with *Modern Projects and Experiments in Organic Chemistry*  
*Miniscale and Standard Taper Microscale, 2nd edition*

**Note to users of the 2nd edition of Modern Projects and Experiments in Organic Chemistry regarding a change in Pasteur pipet nomenclature in the 4th edition of Laboratory Techniques in Organic Chemistry:** We have introduced a second type of the Pasteur pipet filtration method. A “Pasteur filter pipet,” as described in the 2nd edition, is called a “Pasteur filter-tip pipet” in the 4th edition (see page 60); and the term “Pasteur filter pipet” is now used for a microscale gravity filtration apparatus, with the cotton packed at the top of the tip instead of the bottom (see Section 9.3).

We have also replaced Technique with Chapter in the 4th edition of *Laboratory Techniques in Organic Chemistry*.

**Experiment 1: Extraction of Caffeine from Tea**

**Miniscale procedure:**

Miniscale Vacuum Filtration Apparatus: Section 9.4, Figure 9.6  
Miniscale Extraction: Sections 10.4–10.5  
Drying Organic Liquids and Recovering Reaction Products: Chapter 11  
Boiling Stones: Section 6.1

**Microscale procedure:**

Microscale Vacuum Filtration Apparatus: Section 9.4, Figures 9.8 and 9.9  
Microscale Extraction: Sections 10.6, 10.6a, and 10.6c  
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9  
Pasteur Filter Pipet (“microfunnel” in the procedure): Section 9.3, Figures 9.3 and 9.4. Alternatively, filtration and drying can be done simultaneously by Section 11.2, Method 2, Figure 11.4a.

Drying Organic Liquids and Recovering Reaction Products: Chapter 11

**Experiment 2: Purification and Thin-Layer Chromatographic Analysis of Caffeine**

Sand Bath for Heating: Section 6.2, Figure 6.4  
Sublimation: Chapter 16  
Thin-Layer Chromatography: Chapter 18  
Preparation of micropipets for TLC: Section 18.2
Preparation of TLC plate: Sections 18.1–18.3

Analysis of TLC plate: Section 18.5; Figure 18.7

Iodine visualization of TLC plate: Section 18.4 (Note: Method differs from TOC, 2nd ed.)

**Experiment 3: Synthesis of Ethanol by Fermentation of Sucrose**

**Miniscale Procedure**

Boiling Points: Section 12.1

Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7

Miniscale Fractional Distillation: Section 12.4; apparatus: Figure 12.17

Azeotropic Distillation: Section 12.5

**Experiment 4: Synthesis of Salicylic Acid**

**Miniscale Procedure**

Miniscale Reflux: Section 7.1; apparatus: Figure 7.1a

Boiling Stones: Section 6.1

Heating Methods: Section 6.2

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

Melting Points and Melting Ranges: Chapter 14; procedure: Section 14.3

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); Interpreting IR Spectra: Section 21.7–21.9

**Microscale Procedure**

Microscale Reflux: Section 7.1; apparatus: Figure 7.1b

Boiling Stones: Section 6.1

Heating Methods: Section 6.2

Microscale Vacuum Filtration Apparatus: Section 9.4; filtration apparatus: Figures 9.8 and 9.9

Melting Points and Melting Ranges: Chapter 14; procedure: Section 14.3

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); Interpreting IR Spectra: Sections 21.7–21.9
Experiment 5: Analgesics and Synthesis of Aspirin

Miniscale Procedure
Mixed Solvent Recrystallization: Chapter 15.4
Recrystallization: Sections 15.3 and 15.5; apparatus for vacuum filtration: Figure 15.4
Fluted Filter Paper: Section 9.1
Melting Points and Melting Ranges: Chapter 14; procedure: Section 14.3
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); Interpreting IR Spectra: Sections 21.7–21.9
Thin-Layer Chromatography: Chapter 18

Microscale Procedure
Heating Methods: Section 6.2
Mixed Solvent Recrystallization: Section 15.4
Recrystallization: Sections 15.3 and 15.6–15.7; Figures 15.5 and 15.6
Melting Points and Melting Ranges: Chapter 14; procedure: Section 14.3
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); Interpreting IR Spectra: Sections 21.7–21.9
Thin-Layer Chromatography: Chapter 18

Experiment 6: Isolation of Essential Oils from Plants
6.1 (S)-(−)-Carvone from Caraway Seeds

Miniscale Procedure
Miniscale Steam Distillation: Section 12.6, Figure 12.21
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
How to Select a Recrystallization Solvent: Section 15.4
Miniscale Recrystallization: Sections 15.1, 15.3, and 15.4
Melting Points and Melting Ranges: Chapter 14; procedure: Sections 14.3 and 14.5
Gas Chromatography: Chapter 20
Polarimetry: Sections 17.1–17.3
6.2 Isolation of \((R)\)-(+)\)-Limonene from Orange Peels

Miniscale Procedure

Miniscale Steam Distillation: Section 12.6, Figure 12.21
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Polarimetry: Sections 17.1–17.3

Experiment 7: Computational Chemistry

Chapter 8 in the 4th edition of *Laboratory Techniques in Organic Chemistry* has an expanded set of questions.

Experiment 8: Radical Halogenation Reactions

8.1 Radical Chlorination Reactions

Microscale Procedure

Microscale Extraction: Sections 10.6, 10.6a–10.6b
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

8.2 Photobromination of 1,2-Diphenylethane

Microscale Procedure

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.18
Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

Experiment 9: Nucleophilic Substitution Reactions

9.1 \(S_N1/ S_N2\) Reactivity of alkyl Halides

No techniques called out.

9.2 Synthesis and Identification of Alkyl Bromides from Unknown Alcohols

Microscale Procedure

Miniscale Reflux: Section 7.1, Figure 7.1a
Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7
Microscale Extractions: Sections 10.6, 10.6a, and 10.6c
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Miniscale Short-Path Distillation: Section 12.3b; apparatus: Figure 12.8
NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Experiment 10: E2 Elimination of 2-Bromoheptane: Influence of the Base

Microscale Procedure
Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4b
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Microscale Extraction: Sections 10.6a–10.6b
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

Experiment 11: Dehydration of Alcohols

Miniscale Procedure

11.1 Acid-Catalyzed Dehydration of 2-Methyl-2-butanol
Miniscale Fractional Distillation: Section 12.4; apparatus: Figure 12.17
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

11.2 Acid-Catalyzed Dehydration of 2-Methylocyclohexanol

Microscale Procedure
Microscale Distillation: Section 12.3c; apparatus: Figure 12.10
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

Experiment 12: Synthesis of Esters from Alcohols

12.1 Synthesis of Isopentyl Acetate

Miniscale Procedure
Miniscale Reflux: Section 7.1: apparatus: Figure 7.1a
Miniscale Extraction: Sections 10.4–10.5

Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7

Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Microscale Procedure**

Microscale Reflux: Section 7.1; apparatus: Figure 7.1b

Microscale Extraction: Sections 10.6, 10.6a–10.6b

Microscale Distillation: Section 12.3c; apparatus: Figure 12.10 or Figure 12.12

Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Optional Experiment**

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Microscale Boiling Point Determination: Section 12.1; apparatus: Figure 12.2

**12.2 Synthesis of Benzocaine**

**Microscale Procedure**

Recrystallization from Mixed Solvent Pairs: Section 15.4

Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

Miniscale Extraction: Sections 10.4–10.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Microscale Procedure**

Recrystallization from Mixed Solvent Pairs: Section 15.4

Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9
12.3 Determination of the Equilibrium Constant for an Esterification Reaction

Microscale Procedure

Microscale Extraction: Sections 10.6, 10.6a–10.6b
Pasteur Filter-tip Pipets: Section 5.3, Figure 5.9
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

Experiment 13: Green Chemistry: Oxidation of Cyclohexanol Using Sodium Hypochlorite

Miniscale Procedure

Miniscale Steam Distillation: Section 12.6, Figure 12.21
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Microscale Procedure

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Experiment 14: Oxidation of Cinnamyl Alcohol using Pyridinium Chlorochromate

Microscale Procedure

Thin-Layer Chromatography: Chapter 18; visualization: Section 18.4
Liquid Chromatography: Chapter 19; column preparation/procedure: Sections 19.2–19.4
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Experiment 15: Grignard Syntheses

15.1 Synthesis of Phenylmagnesium Bromide for Use as a Synthetic Intermediate

Miniscale Procedure
Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4a and Figure 7.2a or 7.2b

**Microscale Procedure**

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4b

### 15.2 Synthesis of 1.1-Diphenylethanol

**Miniscale Procedure**

Miniscale Extraction: Sections 10.4–10.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Microscale Procedure**

Microscale Extraction: Sections 10.6, 10.6a–10.6b

Microscale Gravity Filtration: Section 9.3; Pasteur filter pipet: Figures 9.3 and 9.4

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

### 15.3 Synthesis of Benzoic Acid

**Microscale Procedure**

Microscale Extraction: Sections 10.6, 10.6a–10.6b

Microscale Vacuum Filtration Apparatus: Section 9.4, Figures 9.8 and 9.9

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

### Experiment 16: Addition Reactions of Alkenes

#### 16.1 Free-Radical Versus ionic Addition of Hydrobromic Acid

**Microscale Procedure**

Microscale Extraction: Sections 10.6, 10.6a–10.6b

Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

#### 16.2 Stereochemistry of Bromine Addition to *trans*-Cinnamic Acid

**Miniscale Procedure**
Miniscale Reflux: Section 7.1; apparatus: Figure 7.1a

Mixed Solvent Recrystallization: Section 15.4

**Microscale Procedure**

Microscale Reflux: Chapter 7.1; apparatus: Figure 7.1b

Mixed Solvent Recrystallization: Section 15.4

Microscale Vacuum Filtration Apparatus: Section 9.4, Figures 9.8 and 9.9

**Experiment 17: Synthesis of Polystyrene**

**Miniscale Procedure**

Liquid Chromatography: Chapter 19; column preparation/procedure: Sections 19.2–19.4

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Experiment 18: Phase Transfer Catalysis**

**Miniscale Procedure**

Miniscale Reflux: Section 7.1; apparatus: Figure 7.1a

Miniscale Extraction: Sections 10.4–10.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Microscale Procedure**

Microscale Reflux: Section 7.1; apparatus: Figure 7.1b

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Experiment 19: Diels-Alder Reaction**

**Miniscale Procedure**

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.16

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Microscale Procedure**

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.18

Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Experiment 20: Selectivity in the Bromination of Acetanilide and 4-Methylacetanlidge**

**Microscale Procedure**

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.18

Mixed Solvent Recrystallization: Section 15.4

Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Experiment 21: Acylation of Ferrocene**

**Microscale Procedure**

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4b

Thin-Layer Chromatography: Chapter 18

Liquid Chromatography: Section 19.5a, Figure 19.7

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Experiment 22: Akylation of 1,4-Dimethoxybenzene**

**Microscale Procedure**
Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

Microscale Extraction: Sections 10.6, 10.6a–10.6b

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

**Experiment 23: Kinetic versus Equilibrium Control in the Alkylation of Chlorobenzene**

**Microscale Procedure**

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c

Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

**Experiment 24: Reduction Reactions of 3-Nitroacetophenone**

**24.1 Reduction of 3-Nitroacetophenone Using Sodium Borohydride**

**Microscale Procedure**

Microscale Gravity Filtration: Section 9.3; Pasteur filter pipet: Sections 9.3 and 9.4

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**24.2 Reduction of 3-Nitroacetophenone Using Tin and Hydrochloric Acid**

**Microscale Procedure**

Microscale Vacuum Filtration Apparatus: Section 9.4, Figures 9.8 and 9.9

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Experiment 25: Horner-Emmons-Wittig Synthesis of Methyl-E-4-Methoxycinnamate**

**Miniscale Procedure**

Mixed Solvent Recrystallization: Section 15.4

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

**Microscale Procedure**
Mixed Solvent Recrystallization: Section 15.4

Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Experiment 26: Synthesis of Triphenylmethane Dyes: Crystal Violet and Malachite Green

Miniscale Procedure

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4a and Figure 7.2a or 7.2b

Experiment 27: Enolate Chemistry: Synthesis of trans-1,2-Dibenzoylcyclopropane

Miniscale Procedure

Miniscale Reaction Apparatus: Section 7.2; Figure 7.2a

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Microscale Procedure

Microscale Reaction Apparatus: Sections 7.2, Figure 7.3a

Microscale Recrystallization: Sections 15.3 and 15.6; filtration apparatus: Figure 15.5

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Experiment 28: Amide Chemistry

28.1 Synthesis of Benzoylamino Acids from Glycine, Alanine, and Valine

Miniscale and Microscale Procedures

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

28.2 Synthesis of a Mosquito Repellant: N,N-Diethyl-meta-Toluamide (DEET)
Miniscale Procedure

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.16

Miniscale Extraction: Sections 10.4–10.5

Liquid Chromatography: Chapter 19; column preparation/procedure: Sections 19.2–19.4

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Microscale Procedure

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4b

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.18

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c

Flash Chromatography: Chapter 19; column preparation/procedure: Sections 19.2–19.4

Thin-Layer Chromatography: Chapter 18

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Project 1: Identification of a White Solid: A Team Approach

Microscale Procedure

Melting Points and Melting Ranges: Chapter 14; procedure: Sections 14.3 and 14.5

Thin-Layer Chromatography: Chapter 18

Optional Experiment

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Project 2: Using Extraction to Separate a Mixture

2.1 Separation and Purification of the Compounds in the Unknown Mixture: First Week
Miniscale Procedure

Miniscale Extraction: Sections 10.4–10.5

Drying Organic Liquids and Recovering Reaction Products: Chapter 11

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

Miniscale Vacuum Filtration Apparatus: Section 9.4, Figure 9.6

2.2 Melting Points, Purity, and Identification of the Compounds in the Unknown Mixture: Second Week

Melting Points and Melting Ranges: Chapter 14; procedure: Sections 14.3 and 14.5

Thin-Layer Chromatography: Chapter 18

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Project 3: Hydrolysis of an Unknown Ester

3.1 Hydrolysis and Azeotropic Distillation: First Week

Miniscale Procedure

Boiling Stones: Section 6.1

Miniscale Reflux: Section 7.1; apparatus: Section 7.1a

Azeotropic Distillation: Section 12.5

Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7

3.2 Recrystallization and Extraction: Second Week

Miniscale Procedure

Miniscale Extraction: Sections 10.4–10.5

Drying Organic Liquids and Recovering Reaction Products: Chapter 11

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

Miniscale Vacuum Filtration Apparatus: Section 9.4; apparatus: Figure 9.6

3.3 Distillation, Boiling Points, and Melting Points: Third Week

Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7

Melting Points and Melting Ranges: Chapter 14; procedure: Section 14.3

3.4 Identification of the Unknown Ester: Third Week
Refractometry: Section 13.3

Project 4: Interconversion of 4-tert-Butylcyclohexanol and 4-tert-Butylcyclohexanone

4.1 Green Chemistry: Sodium Hypochlorite Oxidation of 4-tert-Butylcyclohexanol

Miniscale Procedure
Thin-Layer Chromatography: Chapter 18
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

4.2 Sodium Borohydride Reduction of 4-tert-Butylcyclohexanone

Miniscale Procedure
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Thin-Layer Chromatography: Chapter 18
Gas Chromatography: Chapter 19; analysis of chromatogram: Sections 19.7 and 19.8
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9
NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Microscale Procedure
Microscale Extraction: Sections 10.6, 10.6a–10.6b
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Thin-Layer Chromatography: Chapter 18
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9
NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9
Project 5: Synthesis of 1-Bromobutane and the Grignard Synthesis of an Alcohol

5.1 Synthesis of 1-Bromobutane from 1-Butanol

Miniscale Procedure

Miniscale Reflux: Section 7.1; apparatus: Figure 7.1a
Miniscale Steam Distillation: Section 12.6, Figure 12.21
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7
NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

5.2 Grignard Synthesis of Secondary and Tertiary Alcohols

Miniscale Procedure

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4a and Figure 7.2a or 7.2b
Miniscale Reaction Apparatus: Section 7.3; Figure 7.4a
Miniscale Extraction: Sections 10.4–10.5
Drying Organic Liquids and Recovering Reaction Products: Chapter 11
Miniscale Simple Distillation: Section 12.3a; apparatus: Figure 12.7
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8
IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

Project 6: E1/E2 Elimination Reactions

6.1 Acid-Catalyzed Dehydration of 2-Methyl-2-Butanol

Miniscale Procedure

Miniscale Fractional Distillation: Section 12.4; apparatus: Figure 12.17
Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

6.2 Synthesis of 2-Chloro-2-methyl butane

Miniscale Procedure
Miniscale Extraction: Sections 10.4–10.5

Miniscale Short-Path Distillation: Section 12.3b; apparatus: Figure 12.8

6.3 Base-Catalyzed Dehydrochlorination of 2-Chloro-2-methylbutane

**Miniscale Procedure**

Miniscale Fractional Distillation: Section 12.4; apparatus: Figure 12.17

Gas Chromatography: Chapter 20; analysis of chromatogram: Sections 20.4 and 20.7–20.8

**Project 7: Synthesis and Hydroboration-Oxidation of 1-Phenylcyclohexene**

7.1 Grignard Synthesis of 1-Phenylcyclohexene

**Miniscale Procedure**

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4a and Figure 7.2a or 7.2b

Recovery of an Organic Product: Section 11.4

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

7.2 Acid-Catalyzed Dehydration of 1-Phenylcyclohexanol

**Miniscale Procedure**

Miniscale Extraction: Sections 10.4–10.5

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

7.3 Hydroboration-Oxidation of 1-Phenylcyclohexanol

**Microscale Procedure**

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

7.4 Computational Chemistry Experiment

Computational Chemistry: Chapter 8

Spin-Spin Coupling (Splitting): Section 22.9
Project 8: Electrophilic Aromatic Substitution

8.1 Synthesis of Diacetylferrocene

Microscale Procedure

Anhydrous Reaction Conditions: Section 7.3; apparatus: Figure 7.4b

Microscale Extraction: Sections 10.6, 10.6a, and 10.6c

Thin-Layer Chromatography: Chapter 18

8.2 Purification by Column Chromatography

Microscale Procedure

Preparation and Elution of a Microscale Column: Section 19.5a; apparatus: Figure 19.7

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

8.3 Optional Column Chromatography for Isolation of the Minor Diacetylferrocene Isomer

Miniscale Procedure

Liquid Chromatography: Chapter 19; column preparation/procedure: Sections 19.2–19.4, Figure 19.2 or 19.4

Thin-Layer Chromatography: Chapter 18

Project 9: Stereochemistry of Electrophilic Addition to an Alkene

9.1 Synthesis of Cyclohex-4-ene-cis-1,2-dicarboxylic Acid

Miniscale Procedure

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.16

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

9.2 Bromination of Cyclohex-4-ene-cis-1,2-dicarboxylic Acid

Miniscale Procedure

Miniscale Vacuum Filtration Apparatus: Section 9.4, Figure 9.6

9.3 Synthesis of Dimethyl 4,5-Dibromocyclohexane-cis-1,2-dicarboxylate

Miniscale Procedure
Miniscale Extraction: Sections 10.4–10.5

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

Project 10: Diels-Alder Cycloadditions of Dienophiles to Dimethyl-1,3-Butadiene

10.1 Competition Reactions of 2,3-Dimethyl-1,3-butadiene and 1,3-Cyclohexadiene with Maleic Acid

Microscale Procedure

Gas Chromatography: Chapter 20; determining chromatographic conditions: Section 20.5; analysis of chromatogram: Sections 20.4, 20.7, and 20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

10.2 Competition Reactions of 2,3-Dimethyl-1,3-butadiene with Diethyl-trans-2-Butenedioate and Diethyl-cis-2-Butenedioate

Gas Chromatography: Chapter 20; determining chromatographic conditions: Section 20.5; analysis of chromatogram: Sections 20.4, 20.7, and 20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

10.3 Competition Reactions of 2,3-Dimethyl-1,3-butadiene with Dimethyl-cis-2-Butenedioate and Dimethyl 2-Butynedioate

Gas Chromatography: Chapter 20; determining chromatographic conditions: Section 20.5; analysis of chromatogram: Sections 20.4, 20.7, and 20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

10.4 Designing and Carrying Out a Synthesis of Dimethyl 4,5-Dimethylbenzene-1,2-dicarboxylate by Diels-Alder and Dehydrogenation Chemistry

Gas Chromatography: Chapter 20; determining chromatographic conditions: Section 20.5; analysis of chromatogram: Sections 20.4, 20.7, and 20.8

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); interpreting IR spectra: Sections 21.7–21.9
Project 11: Aldol-Dehydration Chemistry Using Unknown Aldehydes and Ketones

11.1 Preparation and Characterization of the Aldol-Dehydration Product

Miniscale Procedure

How to Select a Recrystallization Solvent: Section 15.4

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Figure 15.4

Project 12: Biochemical Catalysis and the Stereochemistry of Borohydride Reduction

12.1 Thiamine-Catalyzed Benzoin Condensation

Miniscale Procedure

Miniscale Recrystallization: Sections 15.3 and 15.5; filtration apparatus: Section 15.4

Thin-Layer Chromatography: Chapter 18

IR Spectroscopy: Chapter 21; sample preparation: Section 21.5 (transmittance IR), Section 21.6 (ATR); Interpreting IR Spectra: Sections 21.7–21.9

12.2 Stereoselectivity of the Borohydride Reduction of Benzoin

Miniscale Procedure

Thin-Layer Chromatography: Chapter 18

Mixed Solvent Recrystallization: Section 15.4

Miniscale Extraction: Sections 10.4–10.5

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9
Project 13: Designing a Multistep Synthesis

Students need to submit a list of reagents and additional equipment needed for their syntheses.

Project 14: Sugars: Glucose Pentaacetates

14.1 Synthesis of $\alpha$-D-Glucose Pentaacetate and $\beta$-D-Glucose Pentaacetate

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

14.2 Investigation of Kinetic and Equilibrium Control in the Glucose Pentaacetate System

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

14.3 Computational Chemistry Experiment

Project 15: Enantioselective Synthesis by Enzymatic Hydrolysis Using Pig-Liver Esterase

15.1 Synthesis of Dimethyl Cyclohex-4-ene-cis-1,2-dicarboxylate by Diels-Alder Cycloaddition and Esterification

Miniscale Procedure

Removal of Noxious Vapors: Section 7.6; apparatus: Figure 7.16

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9

15.2 Synthesis of ($\pm$)-Methyl Hydrogen Cyclohex-4-ene-cis-1,2-dicarboxylate

Miniscale Procedure

NMR Spectroscopy: Chapter 22; sample preparation: Sections 22.2; interpreting spectra: Sections 22.4–22.9

15.3 Enantioselective Enzymatic Hydrolysis of Dimethyl Cyclohex-4-ene-cis-1,2-dicarboxylate

Miniscale Procedure

NMR Spectroscopy: Chapter 22; sample preparation: Section 22.2; interpreting spectra: Sections 22.4–22.9