

*4th  
edition*



# MICROSCALE ORGANIC LABORATORY

WITH MULTISTEP AND  
MULTISCALE SYNTHESSES

Suranaree University of Technology



31051000516685

*Mayo/Dike/Trumper*

# Contents

## CHAPTER 1

### INTRODUCTION 1

- General Rules for the Microscale Laboratory, 3
- The Organic Chemistry Laboratory, 4

## CHAPTER 2

### SAFETY 5

- Making the Laboratory a Safer Place, 5
  - Nature of Hazards, 5
  - Reduction of Risks, 6
  - Precautionary Measures, 7
- Thinking about the Risks in Using Chemicals, 7
  - Disposal of Chemicals, 8
  - Material Safety Data Sheets, 8
  - Alternate Sources of Information, 12
  - Estimating the Risks from Vapors, 12
  - Concluding Thoughts, 13

## CHAPTER 3

### INTRODUCTION TO MICROSCALE ORGANIC LABORATORY EQUIPMENT AND TECHNIQUES 16

- Microglassware Equipment, 17
  - Standard Taper Joints, 17
  - Conical Vials, 18
  - Condensers, 18
  - Distillation Heads, 18
  - Recrystallization Tubes, 18
  - Miscellaneous Items, 18
  - Gas Chromatographic Fraction Collection Items, 19
- Standard Experimental Apparatus, 19
  - Heating and Stirring Arrangements, 19
  - Sand Bath Technique/Hotplate Calibration, 19
  - Metal Heat-Transfer Devices, 20
  - Stirring, 20

- Reflux Apparatus, 20
- Distillation Apparatus, 22
- Moisture-Protected Reaction Apparatus, 23
- Specialized Pieces of Equipment, 24
- Microscale Laws, 25
  - Rules of the Trade for Handling Organic Materials at the Microscale Level, 25
  - Rules for Working with Liquids at the Microscale Level, 26
  - Rules for Working with Solids at the Microscale Level, 29
- The Laboratory Notebook, 29
- Example of a Laboratory Notebook Entry, 30
- Calculation of Yields, 32

## CHAPTER 4

### DETERMINATION OF PHYSICAL PROPERTIES 33

#### LIQUIDS 34

- Ultramicro-Boiling Point, 34
- Density, 36
- Refractive Index, 38

#### SOLIDS 40

- Melting Points, 40
- Simple Capillary Melting Point, 40
- Evacuated Melting Points, 40
- Mixture Melting Points, 42

## CHAPTER 5

### MICROSCALE LABORATORY TECHNIQUES 44

#### TECHNIQUE 1 Microscale Separation of Liquid Mixtures by Preparative Gas Chromatography 44

- GC Instrumentation, 45
  - Procedure for Preparative Collection, 49

## **xii** CONTENTS

<b>TECHNIQUES 2 and 3 Distillation</b>	<b>50</b>	Removal of Solvent Under Reduced Pressure, 87
Distillation, 50		
<b>TECHNIQUE 2 Simple Distillation at the Semimicroscale Level</b>	<b>50</b>	<b>TECHNIQUE 7 Collection and Control of Gaseous Products</b> <b>90</b>
		Water Insoluble Gases, 90
		Trapping By-product Gases, 91
<b>TECHNIQUE 3 Fractional Semimicroscale Distillation</b>	<b>53</b>	<b>TECHNIQUE 8 Measurement of Specific Rotation</b> <b>92</b>
		Theory, 92
		The Polarimeter, 93
<b>TECHNIQUE 4 Solvent Extraction</b>	<b>55</b>	<b>High Performance Polarimeters and Optical Rotary Dispersion, 95</b>
Solubility, 55		<b>Applications to Structure Determination in Natural Products, 95</b>
Partition Coefficient, 58		Inaccurate Measurements, 95
Extraction, 60		<b>TECHNIQUE 9 Sublimation</b> <b>96</b>
Liquid-Liquid Extraction, 60		Sublimation Theory, 96
<b>Microscale Extraction, 61</b>		<b>Experimental Set-up, 97</b>
<b>Separatory Funnel—Semimicroscale, Macroscale Extraction, 63</b>		<b>Precautions, 97</b>
Continuous Liquid-Liquid Extraction, 64		
Separation of Acids and Bases, 65		
Salting Out, 66		
<b>Solid-Liquid Extraction, 67</b>		
<b>Drying Agents, 67</b>		
Solid-Phase Extraction, 69		
<b>TECHNIQUE 5 Crystallization</b>	<b>71</b>	<b>CHAPTER 6</b>
General Crystallization Procedure, 71		<b>MICROSCALE ORGANIC LABORATORY EXPERIMENTS</b> <b>99</b>
Simple Crystallization, 73		Introduction, 99
Filtration Techniques, 74		<b>EXPERIMENT [1] Measurement of Physical Properties</b> <b>100</b>
Use of the Hirsch Funnel, 74		
<b>A Hirsch Funnel Alternative—Nail-filter Funnel, 75</b>		<b>EXPERIMENT [2] The Separation of a 25-<math>\mu</math>L Mixture of Heptanol (bp 153 °C and Cyclohexanol (bp 160 °C) by Gas Chromatography</b> <b>106</b>
Craig Tube Crystallizations, 75		
<b>TECHNIQUE 6 Chromatography</b>	<b>77</b>	<b>EXPERIMENT [3] Distillation</b> <b>112</b>
Technique 6A Column, Flash, High-Performance Liquid, Paper, and Thin-Layer Chromatography, 77		Experiment [3A] Simple Distillation at the Semimicroscale Level: Separation of Ethyl Acetate from <i>trans</i> -1,2-Dibenzoyl ethylene, 112
Column Chromatography, 78		Experiment [3B] Fractional Semimicroscale Distillation: Separation of Hexane and Toluene, 115
Packing the Column, 79		Experiment [3C] Fractional Semimicroscale Distillation: Separation of 2-Methylpentane and Cyclohexane Using a Spinning Band Column, 117
Sample Application, 79		Experiment [3D] Fractional Semimicroscale Distillation: The Separation of 2-Methylpentane and Cyclohexane Using a Spinning Band in a Hickman-Linkle Still, 120
Elution of the Column, 80		
Fraction Collection, 80		<b>EXPERIMENT [4] Solvent Extraction</b> <b>123</b>
Flash Chromatography, 81		Experiment [4A] Determination of a Partition
Thin-Layer Chromatography, 82		
Paper Chromatography, 85		
High-Performance Liquid Chromatography, 85		
Technique 6B Concentration of Solutions, 86		
Distillation, 86		
Evaporation with Nitrogen Gas, 87		

Coefficient for the System Benzoic Acid, Methylene Chloride, and Water, 123		of a Natural Product: Caffeine and Caffeine 5-Nitrosalicylate, 204	
Experiment [4B] Solvent Extraction I: The System; Benzoic Acid, Methylene Chloride, and 10% Sodium Bicarbonate Solution; An Example of Acid-Base Extraction Techniques, 128		Experiment [11C] Isolation of a Natural Product by Steam Distillation: Cinnamaldehyde from Cinnamon 213	
Experiment [4C] Solvent Extraction II: A Three Component Mixture; An Example of the Separation of an Acid, a Base, and a Neutral Substance, 129		<b>EXPERIMENT [12] Reductive Catalytic Hydrogenation of an Alkene: Octane</b>	<b>218</b>
<b>EXPERIMENT [5] Reduction of Ketones Using a Metal Hydride Reagent: Cyclohexanol and <i>cis</i>- and <i>trans</i>-4-<i>tert</i>-Butylcyclohexanol</b>	<b>133</b>	<b>EXPERIMENT [13] Hydroboration-Oxidation of an Alkene: Octanol</b>	<b>223</b>
Experiment [5A] Cyclohexanol, 135		<b>EXPERIMENT [14] Diels-Alder Reaction: 4-Cyclohexene-<i>cis</i>-1,2-dicarboxylic Acid Anhydride</b>	<b>230</b>
Experiment [5B] <i>cis</i> - and <i>trans</i> -4- <i>tert</i> -Butylcyclohexanol, 139		<i>Optional Semimicroscale Preparation</i>	
<b>EXPERIMENT [6] Photochemical Isomerization of an Alkene: <i>cis</i>-1,2-Dibenzoylethylene</b>	<b>145</b>	<b>EXPERIMENT [15] Diels-Alder Reaction: 9,10-Dihydroanthracene-9,10-<math>\alpha,\beta</math>-succinic Acid Anhydride</b>	<b>241</b>
Experiment [6A] Purification of <i>trans</i> -1,2-Dibenzoylethylene, 148		<i>Optional Semimicroscale and Macroscale Preparations</i>	
Experiment [6B] Isomerization of an Alkene: Thin-Layer Chromatographic Analysis, 149		<b>EXPERIMENT [16] Grignard Reaction with a Ketone: Triphenylmethanol</b>	<b>246</b>
Experiment [6C] Isomerization of an Alkene: Nuclear Magnetic Resonance Analysis, 154		<b>EXPERIMENT [17] Grignard Reaction with an Aldehyde: 4-Methyl-3-heptanol</b>	<b>254</b>
<b>EXPERIMENT [7] The Cannizzaro Reaction with 4-Chlorobenzaldehyde: 4-Chlorobenzoic Acid and 4-Chlorobenzyl Alcohol</b>	<b>155</b>	<b>EXPERIMENT [18] The Perkin Reaction: Condensation of Rhodanine with an Aromatic Aldehyde to Yield <i>o</i>-Chlorobenzylidene Rhodanine</b>	<b>259</b>
<b>EXPERIMENT [8] The Esterification Reaction: Ethyl Laurate, Isopentyl Acetate, and the Use of Acidic Resins</b>	<b>165</b>	<i>Optional Semimicroscale Preparation</i>	
Experiment [8A] Ethyl Laurate, 176		<b>EXPERIMENT [19] Alkene Preparation by the Wittig Reaction: (<i>E</i>)-Stilbene; 1-Methylene-4-<i>tert</i>-butylcyclohexane; and <i>trans</i>-9-(2-Phenylethenyl)anthracene</b>	<b>264</b>
Experiment [8B] Isopentyl Acetate, 178		Experiment [19A] ( <i>E</i> )-Stilbene by the "Instant Ylide" Method, 269	
<i>Semimicroscale Preparation</i>		Experiment [19B] ( <i>E</i> )-Stilbene by the Horner-Wadsworth-Emmons Reaction, 271	
Experiment [8C] Esterification by Acidic Resins, 179		Experiment [19C] Methylene-4- <i>tert</i> -butylcyclohexane, 273	
<i>Semimicroscale Preparations</i>		Experiment [19D] <i>trans</i> -9-(2-Phenylethenyl)anthracene, 275	
<b>EXPERIMENT [9] The E1 Elimination Reaction: Dehydration of 2-Butanol to Yield 1-Butene, <i>trans</i>-2-Butene, and <i>cis</i>-2-Butene</b>	<b>184</b>	<b>EXPERIMENT [20] Aldol Reaction: Dibenzalacetone</b>	<b>279</b>
<b>EXPERIMENT [10] The E2 Elimination Reaction: Dehydrohalogenation of 2-Bromobutane to Yield 1-Butene, <i>trans</i>-2-Butene, and <i>cis</i>-2-Butene</b>	<b>192</b>	<i>Optional Semimicroscale Preparation</i>	
<b>EXPERIMENT [11] The Isolation of Natural Products</b>	<b>199</b>	<b>EXPERIMENT [21] Quantitative Analysis of Grignard Reagents: 1-Methylbutylmagnesium Bromide and Phenylmagnesium Bromide</b>	<b>286</b>
Experiment [11A] Isolation and Characterization of an Optically Active Natural Product: Usnic Acid, 199		Part 1 1-Methylbutylmagnesium Bromide, 288	
Experiment [11B] Isolation and Characterization		Part 2 Phenylmagnesium Bromide, 288	
		<b>EXPERIMENT [22] Williamson Synthesis of Ethers: Propyl <i>p</i>-Tolyl Ether and Methyl <i>p</i>-Ethylphenyl Ether</b>	<b>290</b>

## XIV CONTENTS

Experiment [22A] Propyl <i>p</i> -Tolyl Ether, 291 <i>Optional Macroscale Preparation</i>		Experiment [33A] 9-Fluorenone: CrO <sub>3</sub> Oxidation of 9-Fluorenone, 357	
Experiment [22B] Methyl <i>p</i> -Ethylphenyl Ether, 295 <i>Optional Semimicroscale and Macroscale Preparations</i>		Experiment [33B] 9-Fluorenone: NaOCl Oxidation of 9-Fluorenone, 359	
<b>EXPERIMENT [23] Amide Synthesis: Acetanilide and <i>N,N'</i>-Diacetyl- 1,4-phenylenediamine</b>	<b>302</b>	<b>EXPERIMENT [34] Hypochlorite Oxidation of Methyl Ketones by the Haloform Reaction: Benzoic Acid and <i>p</i>-Methoxybenzoic Acid</b>	<b>362</b>
Experiment [23A] Acetanilide, 304 <i>Optional Semimicroscale Preparation</i>		Experiment [34A] Benzoic Acid, 363	
Experiment [23B] <i>N,N'</i> -Diacetyl- 1,4-phenylenediamine, 306		Experiment [34B] <i>p</i> -Methoxybenzoic Acid, 365 <i>Optional Semimicroscale Preparation</i>	
<b>EXPERIMENT [24] Imide Synthesis: <i>N</i>-Phenylmaleimide</b>	<b>309</b>	<b>EXPERIMENT [35] Conversion of Cyclohexyl Bromide to Cyclohexene—an E2 Elimination Reaction: Factors Affecting the Rate of a Chemical Reaction</b>	<b>367</b>
Experiment [24A] Maleanilic Acid, 311			
Experiment [24B] <i>N</i> -Phenylmaleimide, 312			
<b>EXPERIMENT [25] Synthesis of Cyclic Carboxylic Acid Anhydrides: Succinic Anhydride and Phthalic Anhydride</b>	<b>314</b>	<b>CHAPTER 7</b>	
Experiment [25A] Succinic Anhydride, 316		<b>ADVANCED MICROSCALE ORGANIC LABORATORY EXPERIMENTS</b>	<b>379</b>
Experiment [25B] Phthalic Anhydride, 317		Introduction, 379	
<b>EXPERIMENT [26] Diazonium Coupling Reaction: Methyl Red</b>	<b>319</b>	<b>EXPERIMENT [1<sub>adv</sub>] Diborane Reductions: Thioxanthene and Xanthene</b>	<b>380</b>
<b>EXPERIMENT [27] Friedel-Crafts Acylation: Acetylferrocene and Diacetylferrocene</b>	<b>324</b>	Experiment [1A <sub>adv</sub> ] Thioxanthene, 382	
<b>EXPERIMENT [28] Halogenation: Electrophilic Aromatic Substitution to Yield 4-Bromoacetanilide</b>	<b>330</b>	Experiment [1B <sub>adv</sub> ] Xanthene, 384	
<b>EXPERIMENT [29] Nitration: 2,5- Dichloronitrobenzene; <i>N,N'</i>-Diacetyl-2,3- dinitro-1,4-phenylenediamine; 5-Nitrosalicylic Acid; and 2- and 4-Nitrophenol</b>	<b>335</b>	<b>EXPERIMENT [2<sub>adv</sub>] Heterocyclic Ring Synthesis: Benzimidazole</b>	<b>388</b>
<i>Semimicroscale Preparation of Anhydrous Nitric Acid</i>		<b>EXPERIMENT [3<sub>adv</sub>] Heterocyclic Ring Synthesis: 4-Hydroxycoumarin and Dicoumarol</b>	<b>391</b>
Experiment [29A] 2,5-Dichloronitro- benzene, 338		Experiment [3A <sub>adv</sub> ] 4-Hydroxycoumarin, 394	
Experiment [29B] <i>N,N'</i> -Diacetyl-2,3-dinitro- 1,4-phenylenediamine, 339		Experiment [3B <sub>adv</sub> ] Dicoumarol, 395	
Experiment [29C] 5-Nitrosalicylic Acid, 340		<b>EXPERIMENT [4<sub>adv</sub>] Grignard and Aryl Halide Cross-Coupling Reaction: 1-Methyl-2- (methyl-<i>d</i><sub>3</sub>)-benzene</b>	<b>397</b>
Experiment [29D] 2- and 4-Nitrophenol, 342		<b>EXPERIMENT [5<sub>adv</sub>] Oxidative Coupling of 2-Naphthol: 1,1'-Bi-2-Naphthol</b>	<b>403</b>
<b>EXPERIMENT [30] Nucleophilic Aromatic Substitution: 2,4-Dinitrophenylthiocyanate</b>	<b>345</b>	<b>EXPERIMENT [6<sub>adv</sub>] Beckmann Rearrangement: Benzanilide</b>	<b>408</b>
<b>EXPERIMENT [31] Halogenation Using <i>N</i>-Bromosuccinimide: 9-Bromoanthracene</b>	<b>349</b>	<b>EXPERIMENT [7<sub>adv</sub>] Preparation of an Enol Acetate: Cholesta-3,5-dien-3-ol Acetate</b>	<b>414</b>
<b>EXPERIMENT [32] Hypochlorite Oxidation of an Alcohol: Cyclohexanone</b>	<b>353</b>	<b>CHAPTER 8</b>	
<b>EXPERIMENT [33] Chromium Trioxide—Resin or Hypochlorite Oxidation of an Alcohol: 9-Fluorenone</b>	<b>356</b>	<b>SEQUENTIAL SYNTHESSES: THE TRANSITION FROM MACRO TO MICRO</b>	<b>419</b>
		Introduction, 419	
		<b>SEQUENCE A The Synthesis of Hexaphenylbenzene</b>	<b>421</b>
		Experiment [A1 <sub>a</sub> ] The Benzoin Condensation of Benzaldehyde: Benzoin, 426	

<i>Semimicroscale Preparation and Optional Microscale Preparations</i>		
Experiment [A2 <sub>a</sub> ] Copper(II) Ion Oxidation of Benzoin: Benzil, 431		
<i>Semimicroscale Preparation and Optional Microscale Preparation</i>		
Experiment [A3 <sub>a</sub> ]		
Tetraphenylcyclopentadienone, 435		
<i>Optional Microscale Preparation</i>		
Experiment [A1 <sub>b</sub> ] (E)-Stilbene, 439		
<i>Semimicroscale Preparation</i>		
Experiment [A2 <sub>b</sub> ] Bromination of (E)-Stilbene: <i>meso</i> -Stilbene Dibromide, 441		
<i>Semimicroscale Preparation and Optional Macroscale and Microscale Preparations</i>		
Experiment [A3 <sub>b</sub> ] Dehydrohalogenation of <i>meso</i> -Stilbene Dibromide:		
Diphenylacetylene, 446		
<i>Semimicroscale Preparation and Optional Macroscale and Microscale Preparations</i>		
Experiment [A4 <sub>ab</sub> ] Hexaphenylbenzene, 449		
<b>SEQUENCE B The Stepwise Synthesis of Nylon-6,6</b>	<b>453</b>	
Experiment [B1] Oxidation of Cyclohexanol: Adipic Acid, 454		
<i>Macroscale Preparation</i>		
Experiment [B2] Preparation of an Acid Chloride: Adipoyl Chloride, 457		
<i>Semimicroscale Preparation</i>		
Experiment [B3] Preparation of a Polyamide: Nylon-6,6, 460		
<b>SEQUENCE C The Synthesis of Sulfanilamide</b>	<b>461</b>	
Experiment [C1] Acetylation of Aniline: 2,2,2-Trifluoroacetanilide, 463		
<i>Semimicroscale Preparation</i>		
Experiment [C2] Chlorosulfonation of 2,2,2-Trifluoroacetanilide: <i>p</i> -(Trifluoroacetamido) benzenesulfonyl Chloride, 466		
<i>Semimicroscale Preparation</i>		
Experiment [C3] Preparation of an Arene Sulfonamide: Sulfanilamide, 468		
<i>Semimicroscale Preparation</i>		
<b>SEQUENCE D The Synthesis of 2'-Bromostyrene</b>	<b>471</b>	
Experiment [D1] The Verley-Doebner Modification of the Knoevenagel Reaction: <i>trans</i> -Cinnamic Acid, 474		
<i>Semimicroscale Preparation</i>		
Experiment [D2] Bromination of <i>trans</i> -Cinnamic Acid: <i>erythro</i> -2,3-Dibromo-3-phenylpropanoic Acid, 478		
<i>Semimicroscale Preparation</i>		
Experiment [D3] An Elimination Reaction with <i>erythro</i> -2,3-Dibromo-3-phenylpropanoic Acid: 2'-Bromostyrene, 482		
<i>Semimicroscale Preparation</i>		
<b>SEQUENCE E The Synthesis of Piperonylnitrile from Piperonyl Alcohol</b>	<b>488</b>	
Experiment [E1] Piperonal, 489		
<i>Macroscale Preparation and Optional Microscale Preparations</i>		
Experiment [E2] Piperonal O-(2,4-Dinitrophenyl)-oxime, 495		
<i>Semimicroscale Preparation and Optional Microscale Preparation</i>		
Experiment [E3] Piperonylnitrile, 499		
<b>SEQUENCE F Introduction of Photochromism: The Synthesis of a Photochromic Imine</b>	<b>503</b>	
Experiment [F1] An Aldol Reaction: <i>trans</i> -4-Nitrochalcone, 507		
<i>Semimicroscale Preparation</i>		
Experiment [F2] <i>erythro</i> -2,3-Dibromo-3-(4-nitrophenyl)propiophenone, 512		
<i>Semimicroscale Preparation</i>		
Experiment [F3] <i>trans</i> -2-(4-Nitrophenyl)-3-benzoylaziridine, 517		
<i>Semimicroscale Preparation</i>		
Experiment [F4] A Photochromic Imine: 2- <i>exo</i> -6- <i>exo</i> -2,4-Diphenyl-6-(4-nitrophenyl)-1,3-diazabicyclo[3.1.0]hex-3-ene, 525		
<b>CHAPTER 9</b>		
<b>SPECTROSCOPIC IDENTIFICATION OF ORGANIC COMPOUNDS</b>		<b>532</b>
INTRODUCTION TO INFRARED SPECTROSCOPY, 532		
INTRODUCTION TO GROUP FREQUENCIES, 533		
Interpretation of Infrared Spectra, 533		
STRATEGIES FOR INTERPRETING INFRARED SPECTRA, 534		
A SURVEY OF GROUP FREQUENCIES IDENTIFIED IN ORGANIC MOLECULES, 536		
<i>Group Frequencies of the Hydrocarbons are Listed as Follows:</i>		
Alkanes, 536		
Alkenes C=C Stretching, 536		
Alkene C—H, 536		
Alkynes, 538		
Arenes, 538		
<i>Group Frequencies of Carbonyl Groups</i>		
C=O, 538		
The major factors perturbing carbonyl frequencies, 539		

<i>Group frequencies of the heteroatom functional groups</i>	
Alcohols, 539	
Aldehydes, 540	
Ketones, 540	
Esters, 540	
Acyl Halides, 540	
Carboxylic Acids, 541	
Anhydrides, 541	
Ethers, 541	
Primary Amines, 542	
Nitriles, 542	
Primary amides, 542	
Secondary amides, 542	
Isocyanates, 543	
Thiols, 543	
Alkyl halides, 543	
Aryl halides (Chlorobenzene), 544	
<b>INFRARED SPECTROSCOPY: Instrumentation and Sample Handling, 544</b>	
Instrumentation, 544	
Sample Handling in the Infrared, 545	
Liquid Samples, 545	
Solution Spectra and Spectra of Materials Boiling Below 100, °C, 545	
Solid Samples, 545	
<b>NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY: Introduction to Nuclear Magnetic Resonance, 554</b>	
Nuclear Spin, 554	
Instrumentation, 555	
Chemical Shift, 557	
Spin-Spin Coupling, 558	
Intensities, 561	
Second-Order Effects, 561	
<b>INTERPRETATION OF <sup>1</sup>H NMR SPECTRA, 563</b>	
<sup>1</sup> H Chemical Shifts, 565	
Spin-Spin Coupling, 566	
Geminal Coupling, 566	
Vicinal Coupling, 567	
Long-Range Coupling, 568	
Examples of Complex, Yet First-Order, Coupling, 568	
Ethyl Vinyl Ether, 568	
Allyl Acetate, 570	
<b><sup>13</sup>C NMR SPECTROSCOPY, 573</b>	
<b>TWO-DIMENSIONAL NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY, 579</b>	
<b>NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY SAMPLING, 581</b>	
<b>ULTRAVIOLET-VISIBLE SPECTROSCOPY: Introduction to Absorption Spectroscopy, 585</b>	
UV-Vis Spectroscopy, 586	
Applications to Organic Molecules, 588	
Instrumentation, 595	
Sample Preparation, 598	
<b>MASS SPECTROMETRY, 600</b>	
<b>Instrumentation, 602</b>	
Ion Source, 602	
Mass Analyzer, 603	
Detector, 605	
Tuning the Mass Spectrometer, 605	
Sample Introduction, 605	
Gas Chromatography/Mass Spectrometry, 606	
Capillary Columns, 606	
Split Injection, 608	
Split/Splitless Injection, 608	
<b>Features of the Mass Spectrum, 608</b>	
Terms, 609	
Isotope Peaks, 609	
Recognizing the Molecular Ion, 611	
Mass Spectral Interpretation, 612	
<b>Case Study: Synthesis of Methyl Benzoate, 613</b>	
<hr/> <b>CHAPTER 10</b>	
<hr/> <b>QUALITATIVE IDENTIFICATION OF ORGANIC COMPOUNDS</b>	<b>618</b>
<b>ORGANIC QUALITATIVE ANALYSIS, 618</b>	
Preliminary Tests, 619	
Separation of Impurities, 622	
Detection of Elements Other Than Carbon, Hydrogen, and Oxygen, 622	
Solubility Characteristics, 626	
The Classification Tests, 628	
<b>PREPARATION OF DERIVATIVES, 641</b>	
Carboxylic Acids, 642	
Alcohols, 643	
Aldehydes and Ketones, 644	
Amines, 645	
Acid Chlorides and Anhydrides, 646	
Aromatic Hydrocarbons, 647	
Nitriles, 647	
Phenols, 647	
Aliphatic Hydrocarbons, Halogenated Hydrocarbons, Amides, Nitro Compounds, Ethers, and Esters, 648	
<b>GLOSSARY</b>	<b>652</b>
<b>INDEX</b>	<b>655</b>