale and Microscale Approach 6th Edition by Gilbert

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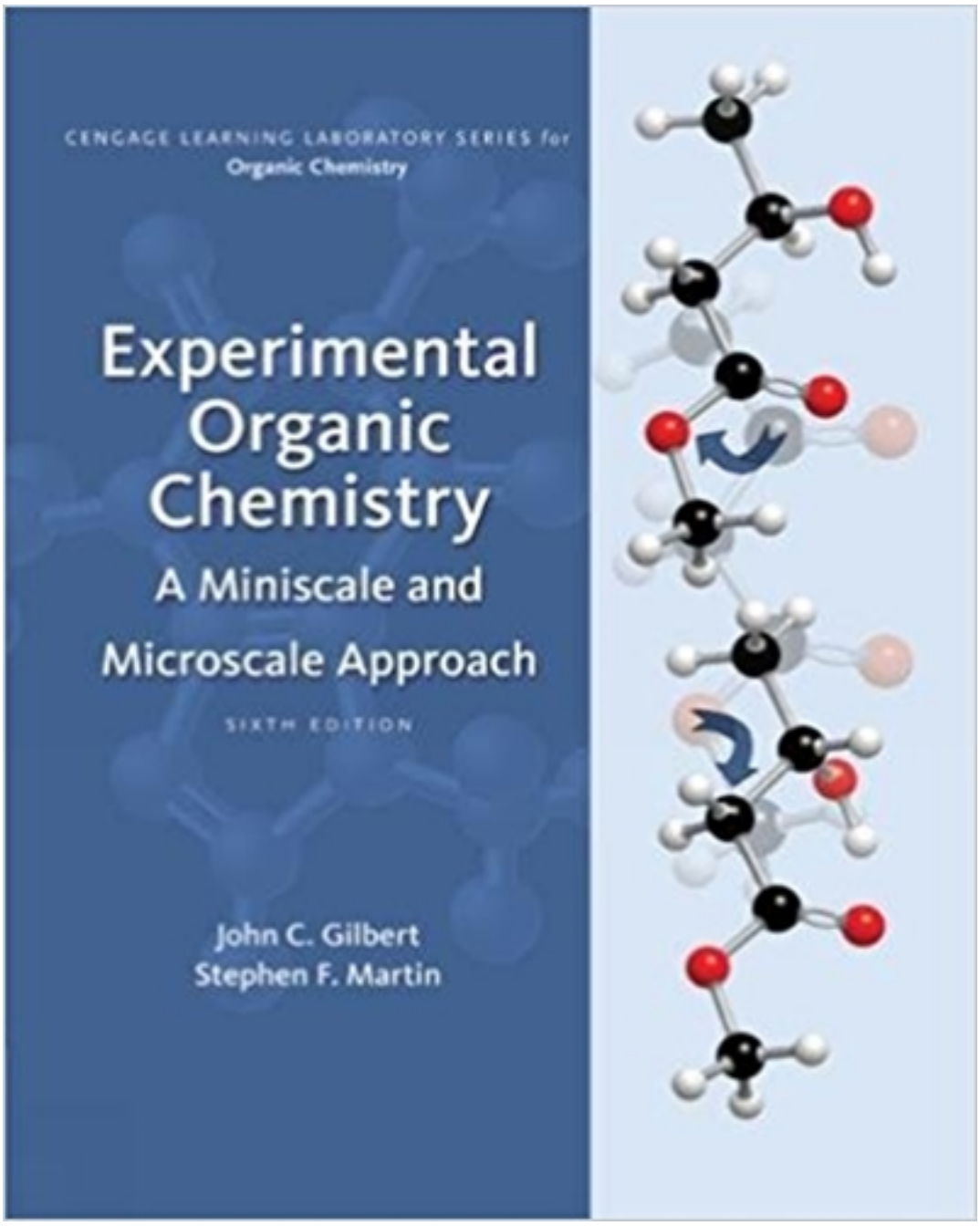

Solutions

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.)

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
p-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 µm pre-coated silica gel TLC plates with fluorescence indicator cut into ~ 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 (*dec.*)
Aspirin	134–136
p-Phenyphenol	164–166
4-Hydroxyacetanilide	168–172
p-Toluic acid	177–180
p-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
n-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 Br_2 in CH_2Cl_2, dissolve 0.01 mL of Br_2 in 10 mL of CH_2Cl_2; store the solution in a tightly stoppered container.

2. Potassium Permanganate

Chemicals:	*Quantity*
Water, *distilled*	2 mL
Potassium permanganate	0.032 g
Ethanol, *95%*	40 mL

Dissolve 0.32 g of $KMnO_4$ 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

Chemicals:	*Quantity*	
	Miniscale	*Microscale*
Ground nutmeg	40 g	10 g
Diethyl ether, *solvent grade*	150 mL	50 mL
Acetone	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	

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

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	*ca*. 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

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		1

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 Br_2 in CH_2Cl_2, dissolve 0.01 mL of Br_2 in 10 mL of CH_2Cl_2; 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	1

CH 10 Alkenes

10.2 Dehydrohalogenation of Alkyl Halides

Part A. Elimination with Alcoholic Potassium Hydroxide

Chemicals:	*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*	
	Miniscale	*Microscale*
4-Methyl-2-pentanol	40 mL	
Sulfuric acid, 9 *M* (50:50 *concentrated* H_2SO_4:H_2O)	25 mL	
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 $AgNO_3$ in ethanol, dissolve 0.4 g of $AgNO_3$ 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

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

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, *95%*	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_2O	200 mL	40 mL

10.7 Hydration of Norbornene

Chemicals:	*Quantity*
Sulfuric acid, *concentrated*	20 mL
Norbornene	10 g
Potassium hydroxide	15 g
Diethyl ether, *solvent grade*	250 mL
Sodium bicarbonate	5 g
Sodium chloride	20 g
Sodium sulfate	20 g

10.8 Hydroboration-Oxidation of Alkenes

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

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

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

Chemicals:	*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:	*Quantity*
Phenyl isocyanate or α-naphthyl isocyanate	5 mL
Pyridine	1 mL
Petroleum ether (60–80 °C)	50 mL

CH 11 Alkynes

11.2 Dehydrobromination of *meso*-Stilbene Dibromide

Chemicals:	*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, *95%*	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

Chemicals:	*Quantity*
Sulfuric acid, *concentrated*	30 mL
Mercuric oxide	2 g
2-methyl-3-butyn-2-ol	36 mL
Potassium carbonate	30 g
Sodium chloride	100 g
Dichloromethane	200 mL
Semicarbazide hydrochloride	5 g
Sodium acetate	8 g
2-Propanol	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:	*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:	*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

CH 13 Kinetic and Thermodynamic Control of a Reaction

Part A. Preparation of Cyclohexanone Semicarbazone

Chemicals:	*Quantity*
Semicarbazide hydrochloride	5 g
Dibasic potassium phosphate	10 g

Part A. Preparation of Cyclohexanone Semicarbazone (cont.)

Cyclohexanone	5 mL
Ethanol, *95%*	25 mL

Part B. Preparation of 2-Furaldehyde Semicarbazone

Chemicals:	*Quantity*
Semicarbazide hydrochloride	5 g
Dibasic potassium phosphate	10 g
Ethanol, *95%*	25 mL
2-Furaldehyde	4 mL

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, *95%*	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)
1-Bromohexane	16.3 g
2-Bromohexane	16.3 g
Sodium methoxide, 1.5 *M* in methanol	140 mL
Potassium *tert*-butoxide, 1.5 *M* in *tert*-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

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
1-Bromopropane	85 mL	8 mL

Equipment:	
Gas trap	10
Syringe, 1-mL	10

15.3 Friedel-Crafts Acylation of Anisole

Chemicals:

	Quantity	
	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	
	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:

	Quantity
25- or 50-mL Buret	10

Part B. Thin-Layer Chromatography

Chemicals:

	Quantity
Dichloromethane	10 mL
Hexane	36 mL
Iodine	10 g
Ethyl acetate	4 mL

Equipment:

	Quantity
Eastman Type K3012R Chromagram sheet or equivalent	1 sheet

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

Chemicals:	*Quantity*
Anisole	3 g
Diphenyl ether	3 g
Acetanilide	3 g
Acetic acid, *glacial*	300 mL
Bromine	3 g
Equipment	
Colorimeter	5
Cuvette	15

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

Chemicals:	*Quantity*
	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

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

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:	
Starch-iodide paper	10 strips

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 H_2O.

2. Preparation of Oximes

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

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

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

Part B. Transfer Hydrogenation of Cinnamic Acid Derivatives

Chemicals:	*Quantity*	
	Miniscale	*Microscale*
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:	*Quantity*
Thiele tube	10
or electric melting point apparatus	1
Packing tube	10
Capillary tube	10
TLC chamber	10
TLC plates with fluorescence indicator, 250-µm precoated as 1 in x 3 in strips	40 strips

Equipment:	*Quantity*
Micropipets	10
Whatman GF/A filter discs 2.1 cm	3

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

Chemicals:	*Quantity*	
	Miniscale	*Microscale*
Cinnamaldehyde	6 g	1.2 g
m-Nitroaniline	6 g	1.2 g
Cyclohexane	100 mL	20 mL
Sodium borohydride	1.5 g	300 mg
Methanol	70 mL	14 mL
Ethanol, *95%*	100 mL	30 mL

Equipment:	
Syringe, 1-mL	10

17.4 Reduction of 9-Fluorenone

Chemicals:	*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

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

18.2 The Wittig and Related Reactions

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

Chemicals:	*Quantity*	
	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
Bromine in dichloromethane solution (see Section 4.7A1)	
Baeyer test (see Section 4.7A2)	
Equipment:	
Light bulb and socket	1

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

Chemicals:	*Quantity*	
	Miniscale	*Microscale*
Potassium *tert*-butoxide, 1 *M* 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:	*Quantity*
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

p-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:	*Quantity*
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:	*Quantity*	
	Miniscale	*Microscale*
2-Naphthalenesulfonic acid	1 g	0.1 g
Toluene	250 mL	25 mL
3-Buten-2-one	35 mL	3.5 mL
2-Methylpropanal	50 mL	5 mL

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:

	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 g
Diethyl 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:	*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 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

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; H_2 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; H_2 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
n-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
n-Butyl 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
n-Propyl benzoate	2.24	13.28
n-Butyl benzoate	3.38	13.70

Method A: Inj. Temp.: 75 °C; Det. Temp: 150 °C; Air Flow: 450 mL/min.; H_2 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; H_2 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; H_2 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:	*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

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:	*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
Methanol	100 mL	10 mL

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

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
Methanol	25 mL	10 mL

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

Chemicals:	*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
Sodium bisulfite	50 g	10 g

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

Chemicals:	*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:	*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

Chemicals:	*Quantity*	
	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:	*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 H_2O. Prepare solution *B* by dissolving 3 g of KOH in 42 mL of distilled H_2O.

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

Formation of Osazones (cont.)

Chemicals:		*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

24.3 Synthesis of the Protected Dipeptide Ala–Phe-OMe

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

Chemicals:	*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

Chemicals:	*Quantity*	
	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
N-Methylmorpholine	6 mL	1.5 mL
Isobutyl chloroformate	4 mL	1 mL

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:	*Quantity*	
	Miniscale	*Microscale*
Trifluoroacetic acid	15 mL	2 mL
Dichloromethane	60 mL	10 mL
Diethyl ether, *technical*	40 mL	10 mL
Ethyl acetate	500 mL	100 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 Br_2.

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* $FeCl_3$ 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* $FeCl_3$ solution, dissolve 1.6 g of ferric chloride hexahydrate in 10 mL of distilled water.

To prepare a 0.5 *M methanolic* $FeCl_3$ 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 H_2O.

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_2H_5NO

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 @ 105	N/A	2

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Slight fire hazard.	Avoid heat, sparks, and flames.	Dry chemical powder, carbon dioxide, water, regular foam.
Inhalation	Irritation, drowsiness, nausea, acidosis and skin eruptions. Irritating to mucous membranes and upper respiratory tract.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation and may be absorbed through skin.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Irritation, corneal damage.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Drowsiness, fatigue, nausea, acidosis and skin eruptions.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Suspected carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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.

4-Acetamidobenzenesulfonamide

$C_8H_{10}N_2O_3S$

CAS No.	PS	Color	Odor	FP	BP	MP	d	VP	VD	Sol
121-61-9	Solid	Off-white	N/A	N/A	N/A	219	N/A	N/A	N/A	Slightly

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Flammable. Emits toxic fumes under fire conditions.	*No* open flames or sparks.	Water spray, carbon dioxide, dry chemical powder or appropriate foam.
Inhalation	May be harmful if inhaled.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	May be irritating to the skin.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Dust, vapor, or mist may be irritating to the eyes.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	May be harmful if swallowed.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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 (g/mL); Sol = solubility in water (g/100mL) at 25 °C unless otherwise state; N/A = not available or not applicable.

4-Acetamidobenzenesulfonyl Chloride

$C_8H_8ClNO_3S$

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 Hazard/Exposure	Acute 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 foam.
Inhalation	Corrosive material, chemical burns, coughing, wheezing, laryngitis, headache, and vomiting.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Corrosive material, chemical burns.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Corrosive material, chemical burns.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Corrosive material, convulsions, muscle weakness, and symptoms as described in acute ingestion.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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

C_8H_9NO

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	4.7	0.5

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Slight fire hazard.	Avoid heat, sparks and flames.	Dry chemical powder, carbon dioxide, water, regular foam.
Inhalation	Irritation, eczematous skin eruptions, poisoning as in ingestion.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation, contact dermatitis, skin eruptions due to systemic poisoning.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Irritation.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Nausea, vomiting, sweating, gastric irritation, chills.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Possible mutagen.

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

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.

Acetic Acid (*glacial*)

$C_2H_4O_2$

CAS No.	PS	Color	Odor	FP	BP	MP	d	VP	VD	Sol
64-19-7	Liquid	Colorless	Vinegary	39	118	N/A	1.049	11.8 @ 20	2.07	Soluble

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Moderate fire hazard. Vapors or gases may ignite at distant ignition sources.	*No* flames, *no* sparks, *no* contact with hot surfaces.	Dry chemical powder, carbon dioxide, water, regular foam, alcohol-resistant foam
Inhalation	Irritation, pharyngeal edema, chronic bronchitis, coughing, shortness of breath, laryngitis, pulmonary edema, and hypotension.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation, pain, blisters, burns and superficial destruction of the skin; readily absorbed through the skin.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with water and 5% aqueous sodium bicarbonate, and seek medical advice.
Eyes	Irritation, lacrimation, corneal erosion, opacification, iritis, and possibly loss of sight.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Severe ulceronecrotic lesions, stricture of the esophagus, diarrhea, shock, vomiting, abdominal spasms.	Do *not* 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.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Possible mutagen.

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

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.

Acetic Anhydride

$C_4H_6O_3$

CAS No.	PS	Color	Odor	FP	BP	MP	d	VP	VD	Sol
108-24-7	Liquid	Colorless	Vinegary	54	138–140	-73	1.0820	10 @ 36	3.52	Reacts

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Moderate fire hazard. Vapors or gases may ignite at distant ignition sources, contact with water or moist air may generate flammable and/or toxic gases.	*No* flames, *no* sparks, *no* contact with hot surfaces.	Carbon dioxide, dry chemical powder, alcohol-resistant foam
Inhalation	Severe irritation, cough, choking, wheezing, chest pain, and pulmonary edema, which may be fatal.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation, white, wrinkled skin, blisters, and severe burns.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Pain, lacrimation, photophobia, and blurred vision. Corneal and conjunctival edema, iritis, corneal erosion, and opacity may be delayed effects.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Severe burns of the mouth, esophagus, and stomach with pain, difficulty swallowing, nausea, vomiting, and diarrhea.	Do *not* eat or drink in the laboratory.	Seek medical advice. Give large amounts of water or milk, and allow vomiting to occur; when vomiting occurs keep head lower than hips.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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.

Acetone

C_3H_6O

CAS No.	PS	Color	Odor	FP	BP	MP	d	VP	VD	Sol
67-64-1	Liquid	Colorless	Paint thinner- like	-17	56	-94	0.791	180 @ 20	2.0	Soluble

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	*Severe* fire hazard; vapors or gases may ignite at distant ignition sources.	*No* flames, *no* sparks, *no* contact with hot sources.	Alcohol-resistant foam, carbon dioxide, dry chemical powder, water.
Inhalation	Irritation, dryness of the mouth and throat, central nervous system depression, headache.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation, cellular damage to the outer layers of the epithelium with edema and hyperemia, small amounts may be absorbed through intact skin.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with 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 min, removing contact lenses if possible, and seek medical advice.
Ingestion	Fruity odor of the breath and mucous membrane, gastroenteric irritation, diarrhea, nausea and vomiting.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Possible mutagen.

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

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.

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	0.0763

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Slightly flammable.	*No* flames, *no* sparks, *no* contact with hot sources.	Water spray, dry chemical powder, alcohol foam, or carbon dioxide.
Inhalation	Cyanosis, dizziness, respiratory depression.	Local exhaust or breathing protection.	Remove from exposure immediately and seek medical advice.
Skin	Possibly a mild irritant.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Possibly irritating to eye tissues.	Safety goggles, or eye protection in combination with breathing protection.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Moderately toxic, causes cyanosis, dizziness, respiratory depression. Cardiac arrest may occur. May result in liver and kidney damage.	Do *not* eat or drink in the laboratory.	Induce vomiting immediately and keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Possible carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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.

Acetophenone

C_8H_8O

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 @ 15	4.14	0.55 @ 20

Types of Hazard/Exposure	Acute Hazards/Symptoms	Prevention	First Aid/Fire
Fire	Moderate fire hazard.	*No* flames, *no* sparks, *no* contact with hot surfaces.	Dry chemical powder, carbon dioxide, water, regular foam
Inhalation	Irritation, coughing and central nervous system depression with headache, dizziness, and narcosis.	Ventilation, local exhaust.	Remove from exposure immediately and seek medical advice.
Skin	Irritation, redness, pain, and mild burns.	Protective gloves and clothing.	Remove contaminated clothes/jewelry, thoroughly wash skin with soap and water, and seek medical advice.
Eyes	Severe reaction with only transient optical irregularity of the corneal epithelium.	Safety goggles.	Thoroughly flush eyes with water for several min, removing contact lenses if possible, and seek medical advice.
Ingestion	Sore throat, abdominal pain, nausea and central nervous system depression with headache, dizziness, and narcosis.	Do *not* eat or drink in the laboratory.	Wash out mouth with water; if vomiting occurs, keep head lower than hips. Seek medical advice immediately.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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.

Acetylsalicylic Acid [Aspirin]

$C_9H_8O_4$

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	3×10^{-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 *not* eat or drink in the laboratory.	If swallowed wash out mouth with water. Seek medial advice.
Carcinogenicity	Not a known carcinogen.	**Mutagenicity**	Not a known mutagen.

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

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; N/A = not available or not applicable.