

***Experimental Organic Chemistry A Miniscale and
Microscale Approach 6th edition Gilbert Solutions
Manual***

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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:

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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):

	<i>Quantity</i>
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:

	<i>Quantity</i>
Thiele tubes	10
or electric melting point apparatus	1
Packing tubes	10

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

Equipment:

Capillary tubes

Quantity

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
<i>trans</i> -Cinnamic acid	133
<i>p</i> -Acetophenetidide (phenacetin)	135
<i>o</i> -Chlorobenzoic acid	142
Salicylamide	142
Benzilic acid	150
Adipic acid	153
Salicylic acid	158
Benzanilide	163
<i>p</i> -Bromoacetanilide	167
<i>p</i> -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.

	<i>Quantity/3 students</i>
Acetanilide	0.6 g
Ethyl <i>p</i> -Hydroxybenzoate	0.6 g
Urea	0.6 g
(<i>E</i>)-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.)

	<i>Quantity/3 students</i>
4-Hydroxyacetanilide	0.6 g
<i>p</i> -Toluic Acid	0.6 g
<i>p</i> -Anisic Acid	0.6 g
<i>Equipment:</i>	<i>Quantity</i>
Thiele tubes	10
or electric melting point apparatus	1
Packing tubes	10
Melting-point capillaries	50
TLC chambers	10
250 μ m 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 ($^{\circ}$ C)
Acetanilide	113–115
Ethyl <i>p</i> -hydroxybenzoate	114–117
Urea	132–135
<i>trans</i> -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.

<i>Equipment:</i>	<i>Quantity</i>
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 (<i>tert</i> -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
<i>tert</i> -Butylbenzene	168
<i>sec</i> -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*

	<i>Miniscale</i>	<i>Microscale</i>
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Simple distillation:

Cyclohexane with non-volatile dye	100 mL	20 mL
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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, <i>solvent grade</i>	300 mL
Calcium chloride, <i>anhydrous</i> , granular	5–10 g
Chemicals for unsaturation tests (see 4.7A1)	

Equipment:

Apparatus for steam distillation using an internal steam source	10
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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:**Quantity*

Water, <i>distilled</i>	2 mL
Potassium permanganate	0.032 g
Ethanol, 95%	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**

<i>Chemicals:</i>	<i>Quantity</i>
Chromic anhydride	10 g
Sulfuric acid, <i>concentrated</i>	10 mL
Water, <i>distilled</i>	30 mL

To prepare chromic acid, add 1 g of chromic anhydride to 1 mL of *concentrated* H₂SO₄ and stir the mixture until a smooth paste is obtained. Then *cautiously* dilute the paste with 3 mL of distilled H₂O and stir this mixture until a clear orange solution is obtained.

CH 5 Extraction**5.3 Base and Acid Extractions**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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, <i>solvent grade</i>	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, <i>anhydrous</i>	10 g	5 g

5.4 Isolation of Trimyristin from Nutmeg

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Ground nutmeg	40 g	10 g
Diethyl ether, <i>solvent grade</i>	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**

<i>Chemicals:</i>	<i>Quantity</i>
Green leaves	10
Petroleum ether (30–60 °C)	150 mL
Ethanol, <i>absolute</i>	30 mL
Sodium sulfate, <i>anhydrous</i>	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

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
Ethyl acetate	10 mL
Ethanol, <i>absolute</i>	10 mL
<i>n</i> -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 μ L capacity
 Syringe, gas-tight

CH 7 Stereoisomers

7.2 Separation of Diastereomeric 1,2-Cyclohexanediols

<i>Chemicals:</i>	<i>Quantity</i>
1,2-Cyclohexanediol, commercial mixture of <i>cis</i> - and <i>trans</i> -isomers	<i>ca.</i> 1 g
<i>trans</i> -1,2-Cyclohexanediol, 98%	<i>ca.</i> 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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Dimethyl maleate	15 mL	5 mL
Bromine in dichloromethane, 0.6 <i>M</i>	20 mL	
Bromine in dichloromethane, 0.1 <i>M</i>		10 mL

7.3 Isomerization of Dimethyl Maleate to Dimethyl Fumarate (cont.)*Chemicals:**Quantity*

	<i>Miniscale</i>	<i>Microscale</i>
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
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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 <i>Chem Sources U.S. A.</i> 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, <i>concentrated</i>	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.)

<i>Chemicals:</i>	<i>Quantity</i>
Sodium hydroxide, 14 <i>M</i>	80 mL
Ether, <i>solvent grade</i>	1.5 L
Sodium chloride	55 g
Sodium sulfate, <i>anhydrous</i>	30 g
Ethanol, <i>absolute</i>	300 mL
<i>Equipment:</i>	
Polarimeter	

CH 9 Alkanes**9.2 Free-Radical Chain Chlorination of 1-Chlorobutane**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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 <i>M</i> (100 g of Na ₂ SO ₄ /4 L of solution)	100 g	10 g
Sodium sulfate, <i>anhydrous</i>	50 g	5 g
Sodium chloride solution (brine)	300 mL	20 mL
<i>Equipment:</i>		
Glass wool, Pyrex		
Gas trap	10	10

9.3 Relative Rates of Free-Radical Chain Bromination

<i>Chemicals:</i>	<i>Quantity</i>
Toluene	5 mL
Ethylbenzene	5 mL
Isopropylbenzene	5 mL
<i>tert</i> -Butylbenzene	5 mL
Cyclohexane	5 mL
Methylcyclohexane	5 mL
Dichloromethane	360 mL
Bromine in dichloromethane, 1 <i>M</i>	70 mL
<i>Equipment:</i>	
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**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Potassium hydroxide in 1-propanol, 4 <i>M</i>	250 mL	25 mL
2-Bromo-2-methylbutane	25 mL	10 mL

Part B. Elimination with Potassium *tert*-Butoxide

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Potassium <i>tert</i> -butoxide in <i>anhydrous tert</i> -butyl alcohol, 1 <i>N</i>	250 mL	
2-Bromo-2-methylbutane	25 mL	

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
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**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
4-Methyl-2-pentanol	40 mL	
Sulfuric acid, 9 <i>M</i> (50:50 concentrated H ₂ SO ₄ :H ₂ O)	25 mL	
Potassium carbonate, <i>anhydrous</i>	20 g	

Part B. Dehydration of Cyclohexanol

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Cyclohexanol	50 mL	10 mL
Sulfuric acid, 9 <i>M</i> (50:50 concentrated H ₂ SO ₄ :H ₂ O)	25 mL	5 mL
Potassium carbonate, <i>anhydrous</i>	20 g	2 g

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
1-Hexene	30 mL	5 mL
Hydrobromic acid, <i>concentrated</i>	140 mL	20 mL
Methyltriocetylammmonium 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, <i>anhydrous</i>	20 g	2 g

Part B. Qualitative Analysis of Alkyl Halides**1. Silver Nitrate Test***Chemicals:*

	<i>Quantity</i>
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:*

	<i>Quantity</i>
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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
(<i>E</i>)-Stilbene	9 g	1.8 g
Dichloromethane	125 mL	25 mL
Bromine in dichloromethane, 1 <i>M</i>	50 mL	10 mL

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

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
(<i>E</i>)-Stilbene	6 g	1.5 g
Hydrobromic acid, <i>concentrated</i>	15 mL	5 mL
Hydrogen peroxide, 30%	10 mL	3 mL
Ethanol, 95%	140 mL	35 mL
Xylene	100 mL	25 mL

Equipment:

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Pipet, 1-mL, graduated	10	10
Pipet, 2 mL, graduated	10	

Part C. Bromination of (*E*)-Cinnamic Acid*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
(<i>E</i>)-Cinnamic acid	8 g	1.5 g
Acetic acid, <i>glacial</i>	100 mL	15 mL
Pyridinium tribromide	17.6 g	3.3 g
Sodium bisulfite, 10% <i>aqueous</i>	50 mL	10 mL
1:1 95% EtOH:H ₂ O	200 mL	40 mL

10.7 Hydration of Norbornene*Chemicals:*

	<i>Quantity</i>
Sulfuric acid, <i>concentrated</i>	20 mL
Norbornene	10 g
Potassium hydroxide	15 g
Diethyl ether, <i>solvent grade</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Borane in THF, 1 <i>M</i>	50 mL	10 mL
Tetrahydrofuran	20 mL	5 mL
Calcium chloride	100 g	10 g

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

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
(+)- 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, <i>solvent grade</i>	200 mL	40 mL
Sodium chloride	4 g	1 g
Sodium sulfate, <i>anhydrous</i>	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:*

	<i>Quantity</i>
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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
<i>meso</i> -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:*

	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
Sulfuric acid, <i>concentrated</i>	30 mL
Mercuric oxide	2 g
2-methyl-3-butyne-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

<i>Chemicals:</i>	<i>Quantity</i>
Silver nitrate, 0.1 <i>M</i>	25 mL
Ammonium hydroxide	50 mL
2-methyl-3-butyne-2-ol	1 mL
Hydrochloric acid, <i>dilute</i>	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**

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

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Dicyclopentadiene	70 mL	10 mL

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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**

<i>Chemical:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
4-Cyclohexene- <i>cis</i> -1,2-dicarboxylic anhydride	10 g	1g

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemical:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Bicyclo[2.2.1]hept-5-en- <i>endo</i> -1,2-dicarboxylic anhydride	10 g	1g

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
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**

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
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.)

<i>Chemicals:</i>	<i>Quantity</i>
Cyclohexanone	3 mL
<i>Equipment:</i>	
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**

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

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

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

<i>Chemicals:</i>	<i>Quantity</i>
2-Chloro-2-methylbutane	10 g
Phenolphthalein indicator solution	20 mL
2-Propanol	8 L
Sodium hydroxide	60 g
<i>Equipment:</i>	<i>Quantity</i>
Buret, 50-mL	10

14.7 Competing Nucleophiles in S_N Reactions

<i>Chemicals:</i>	<i>Quantity</i>
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

<i>Chemicals:</i>	<i>Quantity</i>
	<i>Microscale (for 2 trials. per student)</i>
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 M 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**15.2 Friedel-Crafts Alkylation of *p*-Xylene with 1-Bromopropane**

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

15.3 Friedel-Crafts Acylation of Anisole*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Zinc oxide	4 g	0.4 g
Benzoyl chloride	12 mL	1.2 mL
Anisole	11 mL	1.1 mL
Sodium carbonate, <i>saturated aqueous</i>	50 mL	30 mL
Sodium chloride, <i>saturated aqueous</i>	50 mL	30 mL
Sodium sulfate, <i>anhydrous</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Nitric acid, <i>concentrated</i>	40 mL	5 mL
Sulfuric acid, <i>concentrated</i>	40 mL	5 mL
Bromobenzene	45 mL	5 mL
Ethanol, 95%	160 mL	20 mL

Equipment:

	<i>Quantity</i>
25- or 50-mL Buret	10

Part B. Thin-Layer Chromatography*Chemicals:*

	<i>Quantity</i>
Dichloromethane	10 mL
Hexane	36 mL
Iodine	10 g
Ethyl acetate	4 mL

Equipment:

	<i>Quantity</i>
Eastman Type K3012R Chromagram sheet or equivalent	1 sheet

Part C. Column Chromatography

<i>Chemicals:</i>	<i>Quantity</i>
Silica gel	50 g
Hexane	360 mL
Ethyl acetate	40 mL
<i>Equipment:</i>	
50-mL Buret	10

15.5 Substituent Effects on Electrophilic Aromatic Substitution**Part A. Relative Rates of Electrophilic Aromatic Bromination****1. Qualitative Measurements**

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

2. Quantitative Measurements

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

Part B. Electrophilic Aromatic Bromination of Monosubstituted Arenes

<i>Chemicals:</i>	<i>Quantity</i>
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% <i>aqueous</i>	50 mL
Sodium bicarbonate, <i>saturated aqueous</i>	50 mL
Deuteriochloroform	10 mL
<i>Equipment</i>	<i>Quantity</i>
Gas trap	10
NMR tube	10

Part C. Electrophilic Aromatic Nitration of Monosubstituted Arenes

<i>Chemicals:</i>	<i>Quantity</i>
	<i>Microscale</i>
Toluene	10.0 g
Methyl benzoate	10.0 g
Chlorobenzene	10.0 g
<i>tert</i> -Butylbenzene	10.0 g
Acetanilide	10.0 g
Nitric acid, <i>concentrated</i>	50 mL
Sulfuric acid, <i>concentrated</i>	50 mL
Acetic acid, <i>glacial</i>	10 mL
Sodium carbonate, <i>saturated aqueous</i>	500 mL
Dichloromethane	250 mL

15.6 Azo Dyes and the Chemistry of Dyeing Fabrics

<i>Chemicals:</i>	<i>Quantity</i>
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.)

<i>Chemicals:</i>	<i>Quantity</i>
Ammonium 8-anilino-1-naphthalenesulfonate	3.5 g
Sodium carbonate	1.5 g
Sodium nitrite	2 g
Sodium chloride	10 g
Hydrochloric acid, <i>concentrated</i>	6 mL
Sodium hydroxide, <i>2.5 M aqueous</i>	20 mL
Sodium chloride, <i>saturated aqueous</i>	20 mL
<i>Equipment:</i>	<i>Quantity</i>
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**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Cyclododecanol	5 g	
Acetic acid, <i>glacial</i>	4 mL	
Acetone	12 mL	
Commercial bleach (5.3% sodium hypochlorite)	60 mL	
Diethyl ether, <i>solvent grade</i>	100 mL	
Sodium bicarbonate, <i>saturated solution</i>	50 mL	
Sodium chloride, <i>saturated solution</i>	50 mL	
Sodium bisulfite, <i>saturated solution</i>	50 mL	
Sodium sulfate, <i>anhydrous</i>	50 g	
<i>Equipment:</i>		
Starch-iodide paper	10 strips	

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Calcium hypochlorite, commercial (65%)	26 g	6 g
Acetic acid, <i>glacial</i>	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*

	<i>Miniscale</i>	<i>Microscale</i>
Diethyl ether, <i>solvent grade</i>	300 mL	60 mL
Sodium bicarbonate, <i>saturated solution</i>	200 mL	40 mL
Hydrochloric acid, <i>concentrated</i>	250 mL	50 mL
Methanol	250 mL	50 mL

Equipment:

Starch-iodide paper	10 strips
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Part C. Aerobic Oxidation of Benzylic Alcohols*Chemicals:**Quantity*

	<i>Miniscale</i>	<i>Microscale</i>
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
<i>N</i> -Methylimidazole	5 mL	2 mL
Acetone, <i>reagent grade</i>	250 mL	130 mL
Pentane	300 mL	200 mL
Magnesium sulfate, <i>anhydrous</i>	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 H₂O.

2. Preparation of Oximes*Chemicals:Quantity*

Hydroxylamine hydrochloride	5 g
Sodium hydroxide 3 <i>M aqueous</i>	8 g

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Potassium hydroxide	50 g	10 g
Methanol	25 mL	5 mL
4-Chlorobenzaldehyde	10 g	2 g
Dichloromethane	160 mL	23 mL
Sodium chloride, <i>saturated solution</i>	100 mL	10 mL
Sodium sulfate, <i>anhydrous</i>	50 g	10 g
Hydrochloric acid, <i>concentrated</i>	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**

<i>Chemicals:</i>	<i>Quantity</i>
Chloroplatinic acid, 5% solution	5 mL
Decolorizing carbon	2 g
Sodium borohydride, 1 <i>M</i> solution	16 mL
Sodium hydroxide, 1% solution	50 mL
4-Cyclohexene- <i>cis</i> -1,2-dicarboxylic acid (see Sec. 12.3, Part D)	5 g
Hydrochloric acid, <i>concentrated</i>	20 mL
Diethyl ether, <i>technical</i>	350 mL
Sodium chloride	100 g
Sodium sulfate, <i>anhydrous</i>	10 g

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
Bromine in dichloromethane solution (see Section 4.7A1)	
Baeyer test (see Section 4.7A2)	
Cyclohexene	2 g
<i>Equipment:</i>	
Syringe, plastic 2-mL	10
Balloons	10
Wire	50 cm

Part B. Transfer Hydrogenation of Cinnamic Acid Derivatives*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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 <i>M</i>	500 mL	45 mL
Diethyl ether, <i>technical</i>	300 mL	150 mL
Methanol	55 mL	25 mL
Ethyl acetate:hexane, 70:30	50 mL	50 mL

Equipment:

	<i>Quantity</i>
Thiele tube	10
or electric melting point apparatus	1
Packing tube	10
Capillary tube	10
TLC chamber	10
TLC plates with fluorescence indicator, 250- μ m precoated as 1 in x 3 in strips	40 strips

Equipment:

	<i>Quantity</i>
Micropipets	10
Whatman GF/A filter discs 2.1 cm	3

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

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Cinnamaldehyde	6 g	1.2 g
<i>m</i> -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
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17.4 Reduction of 9-Fluorenone*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
9-Fluorenone	6 g	1 g
Methanol	100 mL	20 mL
Sodium borohydride	0.5 g	0.1 g
Sulfuric acid, 3 <i>M</i>	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:*

	<i>Quantity</i>
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 <i>M aqueous</i>	100 mL
Sodium bicarbonate, <i>saturated solution</i>	200 mL
Sodium chloride, <i>saturated solution</i>	200 mL
Sodium sulfate, <i>anhydrous</i>	10 g

Part B. Enzymatic Reduction of Methyl Acetoacetate*Chemicals:*

	<i>Quantity</i>
Sucrose	400 g
Disodium hydrogen phosphate	2.5 g
Barium hydroxide, 3% <i>aqueous solution</i>	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

<i>Chemicals:</i>	<i>Quantity</i>
Cyclohexene	2 g
Ferric chloride, 0.2 <i>M aqueous solution</i>	2 mL

17.6 Determining Optical Purity

<i>Chemicals:</i>	<i>Quantity</i>
<i>rac</i> -Methyl 3-hydroxybutanoate	0.3 g
Deuteriochloroform	7.5 mL
<i>tris</i> -[3-(Heptafluoropropylhydroxymethylene)-(+)-camphorato]europium(III)	1 g
<i>Equipment:</i>	
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**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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, <i>saturated solution</i>	200 mL	30 mL
Sodium chloride, <i>saturated solution</i>	50 mL	10 mL
Iodine	750 mg	75 mg
Ethanol, 95%	250 mL	50 mL
Sodium sulfate, <i>anhydrous</i>	50 g	10 g

Qualitative Tests

<i>Chemicals:</i>	<i>Quantity</i>
Cyclohexene	2 g
Bromine in dichloromethane solution (see Section 4.7A1)	
Baeyer test (see Section 4.7A2)	
<i>Equipment:</i>	
Light bulb and socket	1

Part B. Preparation of a Stilbene by the Horner-Wadsworth-Emmons Reaction*Chemicals:**Quantity*

	<i>Miniscale</i>	<i>Microscale</i>
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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:**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

<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:**Quantity*

Cyclohexene	2 g
Bromine in dichloromethane solution (see Section 4.7A1)	
Baeyer test (see Section 4.7A2)	

Equipment:

Syringe, 1-mL	20	20
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18.4 Preparation of 4,4-Dimethyl-2-cyclohexen-1-one*Chemicals:**Quantity*

	<i>Miniscale</i>	<i>Microscale</i>
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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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Sodium bicarbonate	2 g	1 g
Sodium sulfate, <i>anhydrous</i>	30 g	3 g
Diethyl ether, <i>solvent grade</i>	30 mL	

Qualitative Tests*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Benzaldehyde	13.3 g	2.7 g
Ethyl acetoacetate	16.3 g	3.3 g
Urea	9.5 g	1.9 g
<i>p</i> -Toluenesulfonic acid	1.0 g	0.2 g
Ethanol, <i>anhydrous</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Magnesium turnings	5 g	0.5 g
Diethyl ether, <i>anhydrous</i>	150 mL	50 mL
Bromobenzene	39 g	3.9 g

n-Butylmagnesium Bromide*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
1-Bromobutane	34 g	3.4 g
Magnesium turnings	5 g	0.5 g
Diethyl ether, <i>anhydrous</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Methyl benzoate	12 mL	1.2 mL
Diethyl ether, <i>anhydrous</i>	100 mL	20 mL
Diethyl ether, <i>solvent grade</i>	200 mL	40 mL
Sulfuric acid, 6 <i>M</i>	150 mL	

Chemicals:

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Sulfuric acid, 3 <i>M</i>	150 mL	20 mL
Sodium bicarbonate, <i>saturated solution</i>	100 mL	10 mL
Sodium chloride, <i>saturated solution</i>	50 mL	5 mL
Sodium sulfate, <i>anhydrous</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Dry ice	100 g	10 g
Diethyl ether, <i>anhydrous</i>	50 mL	10 mL

Part B. Preparation of Benzoic Acid (cont.)*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Diethyl ether, <i>solvent grade</i>	400 mL	40 mL
Sulfuric acid, 3 <i>M</i>	100 mL	15 mL
Sodium hydroxide, 1 <i>M</i>	200 mL	20 mL
Hydrochloric acid, 6 <i>M</i>	100 mL	10 mL

Equipment:

Screw-cap centrifuge tube	20
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Part C. Preparation of 2-Methyl-3-heptanol*Chemicals:*

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

19.5 Preparation of 3-Ethylhex-5-en-3-ol*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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 <i>anhydrous</i>	130 mL	20 mL
Diethyl ether	50 mL	5 mL
Hydrochloric acid, 1 <i>M aqueous</i>	30 mL	10 mL
Sodium chloride, <i>saturated solution</i>	50 mL	20 mL
Sodium bicarbonate, <i>saturated solution</i>	50 mL	20 mL
Sodium thiosulfate, <i>saturated solution</i>	50 mL	10 mL
Sodium sulfate, <i>anhydrous</i>	5.0 g	0.5 g

19.6 Preparation of 4'-Methyl-(1,1'-biphenyl)-4-methanol*Chemicals:**Quantity**Miniscale Microscale*

4-Methylphenylboronic acid	7.0 g	1.4 g
4-Bromobenzyl alcohol	10.0 g	2.0 g
Palladium, 1000 ppm <i>aqueous solution</i>	10 mL	2 mL
Potassium hydroxide, 1 <i>M ethanolic solution</i>	100 mL	20 mL
Ethanol, 95%	120 mL	60 mL
Dichloromethane	250 mL	100 mL
Magnesium sulfate, <i>anhydrous</i>	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*

<i>p</i> -Aminobenzoic acid	10 g	2 g
Ethanol, <i>absolute</i>	130 mL	25 mL
Sulfuric acid, <i>concentrated</i>	15 mL	3 mL
Sodium carbonate, 10% <i>aqueous solution</i>	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 (<i>reagent grade</i>)	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% <i>aqueous solution</i>	30 mL
Sodium chloride, <i>saturated solution</i>	30 mL
Sulfuric acid, <i>concentrated</i>	3 mL
Diethyl ether, <i>technical</i>	30 mL
Sodium sulfate, <i>anhydrous</i>	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
<i>n</i> -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
<i>n</i> -Propyl propanoate	14.68	7.52
<i>n</i> -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
<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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
3-Methylbenzoic acid (<i>m</i> -toluic acid)	20 g	2 g
Thionyl chloride	22 mL	2 mL
Diethylamine	50 mL	5 mL
Diethyl ether, <i>anhydrous</i>	400 mL	30 mL
Sodium hydroxide, 2.5 <i>M</i>	150 mL	15 mL
Hydrochloric acid, 3 <i>M</i>	150 mL	15 mL
Sodium sulfate, <i>anhydrous</i>	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:*

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

Part B. Chemiluminescence*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Sodium hydroxide, 3 <i>M</i>	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**

<i>Chemicals:</i>	<i>Quantity</i>
Nitrobenzene	52 mL
Tin powder	131 g
Hydrochloric acid, <i>concentrated</i>	325 mL
Sodium hydroxide, 12 <i>M</i>	500 mL
Sodium chloride	300 g
Diethyl ether, <i>solvent grade</i>	600 mL
Sodium sulfate, <i>anhydrous</i>	100 g
<i>Equipment:</i>	
Steam distillation apparatus	10

Part B. Preparation of Acetanilide

<i>Chemicals:</i>	<i>Quantity</i>
Aniline	36 mL
Hydrochloric acid, 0.4 <i>N</i> (33 mL of <i>concentrated</i> 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

<i>Chemicals:</i>	<i>Quantity</i>
Acetanilide (Not required if prepared in Part B.)	27 g
Chlorosulfonic acid	80 mL
Dichloromethane	100 mL
<i>Equipment:</i>	
Gas trap	10

Part D. Preparation of 4-Acetamidobenzenesulfonamide

<i>Chemicals:</i>	<i>Quantity</i>
Ammonium hydroxide, <i>concentrated</i>	150 mL

Part E. Preparation of Sulfanilamide

<i>Chemicals:</i>	<i>Quantity</i>
Hydrochloric acid, 6 <i>M</i>	300 mL
Sodium carbonate	10 g

Solubility Tests

<i>Chemicals:</i>	<i>Quantity</i>
Hydrochloric acid, 1.5 <i>M</i> (0.4 mL of <i>concentrated</i> HCl/3 mL of solution)	3 mL
Sodium hydroxide, 1.5 <i>M</i> (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**

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Bromine	32 mL	1.5 mL
Acetic acid, <i>glacial</i>	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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Hydrochloric acid, <i>concentrated</i>	230 mL	10 mL
Acetic acid, <i>glacial</i>	280 mL	13 mL
Sodium Chlorate	28 g	1.5 g
Methanol	100 mL	10 mL

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Hydrochloric acid, <i>concentrated</i>	130 mL	5 mL
Ethanol, 95%	200 mL	20 mL
Sodium hydroxide, 14 <i>N</i>	120 mL	50 mL
Methanol	25 mL	10 mL

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Acetic acid, <i>glacial</i>	750 mL	75 mL
Iodine monochloride, <i>technical</i>	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:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Sulfuric acid, <i>concentrated</i>	40 mL	4 mL
Ethanol, <i>absolute</i>	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
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21.4 Lidocaine: Synthesis of an Anesthetic Agent**Part A. Preparation of 2,6-Dimethylaniline***Chemicals:*

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

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

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
α -Chloroacetyl chloride	28 g	2.8 g
Acetic acid, <i>glacial</i>	200 mL	20 mL
Sodium acetate trihydrate	43 g	5 g

Part C. Preparation of Lidocaine*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
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, <i>solvent grade</i>	300 mL	30 mL

Part C. Preparation of Lidocaine (cont.)*Chemicals:*

	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Sodium sulfate, <i>anhydrous</i>	25 g	3 g
Sulfuric acid, 2.2 <i>M</i> 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:*

	<i>Quantity</i>
Styrene, commercial	100 mL
Sodium hydroxide, 3 <i>M</i> (4.8 g of NaOH/40 mL of solution)	40 mL
Calcium chloride	8 g

Part B. Polymerization of Pure Styrene*Chemicals:*

	<i>Quantity</i>
Styrene, <i>anhydrous</i> (see Part A)	30 mL
<i>tert</i> -Butyl peroxybenzoate	2 mL

Equipment:

Microburners	10
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Part C. Solution Polymerization of Styrene*Chemicals:*

	<i>Quantity</i>
Styrene, <i>anhydrous</i> (see Part A)	30 mL
Xylene, commercial mixture of isomers	60 mL
<i>tert</i> -Butyl peroxybenzoate	1 mL
Methanol	250 mL

22.3 Preparation of Nylon-6,10*Chemicals:*

	<i>Quantity</i>
Decanedioyl dichloride (sebacoyl chloride)	20 mL
Dichloromethane	1 L
1,6-Hexanediamine (hexamethylenediamine) crystals <i>or</i>	12 g
80–95% <i>aqueous solution</i>	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
<i>either</i> Drum, made from a coffee, juice, or motor oil can	5
forceps	5
<i>or</i> Copper wire	100 cm

CH 23 Carbohydrates**23.3 Hydrolysis of Sucrose**

<i>Chemicals:</i>	<i>Quantity</i>
Sucrose	75 g
Hydrochloric acid, <i>concentrated</i>	5 mL

23.4 Classification Tests for Carbohydrates**Tollens's Test**

<i>Chemicals:</i>	<i>Quantity</i>
Silver nitrate	2.5 g
Water, <i>distilled</i>	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* 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

<i>Chemicals:</i>	<i>Quantity</i>
Sodium citrate, dihydrate	26 g
Sodium carbonate, <i>anhydrous</i>	15 g
Cupric sulfate	2.6 g

Barfoed's Test

<i>Chemicals:</i>	<i>Quantity</i>
Cupric acetate	6 g
Acetic acid, <i>glacial</i>	0.9 mL

Formation of Osazones

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

Formation of Osazones (cont.)

<i>Chemicals:</i>	<i>Quantity</i>
<i>either</i> Acetic acid, <i>glacial</i> 6 mL	
Sodium acetate	6 g
Phenylhydrazine	4 g
<i>or</i> 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**

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

Part B. Preparation of Methyl L-Phenylalaninate Hydrochloride

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
L-Phenylalanine	10 g	2 g
Methanol	100 mL	25 mL
Thionyl chloride	5 mL	1 mL
Diethyl ether, <i>technical</i>	500 mL	100 mL

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

<i>Chemicals:</i>	<i>Quantity</i>	
	<i>Miniscale</i>	<i>Microscale</i>
Dimethylformamide	200 mL	20 mL
<i>N</i> -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*

	<i>Miniscale</i>	<i>Microscale</i>
Diethyl ether, <i>technical</i>	750 mL	200 mL
Hydrochloric acid, 1 <i>M</i>	500 mL	80 mL
Sodium bicarbonate, <i>saturated solution</i>	250 mL	50 mL
Sodium chloride, <i>saturated solution</i>	250 mL	50 mL
Sodium sulfate, <i>anhydrous</i>	25 g	5 g
Hexanes	150 mL	40 mL

Part D. Preparation of Methyl L-Alanyl-L-phenylalaninate Trifluoroacetate*Chemicals:**Quantity*

	<i>Miniscale</i>	<i>Microscale</i>
Trifluoroacetic acid	15 mL	2 mL
Dichloromethane	60 mL	10 mL
Diethyl ether, <i>technical</i>	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 about 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* 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.

Hydron 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 H₂O 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 H₂O.

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 H₂O and then add 4 mL of saturated aqueous sodium bisulfite. Allow this solution to stand for 1 h 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* 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. 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 C₂H₅NO										
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 <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.				
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 $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3\text{S}$										
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 <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.				
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 ₈ H ₈ ClNO ₃ S										
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.

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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	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 <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.				
Carcinogenicity		Not a known carcinogen.		Mutagenicity		Possible mutagen.				

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Acetic Acid (<i>glacial</i>) C₂H₄O₂										
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.		<i>No</i> flames, <i>no</i> sparks, <i>no</i> 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 <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.				
Carcinogenicity		Not a known carcinogen.		Mutagenicity		Possible mutagen.				

For more detailed information, consult the [Material Safety Data Sheet](#) for this compound.

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Acetic Anhydride C₄H₆O₃										
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.			

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Acetone C₃H₆O										
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.

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<i>p</i>-Acetophenetidide [Phenacetin]										
C₁₀H₁₃NO₂										
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, <i>no</i> sparks, <i>no</i> 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 <i>not</i> 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.

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Acetophenone C₈H₈O										
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.

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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 <i>not</i> eat or drink in the laboratory.			If swallowed wash out mouth with water. Seek medical advice.		
Carcinogenicity		Not a known carcinogen.			Mutagenicity			Not a known mutagen.		

For more detailed information consult the [Material Safety Data Sheet](#) for this compound.

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