

contents overview

- 1** Introduction, Laboratory Safety, and Laboratory Notebook 1
- 2** Techniques and Apparatus 18
- 3** Solids: Recrystallization and Melting Points 65
- 4** Liquids: Distillation and Boiling Points 92
- 5** Extraction 126
- 6** Chromatography 138
- 7** Stereoisomers 162
- 8** Introduction to Spectroscopy; Infrared Spectroscopy 188
- 9** Nuclear Magnetic Resonance Spectroscopy 234
- 10** Spectroscopy: Structure Determination using IR and NMR 287
- 11** Ultraviolet and Visible Spectroscopy and Mass Spectrometry 305
- 12** Alkanes 320
- 13** Alkenes: Preparations and Reactions 336
- 14** Alkynes 376
- 15** Dienes: The Diels-Alder Reaction 384
- 16** Kinetic and Equilibrium Control of a Reaction 406
- 17** Electrophilic Aromatic Substitution 417
- 18** Nucleophilic Aliphatic Substitution: Preparation of Haloalkanes and Chemical Kinetics 441
- 19** Oxidation Reactions of Alcohols, Carbonyl Compounds, and Arenes 463
- 20** Reduction Reactions of Double Bonds: Alkenes, Carbonyl Compounds, and Imines 482
- 21** Reactions of Carbonyl Compounds 500

22	Organometallic Chemistry	545
23	Multistep Organic Syntheses	572
24	Polymers	619
25	Carbohydrates	637
26	Amino Acids and Peptides	656
27	Identification of Organic Compounds	676
28	The Literature of Organic Chemistry	764

contents

PREFACE iii

LIST OF IR, NMR, AND UV SPECTRA xiv

LIST OF EXPERIMENTAL PROCEDURES xix

1 Introduction, Laboratory Safety, and Laboratory Notebook 1

- 1.1 Introduction 1
- 1.2 Laboratory Safety 3
- 1.3 The Laboratory Notebook 6

2 Techniques and Apparatus 18

- 2.1 Glassware: Precautions and Handling 18
- 2.2 Standard-Taper Glassware 19
- 2.3 Melting-Point Methods and Apparatus 20
- 2.4 Boiling-Point Apparatus 25
- 2.5 Heating Methods 26
- 2.6 Stirring Methods 33
- 2.7 Simple Distillation 36
- 2.8 Fractional Distillation 38
- 2.9 Vacuum Distillation 39
- 2.10 Steam Distillation 43
- 2.11 Fluted Filter Paper 46
- 2.12 Gravity Filtration 46
- 2.13 Hot Gravity Filtration 47

- 2.14 Decolorizing Carbon 48
- 2.15 Decanting Solutions 49
- 2.16 Vacuum Filtration 50
- 2.17 Sublimation 57
- 2.18 Separatory Funnels and Their Use 53
- 2.19 Heating Under **Reflux** 57
- 2.20 Gas Traps 59
- 2.21 Drying Agents, Desiccants, and Drying Liquids 59
- 2.22 Drying Solids 63
- 2.23 Drying Tubes 63
- 2.24 Drying of Apparatus 64

3 Solids: Recrystallization and Melting Points 65

- 3.1 Recrystallization 66
- 3.2 Physical Constants: Melting Points 78

4 Liquids: Distillation and Boiling Points 92

- 4.1 Boiling Points of Pure Liquids 92
- 4.2 Purification of Liquids by Distillation: A Survey 96
- 4.3 Simple Distillation 97
- 4.4 Fractional Distillation of Ideal Solutions 98
- 4.5 Fractional Distillation of Nonideal Solutions 112
- 4.6 Vacuum Distillation; Distillation Under Reduced Pressure 115
- 4.7 Steam Distillation 115

5 Extraction 126

- 5.1 Theory of Extraction 126
- 5.2 Continuous Extraction 128
- 5.3 Extraction of Natural Products 130
- 5.4 Acid and Base Extractions 133

6 Chromatography 138

- 6.1 Column Chromatography 139
- 6.2 Thin-Layer Chromatography 144
- 6.3 Paper Chromatography 149
- 6.4 Ion-Exchange Chromatography 150
- 6.5 High-Pressure Liquid Chromatography 150
- 6.6 Gas Chromatography 152

7 Stereoisomers 162

- 7.1 Introduction 162
- 7.2 Separation of the Diastereomeric *cis,trans* Isomers of 1,2-Cyclohexanediol 165
- 7.3 Isomerization of **Dimethyl Maleate** to **Dimethyl Fumarate** 168
- 7.4 Polarimetry 173

- 7.5 Isolation of the Enantiomers of **Carvone** from Essential Oils 175
- 7.6 Resolution of Racemic 1-Phenylethylamine 184

8 Introduction to Spectroscopy; Infrared Spectroscopy 188

- 8.1 Overview of Spectral Methods 189
- 8.2 Theory of Infrared Spectroscopy 191
- 8.3 The Infrared Spectrophotometer 194
- 8.4 Infrared Spectroscopy of Liquids, Solids, and Gases 196
- 8.5 Experimental Procedures for Spectrum Determination 201
- 8.6 Uses of Infrared Spectroscopy: Identification of Functional Groups and Compounds 208
- 8.7 Interpretation of Infrared Spectra 215

9 Nuclear Magnetic Resonance Spectroscopy 234

- 9.1 The Concept of Nuclear Magnetic Resonance Spectroscopy; the NMR Spectrometer 235
- 9.2 Introduction to PMR Spectra 238
- 9.3 Chemical Shift and Structure 241
- 9.4 Spin-Spin Splitting Patterns and Coupling Constants 248
- 9.5 Hydrogen Counting and Peak Areas 253
- 9.6 Procedure for Analyzing PMR Spectra 254
- 9.7 Examples of Structure Identification Using PMR 255
- 9.8 Examples of PMR Spectra of Compounds Containing Common Functional Groups 260
- 9.9 Carbon-13 Magnetic Resonance (CMR) Spectroscopy 278
- 9.10 Preparation of NMR Samples 282

10 Spectroscopy: Structure Determination Using IR and NMR 287

- 10.1 Analysis of IR and PMR Spectra of a Known Compound 288
- 10.2 Analysis of IR and CMR Spectra of a Known Compound 290
- 10.3 Analysis of IR and PMR Spectra of Unknown Compounds 290
- 10.4 Analysis of IR and CMR Spectra of an Unknown Compound 296

11 Ultraviolet and Visible Spectroscopy and Mass Spectrometry 305

- 11.1 Ultraviolet and Visible Spectroscopy 305
- 11.2 Mass Spectrometry 314

12 Alkanes 320

- 12.1 Chlorination by Means of Sulfuryl Chloride 320
- 12.2 Bromination: Relative Ease of Substitution of Hydrogen in Different Environments 330

13 Alkenes: Preparations and Reactions 336

13.1 Dehydrohalogenation of Haloalkanes 337

13.2 Dehydration of Alcohols 348

13.3 Addition Reactions of Alkenes 364

14 Alkynes 376

15 Dienes: The Diels-Alder Reaction 384

16 Kinetic and Equilibrium Control 406

17 Electrophilic Aromatic Substitution 417

17.1 Introduction 417

17.2 Friedel-Crafts Alkylation of p-Xylene with 1-Bromopropane 418

17.3 Nitration of Bromobenzene 429

17.4 Relative Rates of Electrophilic Aromatic Substitution 434

18 Nucleophilic Aliphatic Substitution | Preparation of Haloalkanes and Chemical Kinetics 441

18.1 Theory of Nucleophilic Aliphatic Substitution; Competition with Elimination 441

18.2 Preparation of 1-Bromobutane: An S_N2 Reaction 444

18.3 Preparation of Tertiary Chloroalkanes: An S_N1 Reaction 450

18.4 Chemical Kinetics: Evidence for Nucleophilic Substitution Mechanisms 454

19 Oxidation Reactions of Alcohols, Carbonyl Compounds, and Arenes 463

19.1 Introduction 463

19.2 Preparation of Aldehydes and Ketones by Oxidation of Alcohols 465

19.3 Base-Catalyzed Oxidation-Reduction of Aldehydes: The Cannizzaro Reaction 475

19.4 Carboxylic Acids 479

20 Reduction Reactions of Double Bonds | Alkenes, Carbonyl Compounds, and Imines 482

20.1 Introduction 482

20.2 Catalytic Hydrogenation of the Carbon-Carbon Double Bond: Hydrogenation of 4-Cyclohexene-cis-1,2-dicarboxylic Acid 484

20.3 Reduction of Imines; Preparation of Amines 489

20.4 Reduction of Carbonyl Compounds; Preparation of Alcohols 495

21 Reactions of Carbonyl Compounds 500

21.1 Reactions Involving Nucleophilic Addition to the Carbonyl Group 500

21.2 Reactions of Stabilized Carbanions Derived from Carbonyl Compounds 511

21.3 Conjugate Addition to an α,β -Unsaturated Ketone 534

22 Organometallic Chemistry 545

- 22.1 Introduction 545
- 22.2 The **Grignard** Reagent: Its Preparation and Reactions 546
- 22.3 The Organolithium Reagent 563
- 22.4 Organocopper Reagents 565

23 Multistep Organic Syntheses 572

- 23.1 Introduction 572
- 23.2 The Synthesis of Sulfanilamide 574
- 23.3 4-Aminoquinolines 590
- 23.4 The Synthesis of Lidocaine 604
- 23.5 Additional Multistep Synthetic Sequences 617

24 Polymers 619

- 24.1 Chain-Reaction Polymerizations 619
- 24.2 Step-Growth Polymerization 626
- 24.3 Polyurethanes 632

25 Carbohydrates 637

- 25.1 Introduction 637
- 25.2 Mutarotation of Glucose 639
- 25.3 The Hydrolysis of Sucrose 645
- 25.4 Isolation of α,α -Trehalose 647
- 25.5 Carbohydrates: Their Characterization and Identification 650

26 Amino Acids and Peptides 656

- 26.1 Introduction 656
- 26.2 Analysis of Amino Acids 658
- 26.3 Determination of Primary Structure of Polypeptides 668

27 Identification of Organic Compounds 676

- 27.1 Overview of Organic Analysis 676
- 27.2 Classical Qualitative Analysis Procedure for Identification of a Pure Compound 678
- 27.3 Separation of Mixtures of Organic Compounds 689
- 27.4 Applications of Spectroscopic Methods to Qualitative Organic Analysis 692
- 27.5 Qualitative Classification Tests and Preparation of Derivatives 699
- 27.6 Tables of Compounds and Derivatives 729

28 The Literature of Organic Chemistry 764

- 28.1 Classification of the Literature of Organic Chemistry 764
- 28.2 Use of the Literature of Organic Chemistry in an Introductory Organic Course 771