Preface to the Third Edition ix Preface to the First Edition xi

Introduction 1

Safety 1

Laboratory Procedures 7 Research Notebook 11

Standard References in Inorganic Chemistry 12

Part I. SOLID-STATE CHEMISTRY

EXPERIMENT 1. The 1-2-3 Superconductor YBa₂Cu₃O₇ 17

> Tube Furnace Preparation 19 Superconductivity 20

Iodometric Titrations 21

2. The Layered Solids VO(PO₄)(H₂O)₂ and VO(HPO₄)(H₂O)_{0.5} 27 **EXPERIMENT**

Oxidation of Alcohols to Ketones 29

Powder X-ray Diffraction 30

3. The Molecular Sieve Zeolite-X 37 **EXPERIMENT**

Ion Exchange in Microporous Solids 38

High-Pressure Synthesis 40

Part II. MAIN GROUP CHEMISTRY

4. The Borane–Amine Adduct BH₃:NH₂C(CH₃)₃ 47 **EXPERIMENT** Lewis Acids and Bases 48

EXPERIMENT 5. Buckminsterfullerene (C₆₀) and Its Electrochemistry

> Soxhlet Extraction 57 Cyclic Voltammetry 58

6. Vacuum Line Synthesis of GeH₄ 65 **EXPERIMENT**

Vacuum Line and Trap-to-Trap Purification 65

Gas-Phase Infrared Spectroscopy 68

7. Tin Chemistry: Coordination Complexes and Organometallic Derivatives 75 **EXPERIMENT**

Linkage Isomerism 75 Nuclear Magnetic Resonance Spectroscopy 77

8. Synthesis of $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2$ in Liquid Ammonia 85 **EXPERIMENT**

Metal-Ammonia Solutions 85

EXPERIMENT 9. Electrolytic Synthesis of K₂S₂O₈ 93

Electrolytic Preparation 93

Part III. COORDINATION CHEMISTRY

- EXPERIMENT 10. Ion Exchange Separation of Chromium Complexes 103

 Ion Exchange Chromatography 103

 Ligand Exchange Reactions 105
- EXPERIMENT 11. Metal-Metal Quadruple Bonds 111

 Molecular Orbital Theory 112
- EXPERIMENT 12. The Paramagnetic Complex Mn(acac)₃ 117

 Magnetic Susceptibility 120

 The Gouy Method 123

 The Evans Method 125
- EXPERIMENT 13. Preparation and Aquation of Co(NH₃)₅Cl²⁺ 131

 Electrical Conductance of Solutions of Ionic Compounds 133

 Ligand Exchange Kinetics 134
- EXPERIMENT 14. Optical Resolution of Co(en)₃³⁺ 143
 Optical Rotations 144
 Resolution of Optical Isomers 146
- EXPERIMENT 15. Metal Dithiolenes and the Use of Quaternary Ammonium Salts 151

 Quaternary Ammonium Salts 152

Part IV. ORGANOMETALLIC CHEMISTRY

- EXPERIMENT 16. The Metal—Arene Complex [1,3,5-C₆H₃(CH₃)₃]Mo(CO)₃ 161 Infrared Spectra of Metal Carbonyls 163
- EXPERIMENT 17. Organoiron Chemistry: $(C_5H_5)_2Fe_2(CO)_4$ and $(C_5H_5)Fe(CO)_2CH_3$ 171

 Schlenk Line Techniques 172

 Infrared Spectra of Solutions 172
- EXPERIMENT 18. The Metal Carbonyl Cluster Fe₃(CO)₁₂ 181

 Mass Spectrometry 183
- EXPERIMENT 19. Microscale Synthesis of Vaska's Complex IrCl(CO)[P(C₆H₅)₃]₂ 189
 Oxidative Addition Reactions 190
 Infrared Spectra of Solids 191
- EXPERIMENT 20. The Air-Sensitive Sandwich Complex Nickelocene 197

 Diels-Alder Additions 199

 Sublimation 202

 Glovebag Manipulations 203

Part V. BIOINORGANIC CHEMISTRY

- EXPERIMENT 21. Cobaloximes: Models of Vitamin B₁₂ 211 Enzyme Models 211
- EXPERIMENT 22. Amino Acid Complexes: Stability Constants of Ni(glycinate)_n (2-n)+ 219
 Amino Acid Chemistry 219
 pH Titrations of Metal Complexes 223
- EXPERIMENT 23. Bioinorganic Coordination Chemistry: Copper(II) Tetraphenylporphyrinate 233

 Metalloporphyrin Chemistry 233

 Thin-layer and Column Chromatography 235

Notes to Instructor 245 Appendices 253

- Approximate Concentrations and Densities of Commercial, Reagent Grade Acids and Bases 253
- 2. Expected Molar Conductance ($\Lambda_{\rm M}$) Ranges for 2, 3, 4, and 5 Ion Electrolytes in Some Common Solvents 254
- Transmission Ranges of Cell Materials for Infrared and Ultraviolet-Visible Spectroscopy 255
- 4. Properties of Commercial Glasses 256
- 5. Common Solvents and Their Properties 257
- 6. Drying (Dehydrating) Agents 258
- 7. Natural Abundances and Nuclear Spins of Stable Isotopes 259
- 8. Baths for Cooling and Heating 262
- 9. SI Units 263
- 10. Selected Spectra 265

General Index 269