

Contents

Preface xxi

Introduction 1

Important Features of the Organic Lab 1

Goals for the Organic Laboratory 1

Working in the Laboratory 2

Laboratory Safety 3

Material Safety Data Sheet (MSDS) 5

Your Laboratory Notebook 6

Laboratory Reports 6

How to Be a Successful Organic Laboratory Student 7

Chapter One **Techniques in the Organic Chemistry Laboratory** 9

Technique A **Glassware and Equipment: *Heating and Cooling*** 9

Microscale Glassware and Related Equipment 9

Miniscale Glassware 11

Additional Glassware and Equipment 13

How to Clean Glassware 13

How to Heat and Cool Glass Reaction Vessels 14

Technique B **Weighing and Measuring** 17

How to Weigh Solids and Liquids 17

Introduction to Measuring Volumes of Liquids 17

How to Use a Calibrated Glass Pipet 18

How to Use an Automatic Delivery Pipet 19

How to Use a Syringe 19

Exercise B.1: Determining Density of an Aqueous Solution 20

Exercise B.2: Determining Density of an Organic Liquid 21

Exercise B.3: Calibrating a Pasteur Pipet 21

Technique C **Melting Points** 22

Mixed Melting Behavior 23

Melting Behavior of Solids 23

Calibration of the Thermometer 24

Apparatus for Measuring Melting Points 24

How to Determine a Melting Point 24

Exercise C.1: Calibration of a Thermometer 26

Exercise C.2: Melting Point of an Unknown Solid 26

Exercise C.3: Mixed Melting Point 27









Technique D	Boiling Points	28
	Intermolecular Attractions	28
	How to Do a Microscale Boiling Point Determination	29
	How to Do a Miniscale Boiling Point Determination	29
	<i>Exercise D.1: Determining the Micro Boiling Point of an Unknown Liquid</i>	30
	<i>Exercise D.2: Determining the Boiling Point of an Unknown Liquid</i>	30
Technique E	Index of Refraction	32
	How to Use the Abbe Refractometer	33
	<i>Exercise E.1: Measuring the Refractive Index of an Unknown Liquid</i>	33
Technique F	Recrystallization, Filtration, and Sublimation	34
	Solvents for Recrystallization	35
	Choosing a Solvent	38
	Choosing a Solvent Pair	38
	How to Do a Microscale Recrystallization	39
	How to Do a Miniscale Recrystallization	41
	Important Tips Concerning Recrystallization	42
	Introduction to Filtration	43
	How to Use a Microscale Filter Pipet	44
	How to Do a Miniscale Suction Filtration	44
	How to Do a Miniscale Gravity Filtration	44
	Important Tips Concerning Filtration	45
	Introduction to Sublimation	45
	<i>Exercise F.1: Recrystallizing an Impure Solid (microscale)</i>	48
	<i>Exercise F.2: Recrystallizing an Impure Solid (miniscale)</i>	48
	<i>Exercise F.3: Recrystallizing an Impure Solid with Hot Gravity Filtration</i>	49
	<i>Exercise F.4: Purifying an Unknown Solid by Solvent-Pair Recrystallization</i>	50
	<i>Exercise F.5: Sublimation of Caffeine</i>	50
	<i>Exercise F.6: Sublimation of Caffeine</i>	51
Technique G	Distillation and Reflux	52
	Microscale Apparatus for Simple Distillation and Assembly	56
	How to Do a Simple Microscale Distillation	56
	Important Tips Concerning Microscale Distillation	58
	Miniscale Apparatus for Simple Distillation	59
	How to Do a Simple Miniscale Distillation	59
	Important Tips Concerning Miniscale Distillation	60
	Introduction to Reflux	61
	How to Do a Microscale Reaction with Reflux	61

	How to Do a Miniscale Reaction with Reflux	62
	Important Tips on Reflux	62
	Evaporation of Solvents	62
	<i>Exercise G.1: Distilling a Mixture of Cyclohexane and Toluene (microscale)</i>	63
	<i>Exercise G.2: Distilling a Mixture of Cyclohexane and Toluene (miniscale)</i>	64
Technique H	Fractional Distillation and Steam Distillation	65
	How to Do a Miniscale Fractional Distillation	67
	Practical Tips about Miniscale Fractional Distillation	68
	Introduction to Steam Distillation	69
	<i>Exercise H.1: Miniscale Fractional Distillation of a Mixture of Cyclohexane and Toluene</i>	70
	<i>Exercise H.2: Microscale Fractional Distillation of Cyclohexane and Toluene</i>	70
	<i>Exercise H.3: Steam Distillation of Lemon Grass Oil (miniscale)</i>	71
Technique I	Extraction and Drying	72
	How to Do a Microscale Extraction	74
	How to Do a Miniscale Extraction	76
	Important Tips Concerning Extraction	76
	Drying and Drying Agents	78
	How to Do Microscale Drying	78
	How to Do Miniscale Drying	79
	Important Tips Concerning Drying Agents	79
	<i>Exercise I.1: Determining the Distribution Coefficient of Caffeine (microscale)</i>	80
	<i>Exercise I.2: Determining the Distribution Coefficient of Caffeine (miniscale)</i>	80
	<i>Exercise I.3: Using Distribution Coefficients to Identify an Unknown Solid (microscale)</i>	81
Technique J	Gas-Liquid Chromatography	83
	Gas-Liquid Chromatography Basics	83
	Quantitative Analysis	87
	How to Use the Gas Chromatograph	89
	<i>Exercise J.1: Determining Relative Detector Response Factors in GC</i>	89
	<i>Exercise J.2: Determining Mass Percent of a Mixture Using GC</i>	90
	<i>Exercise J.3: Determining Mass Percent of a Mixture of Alcohols</i>	90
Technique K	Thin-Layer, Column, and High-Performance Liquid Chromatography	92
	Introduction to Thin-Layer Chromatography (TLC)	92
	Introduction to Column Chromatography	97
	How to Do a Miniscale Gravity Column Chromatography	98



	How to Do Microscale Column Chromatography	99
	Introduction to High-Performance Liquid Chromatography (HPLC)	99
	<i>Exercise K.1: Analysis of Analgesic Tablets by TLC</i>	101
	<i>Exercise K.2: Separating Ferrocene and Acetylferrocene Using Column Chromatography (microscale)</i>	101
	<i>Exercise K.3: Separating Ferrocene and Acetylferrocene Using Column Chromatography (miniscale)</i>	102
	<i>Exercise K.4: HPLC Analysis of Benzaldehyde and Benzyl Alcohol</i>	102
Technique L	Polarimetry	103
	Optical Purity and Enantiomeric Excess	104
	How to Measure Optical Rotation	106
	<i>Exercise L.1: Determining the Optical Purity of an Unknown Liquid</i>	107
	<i>Exercise L.2: Determining the Melting Points of Enantiomers and Racemates</i>	107
Chapter Two	Spectroscopic Methods and Molecular Modeling	109
Technique M	Infrared Spectroscopy	110
	General Approach to Solving an IR Spectrum	116
	Recording an IR Spectrum	116
	How to Prepare a Sample for IR Analysis	118
	Important Tips Concerning IR	120
	<i>Exercise M.1: Recording the IR Spectrum of an Organic Liquid</i>	122
	<i>Exercise M.2: Recording the IR Spectrum of an Organic Solid</i>	122
	<i>Exercise M.3: Spectroscopic Identification of Unknowns</i>	122
Technique N	Nuclear Magnetic Resonance Spectroscopy	125
	Chemical Shifts in ^1H NMR Spectroscopy	127
	Equivalence of Protons in ^1H NMR Spectroscopy	129
	Integration in ^1H NMR Spectroscopy	130
	Splitting (coupling) in ^1H NMR Spectroscopy	131
	Coupling Constants	134
	Protons in a Chiral Environment	135
	Diastereotopic Protons in Alkenes	136
	Protons on Heteroatoms	137
	Solvents for ^1H NMR Spectroscopy	139
	How to Prepare a Sample for 60–90 MHz CW ^1H NMR Spectroscopy	140
	How to Prepare a Sample for FT NMR	140
	Structural Identification from the NMR Spectrum	140
	Introduction to ^{13}C NMR Spectroscopy	143
	Solvents for ^{13}C NMR Spectroscopy	146
	How to Prepare a Sample for ^{13}C NMR Spectroscopy	146

	General Approach to Determining an Unknown Structure	146
	<i>Exercise N.1: Recording a ^1H NMR Spectrum</i>	148
	<i>Exercise N.2: Recording a ^{13}C NMR Spectrum</i>	148
	<i>Exercise N.3: ^1H NMR Spectral Problems</i>	148
	<i>Exercise N.4: ^{13}C NMR Spectral Problems</i>	150
	<i>Exercise N.5: Spectral Identification Using ^1H and ^{13}C NMR</i>	151
Technique O	Ultraviolet and Visible Spectroscopy	154
	Uses of UV-Visible Spectroscopy	156
	How to Operate a Spectronic 20	158
	How to Operate a UV-Visible Spectrometer	159
Technique P	Mass Spectrometry	160
	Principles of Mass Spectrometry	160
	Fragmentation Patterns	163
	Molecular Ion Peak	166
	Isotopes	167
	Gas Chromatography/Mass Spectrometry (GC/MS)	167
	Strategy for Solving Structural Problems Using MS, IR, and NMR	168
	Mass Spectrometric Analysis Guidelines	168
	<i>Exercise P.1: Solving Problems in Mass Spectrometry</i>	172
Technique Q	Molecular Modeling	173
	Organic Molecular Models	174
	Drawing Organic Molecules Using Computer Software	174
	Molecular Modeling Using Computer Software	174
	Molecular Mechanics	175
	Quantum Mechanics	178
	Exercises	180
	<i>Exercise Q.1: Conformational Analysis of Butane and Other Molecules</i>	180
	<i>Exercise Q.2: Conformational Analysis of Propane</i>	180
	<i>Exercise Q.3: Conformational Analysis of 2-Methylbutane</i>	181
	<i>Exercise Q.4: Identifying Reactive Sites</i>	181
	<i>Exercise Q.5: Heats of Formation and Dipole Moments</i>	181
	<i>Exercise Q.6: LUMO Energies of Alkenes</i>	181
	<i>Exercise Q.7: Conformational Analysis of 3-Fluoropropene</i>	182
Chapter Three	Applications Using Laboratory Resources and Techniques	183
Experiment 3.1	Scavenger Hunt: Introduction to Chemical Data Reference Books and Calculations	184
	<i>Part A: Scavenger Hunt in the Chemical Reference Books</i>	187
	<i>Part B: Scavenger Hunt and Stoichiometric Calculations</i>	188

-  **Experiment 3.2 Identification of Organic Liquids by Physical Properties** 189
General Directions 190
Part A: Boiling Point Determination 190
Part B: Refractive Index Determination 190
-  **Experiment 3.3 Relationships Between Structure and Physical Properties** 192
Part A: Determination of the Effect of Structure on Boiling Point 194
Part B: Determination of the Effect of Structure on Refractive Index 194
Part C: Determination of the Effect of Structure on Melting Point 194
- Experiment 3.4 Properties of Solvents and Recrystallization of Organic Solids** 197
 *Part A: Selecting an Appropriate Solvent* 198
Part B: Microscale Recrystallization of an Organic Solid 199
- Experiment 3.5 Separations Based upon Acidity and Basicity** 200
 *Part A: Determination of Solubilities* 202
Part B: Microscale Separation of Naphthalene, Benzoic Acid, and Ethyl 4-Aminobenzoate 203
Part C: Miniscale Separation of Benzoic Acid and Ethyl 4-Aminobenzoate 204
- Experiment 3.6 Isolation of a Natural Product** 205
Part A: Miniscale Extraction of Caffeine from Tea Leaves 207
Part B: Miniscale Extraction of Caffeine from Instant Coffee 208
Part C: Miniscale Isolation of Caffeine from NoDoz 209
Part D: Miniscale Isolation of Cholesterol from Simulated Gallstones 209
- Experiment 3.7 Solvent and Polarity Effects in Thin-layer Chromatography (TLC)** 210
 *Part A: Determining the Effect of Polarity on Elution* 212
 *Part B: Separation and Identification of Components of a Mixture of trans-Stilbene, 9-Fluorenone, and Benzoic Acid* 214
-  **Experiment 3.8 Purification and Analysis of a Liquid Mixture: Simple and Fractional Distillation** 216
Part A: Microscale Distillation 217
Part B: Microscale Fractional Distillation 218
Part C: Miniscale Distillation 218
Part D: Miniscale Fractional Distillation 219
- Chapter Four Alcohols and Alkyl Halides** 221
- Experiment 4.1 Synthesis of an Alkyl Halide from an Alcohol** 221
-  **Experiment 4.2 Selectivity of Free Radical Chlorination of 2,3-Dimethylbutane** 224

Chapter Five **Synthesis of Alkenes** 229**Experiment 5.1** **Alkenes Via Acid-catalyzed Dehydration of Alcohols** 229

Part A: Microscale Synthesis of Alkenes Via Acid-catalyzed Dehydration of 3-Methyl-3-pentanol 231

Part B: Miniscale Synthesis of Alkenes Via Acid-catalyzed Dehydration of 3,3-Dimethyl-2-butanol 231

Part C: Microscale Synthesis of Alkenes Via Acid-catalyzed Dehydration of an Unknown Alcohol 232

**Experiment 5.2** **Alkenes Via Base-Induced Elimination of Alkyl Halides** 234**Chapter Six** **Alkene Addition Reactions** 237**Experiment 6.1** **Catalytic Hydrogenation of Alkenes** 237

Part A: Microscale Hydrogenation of 1-Decene 239

Part B: Microscale Hydrogenation of Allylbenzene 240

Part C: Microscale Partial Hydrogenation of Olive Oil 241

**Experiment 6.2** **Hydration of Alkenes** 242

Part A: Microscale Hydration of 2-Ethyl-1-butene 244

Part B: Microscale Hydration of Norbornene 245

Part C: Miniscale Hydration of Norbornene 245

Experiment 6.3 **Preparation of Alcohols from Alkenes by Hydroboration-oxidation** 246

Part A: Microscale Hydroboration-oxidation of 3,3-Dimethyl-1-butene 249

Part B: Miniscale Hydroboration-oxidation of 3,3-Dimethyl-1-butene 249

Experiment 6.4 **Addition Polymers: Preparation of Polystyrene and Polymethyl Methacrylate** 251

Part A: Miniscale Polymerization of Styrene (Bulk Method) 253

Part B: Microscale Polymerization of Styrene (Solution Method) 253

Part C: Miniscale Polymerization of Methyl Methacrylate (Bulk Method) 253

Chapter Seven **Stereochemistry** 255**Experiment 7.1** **Stereochemistry of Alkenes and Derivatives** 255

Part A: Microscale Cis-Trans Isomerization of an Alkene 258









Part B: Microscale Addition of Bromine to Fumaric Acid 258

Part C: Determining the Mechanism of the Isomerization of Dimethyl Maleate to Dimethyl Fumarate (Miniscale) 259

Chapter Eight **Introduction to Nucleophilic Substitution Reactions** 261**Experiment 8.1** **Relative Rates of Nucleophilic Substitution Reactions** 261






Part A: Determination of Factors Affecting the Relative Rates of S_N2 Reactions 263

Part B: Determination of Factors Affecting the Relative Rates of S_N1 Reactions 264

- Experiment 8.2 Nucleophilic Aliphatic Substitution: Synthesis of 1-Bromobutane** 267
Part A: Microscale Synthesis of 1-Bromobutane 268
Part B: Miniscale Synthesis of 1-Bromobutane 269
- Chapter Nine Dienes and Conjugation** 271
- Experiment 9.1 Dienes and the Diels-Alder Reaction** 271
Part A: Microscale Reaction of 1,3-Butadiene with Maleic Anhydride 276
Part B: Miniscale Reaction of 2,3-Dimethyl-1,3-butadiene with Maleic Anhydride 277
Part C: Microscale Reaction of Cyclopentadiene with Maleic Anhydride 277
 *Part D: Miniscale Reaction of Cyclopentadiene with Maleic Anhydride* 277
 *Part E: Miniscale Reaction of Anthracene with Maleic Anhydride* 278
- Chapter Ten Qualitative Organic Analysis I** 281
-  **Experiment 10.1 Qualitative Analysis of Alkyl Halides, Alkenes, Dienes, and Alkynes** 281
- Chapter Eleven Reactions of Aromatic Side Chains** 290
- Experiment 11.1 Benzylic Oxidation: Benzoic Acid from Toluene; A Phthalic Acid from an Unknown Xylene** 290
 *Part A: Microscale Oxidation of Toluene to Benzoic Acid* 292
Part B: Microscale Oxidation of a Xylene to a Phthalic Acid 293
- Chapter Twelve Electrophilic Aromatic Substitution** 298
-  **Experiment 12.1 Activating and Deactivating Effects of Aromatic Substituents: Relative Rates of Bromination** 299
- Experiment 12.2 Nitration of Methyl Benzoate or an Unknown** 304
Part A: Microscale Nitration of Methyl Benzoate 306
Part B: Miniscale Nitration of Methyl Benzoate 306
 *Part C: Miniscale Nitration of an Unknown Aromatic Compound* 307
- Experiment 12.3 Friedel-Crafts Acylation Reactions** 310
 *Part A: Microscale Procedure for Acetylation of Biphenyl* 312
Part B: Microscale Procedure for Acetylation of Phenanthrene with TLC Analysis 313
Part C: Microscale Benzoylation of Ferrocene with Column Chromatography 314
- Experiment 12.4 Aromatic Bromination** 316
 *Part A: Microscale Bromination of Acetanilide* 317
Part B: Microscale Bromination of p-Methylacetanilide with TLC and Column Chromatography or HPLC Analysis 319
Part C: Experimental Design of a Procedure for Bromination of an Acetanilide Derivative 320


- Chapter Thirteen** **Combined Spectroscopy and Advanced Spectroscopy** 323
- Experiment 13.1** **Infrared and Nuclear Magnetic Resonance Spectroscopy of Alcohols, Ethers, and Phenols** 323
- Part A: Experimental Procedure for Infrared Spectroscopy* 326
- Part B: Experimental Procedure for IR and NMR Spectroscopy* 326
- Part C: Exercises for IR and NMR Spectroscopy* 327
- Experiment 13.2** **Combined Spectral Analysis: Infrared, Ultraviolet, and Nuclear Magnetic Resonance Spectroscopy and Mass Spectrometry** 332
- Solved Examples of the Use of Combined Spectroscopy** 333
- Part A: Purification of the Unknown* 341
- Part B: Preparation of the Sample and Spectroscopic Analysis* 341
- Part C: Spectroscopic Exercises* 342
- Chapter Fourteen** **Organometallics** 351
- Experiment 14.1** **Grignard Synthesis: Preparation of Triphenylmethanol and 3-Methyl-2-phenyl-2-butanol** 351
- Part A: Microscale Synthesis of Triphenylmethanol from Benzophenone* 354
- Part B: Miniscale Synthesis of Triphenylmethanol from Ethyl Benzoate* 355
- Part C: Microscale Synthesis of 3-Methyl-2-phenyl-2-butanol* 358
-  **Experiment 14.2** **Using Indium Intermediates: Reaction of Allyl Bromide with an Aldehyde** 360
- Chapter Fifteen** **Alcohols and Diols** 364
-  **Experiment 15.1** **Stereoselective Reduction of Ketones with Sodium Borohydride** 364
- Part A: Microscale Reduction of Benzil* 366
- Part B: Microscale Reduction of (1R)-(+)-Camphor* 366
-  **Experiment 15.2** **Experimental Design for an Alcohol Oxidation** 369
-  **Experiment 15.3** **Photochemical Oxidation of Benzyl Alcohol** 373
- Chapter Sixteen** **Ethers** 376
- Experiment 16.1** **Ether Synthesis by S_N2 Displacement** 376
- Part A: Microscale Preparation of an Alkyl Halophenyl Ether* 378
- Part B: Miniscale Preparation of Benzyl tert-Butyl Ether* 379
-  **Experiment 16.2** **Nucleophilic Aliphatic Substitution Puzzle: Substitution Versus Elimination** 380
- Chapter Seventeen** **Aldehydes and Ketones** 385
- Experiment 17.1** **Stereoselective Synthesis of Alkenes** 386
- Part A: Microscale Wittig Synthesis of trans-9-(2-phenylethenyl)anthracene* 388

- Part B: Miniscale Wittig Synthesis of *trans*-9-(2-phenylethenyl)anthracene** 389
- Part C: Microscale Horner-Emmons Reaction of Diethylbenzyl Phosphonate and Benzaldehyde** 390
- Experiment 17.2 Conversion of Cyclohexanone to Caprolactam** 393
- Part A: Microscale Conversion of Cyclohexanone to Caprolactam* 395
- Part B: Miniscale Conversion of Cyclohexanone to Caprolactam* 395
- Experiment 17.3 Pinacol Rearrangement and Photochemical Synthesis of Benzopinacol** 399
- Part A: Microscale Synthesis of Pinacolone* 400
- Part B: Miniscale Photoreduction of Benzophenone* 401
- Part C: Miniscale Synthesis of Benzopinacolone* 402
- Chapter Eighteen Enols, Enolates, and Enones** 404
- Experiment 18.1 Preparation of α,β -Unsaturated Ketones Via Mixed Aldol Condensation** 404
- Part A: Miniscale Synthesis of 1,9-Diphenyl-1,3,6,8-nonatetraen-5-one (Dicinnamalacetone)* 407
- Part B: Microscale Synthesis of Dibenzalacetone (1,5-Diphenyl-1,4-pentadien-3-one)* 408
- Part C: Identification of a Mixed Aldol Product from the Miniscale Condensation of an Unknown Aromatic Aldehyde with a Ketone* 410
- Experiment 18.2 Reduction of Conjugated Ketones with Sodium Borohydride** 411
- Part A: Microscale Reduction of 2-Cyclohexenone* 412
- Part B: Microscale Reduction of *trans*-4-Phenyl-3-buten-2-one* 413
- Experiment 18.3 Identification of Products of Catalytic Transfer Hydrogenation of an Enone** 414
- Part A: Microscale Reaction of 2-Cyclohexenone* 416
- Part B: Miniscale Reaction of 2-Cyclohexenone* 416
- Chapter Nineteen Carboxylic Acids** 419
- Experiment 19.1 Synthesis and Identification of an Unknown Carboxylic Acid** 419
- Experiment 19.2 Synthesis of *trans*-Cinnamic Acid Via the Haloform Reaction** 425
- Chapter Twenty Carboxylic Acid Esters** 430
- Experiment 20.1 Combinatorial Chemistry and the Synthesis of Fruity Esters** 430
- Part A: Combinatorial Selection* 432
- Part B: Microscale Synthesis of an Ester* 433
- Experiment 20.2 Synthesis of Esters by Baeyer-Villiger Oxidation** 436
- Part A: Microscale Oxidation of an Unknown Cyclic Ketone* 437
- Part B: Miniscale Oxidation of Cyclohexanone* 438

- Chapter Twenty-One** **Dicarbonyl Compounds** 441
-  **Experiment 21.1** **Base-Catalyzed Condensations of Dicarbonyl Compounds** 441
- Part A: Microscale Preparation of Coumarin or a Coumarin Derivative* 444
- Part B: Miniscale Preparation of Coumarin or a Coumarin Derivative* 444
-  **Experiment 21.2** **Reactions of Diketones: Synthesis of 2,5-Dimethyl-1-phenylpyrrole and Preparation of an Unknown Pyrrole** 445
- Part A: Microscale Synthesis of 2,5-Dimethyl-1-phenylpyrrole* 447
- Part B: Microscale Preparation of an Unknown Pyrrole* 448
- Chapter Twenty-Two** **Amines** 450
- Experiment 22.1** **Relating Color to Structure: Synthesis of Azo Dyes** 450
- Part A: Miniscale Diazotization of an Aromatic Amine* 454
- Part B.1: Miniscale Coupling with a Phenol* 455
- Part B.2: Miniscale Coupling with an Amine* 455
- Part C: Direct Dyeing with an Azo Dye* 455
- Part D: Recording the UV-Visible Spectrum of the Prepared Dyes* 455
- Part E: Determining the pH Indicator Range of the Prepared Dyes* 455
- Part F: Determining Antibacterial Properties of the Dye* 456
-  **Experiment 22.2** **Synthesis of Pyrazole and Pyrimidine Derivatives** 457
- Part A: Microscale Synthesis of a Five-Member Ring Heterocycle from Hydrazine and an Unknown Diketone* 461
- Part B: Miniscale Synthesis of a Five-Member Ring Heterocycle from Hydrazine and an Unknown Diketone* 461
- Part C: Microscale Synthesis of a Substituted Pyrimidine* 462
- Part D: Miniscale Synthesis of a Substituted Pyrimidine* 462
-  **Experiment 22.3** **Synthesis of Heterocyclic Compounds** 464
- Part A: Miniscale Reaction of Benzaldehyde and Pyrrole* 466
- Part B: Microscale Reaction of o-Phenylenediamine and Formic Acid* 467
-  **Experiment 22.4** **Synthesis of Heterocycles and Kinetics by Spectroscopic Analysis** 469
- Part A: Miniscale Preparation of Lophines (2-Aryl-4,5-diphenylimidazoles)* 471
- Part B: Miniscale Synthesis of Substituted Lophinyl (2-Aryl-4,5-diphenylimidazole) Dimers* 471
- Part C: Kinetics of the Recombination Reaction* 472
- Chapter Twenty-Three** **Aryl Halides** 475
- Experiment 23.1** **Nucleophilic Aromatic Substitution** 475
- Part A: Microscale Reaction of Sodium Ethoxide with 1-Bromo-2,4-dinitrobenzene* 477
- Part B: Microscale Reaction of Sodium Ethoxide with p-Fluoronitrobenzene* 477

- Chapter Twenty-Four Phenols** 480
- Experiment 24.1 Exploring Structure-function Relationships of Phenols: Synthesis of Salicylic Acid, Aspirin, and Vanillin Derivatives** 480
- Part A: Miniscale Reactions of Vanillin* 483
- Part B: Miniscale Hydrolysis of Methyl Salicylate* 485
- Part C: Miniscale Synthesis of Acetylsalicylic Acid* 486
- Chapter Twenty-Five Carbohydrates** 489
-  **Experiment 25.1 Classification of Sugars and Identification of an Unknown Sugar** 490
- General Instructions for the Microscale Identification of an Unknown Sugar** 495
- Part A: Osazone Formation* 496
- Part B: Chemical Tests* 496
- Experiment 25.2 Esterification of Sugars: Preparation of Sucrose Octaacetate and α - and β -D-Glucopyranose Pentaacetate** 498
- Part A: Miniscale Preparation of D-Sucrose Octaacetate* 500
- Part B: Microscale Preparation of β -D-Glucopyranose Pentaacetate* 501
- Part C: Microscale Conversion of β -D-Glucopyranose Pentaacetate to the α -Anomer* 502
- Part D: Miniscale Preparation of α -D-Glucose Pentaacetate and Measurement of Optical Rotation* 503
- Chapter Twenty-Six Lipids** 507
-  **Experiment 26.1 Soap from a Spice: Isolation, Identification, and Hydrolysis of a Triglyceride** 507
- Part A: Miniscale Isolation of a Triglyceride from Nutmeg* 509
- Part B: Microscale Hydrolysis of a Triglyceride* 510
- Part C: Determination of Properties of the Soap from Nutmeg* 511
-  **Experiment 26.2 Preparation of Esters of Cholesterol and Determination of Liquid Crystal Behavior** 512
- Chapter Twenty-Seven Amino Acids and Derivatives** 518
- Experiment 27.1 Conversion of an Amino Acid to a Sunscreen: Multistep Preparation of Benzocaine or a Benzocaine Analog** 518
- Part A: Miniscale Synthesis of p-Methylacetanilide* 521
- Part B: Miniscale Synthesis of p-Acetamidobenzoic Acid* 522
- Part C: Miniscale Synthesis of p-Aminobenzoic Acid* 523
- Part D: Microscale Esterification of p-Aminobenzoic Acid* 525
- Part E: Determining the Effectiveness of the p-Aminobenzoic Acid Ester as a Sunscreen* 526
- Chapter Twenty-Eight Qualitative Organic Analysis II** 529
-  **Experiment 28.1 Designing a Classification Scheme for Characterizing an Organic Compound** 529
- Part A: Microscale Solubility Tests* 530
- Part B: Microscale Chemical Tests* 531

	<i>Part C: Microscale Classification of an Unknown Compound</i>	536
Experiment 28.2	Experimental Methods of Qualitative Organic Analysis	537
	Introduction to Qualitative Organic Analysis	537
	Overall Approach to Identifying the Unknown	538
	Step 1: Purify	538
	Step 2: Determine the Physical Properties	538
	Step 3: Examine the Physical State	538
	Step 4: Perform Solubility Tests	538
	Step 5: Identify the Functional Groups Present	540
	Step 6: Select an Appropriate Derivative	551
	Step 7: Prepare and Purify the Derivative	552
	Step 8: Identify the Unknown	557
	Experimental Procedure	557
	<i>Part A: Purification of the Unknown</i>	559
	<i>Part B: Solubility Tests</i>	559
	<i>Part C: Chemical Tests (Arranged Alphabetically)</i>	560
	<i>Part D: Derivatives</i>	566
Part E	Qualitative Organic Analysis of Unknowns	574
	<i>Part E.1: Qualitative Analysis of Aldehydes and Ketones</i>	574
	<i>Part E.2: Qualitative Analysis of Alcohols and Phenols</i>	575
	<i>Part E.3: Qualitative Analysis of Amines and Carboxylic Acids</i>	575
	<i>Part E.4: Qualitative Analysis of a General Unknown</i>	575
	<i>Part E.5: Qualitative Analysis and Spectroscopic Analysis of a General Unknown</i>	575
Chapter Twenty-Nine	Projects	578
Experiment 29.1	Multistep Synthesis of 1-Bromo-3-chloro-5-iodobenzene from Aniline	579
	<i>Part A: Synthesis of Acetanilide from Aniline</i>	583
	<i>Part B: Miniscale Synthesis of 4-Bromoacetanilide from Acetanilide</i>	585
	<i>Part C: Synthesis of 4-Bromo-2-chloroacetanilide from 4-Bromoacetanilide</i>	586
	<i>Part D: Synthesis of 4-Bromo-2-chloroaniline from 4-Bromo-2-chloroacetanilide</i>	587
	<i>Part E: Miniscale Synthesis of 4-Bromo-2-chloro-6-iodoaniline from 4-Bromo-2-chloroaniline</i>	589
	<i>Part F: Synthesis of 1-Bromo-3-chloro-5-iodobenzene from 4-Bromo-2-chloro-6-iodoaniline</i>	590
Experiment 29.2	Multistep Synthesis of Sulfanilamide Derivatives as Growth Inhibitors	592
	<i>Part A: Preparation of Acetanilide</i>	594
	<i>Part B: Preparation of p-Acetamidobenzenesulfonyl Chloride</i>	595

	<i>Part C: Reaction of p-Acetamidobenzenesulfonyl Chloride with an Amine</i>	595
	<i>Part D: Hydrolysis of the Acetamido Group</i>	596
	<i>Part E: Bacterial Testing of Antibiotic Susceptibility</i>	596
	<i>Part A: Miniscale Synthesis of Acetanilide</i>	597
	<i>Part B: Miniscale Synthesis of p-Acetamidobenzenesulfonyl Chloride</i>	597
	<i>Part C: Miniscale Synthesis of Sulfonamides</i>	598
	<i>Part D: Miniscale Synthesis of Sulfanilamides</i>	599
	<i>Part E: Bacterial Testing of Antibiotic Susceptibility</i>	600
Experiment 29.3	Structural Determination of Isomers Using Decoupling and Special NMR Techniques	602
	<i>Part A: Homonuclear Decoupling of a Known Alkene and Determination of Coupling Constants</i>	612
	<i>Part B: Characterization of an Unknown Compound Using Homonuclear Decoupling in ^1H NMR</i>	612
	<i>Part C: Characterization of an Unknown Compound Using ^1H NMR, ^{13}C NMR, ^1H-^1H (COSY) and HETCOR (^1H-^{13}C COSY)</i>	613
Experiment 29.4	The Library-Laboratory Connection	614
	<i>Part A: Chemical Abstracts (CA) and Beilstein</i>	614
	<i>Part B: Searching Science Library Databases</i>	619
	<i>Part C: Searching on the Web</i>	620
 Experiment 29.5	Stereochemistry, Molecular Modeling, and Conformational Analysis	621
	<i>Part A: Molecular Models Using a Model Kit</i>	624
	<i>Part B: Computer Modeling</i>	625
	<i>Part C: Conformational Analysis of Rotational Isomers</i>	625
	<i>Part D: Using Molecular Modeling to Analyze Rotational Conformations of Stereoisomers</i>	625
Appendix A	Tables of Derivatives for Qualitative Organic Analysis	627
Appendix B	Laboratory Skills and Calculations	632
Appendix C	Designing a Flow Scheme	637
Appendix D	Material Safety Data Sheet	639
Appendix E	Tables of Common Organic Solvents and Inorganic Solutions	643
	<i>Index</i>	645