Contents

Preface *vii*
Acknowledgments *ix*

1 Introduction *1*
Reference *3*

2 Reaction Setup and Running *5*
2.1 General Considerations *5*
2.2 Reactions at Room Temperature *6*
2.3 Reactions at Lowered Temperature *7*
2.4 Reactions at Elevated Temperature *7*
2.5 Reactions Under Exclusion of Moisture *8*
2.6 Reactions Under Exclusion of Moisture and Air *8*
2.7 Catalytic Hydrogenation Reactions *11*
2.8 Solid-phase Reactions *18*
References *20*

3 Reaction Monitoring *23*
3.1 General Considerations *23*
3.2 Thin-layer Chromatography *24*
3.3 Liquid Chromatography–Mass Spectrometry *37*
3.4 Monitoring of Solid-Phase Reactions *42*
References *43*

4 Reaction Workup *45*
4.1 General Considerations *45*
4.2 Liquid–Liquid Washing *45*
4.3 Filtration *54*
4.4 Evaporation *59*
4.5 Distillation *65*
4.6 Freeze-drying ("Lyophilization") *72*
4.7 Crystallization/Recrystallization *75*
4.8 Column Chromatography *84*
References *91*

5 Reaction Product Characterization *93*
5.1 General Considerations *93*
5.2 NMR Spectroscopy (Sample Preparation) *94*
5.3 Mass Spectrometry (Sample Preparation) *95*
5.4 Optical Rotation *97*
5.5 Melting Point *100*
References *104*

6 Documentation *105*
6.1 General Considerations *105*
6.2 Lab Notes Handwritten on Paper *106*
6.3 Lab Notes on Electronic Media Created with Word-processing Software *106*
6.4 Lab Notes on Electronic Media Created with Specialized ELN (Electronic Lab Notes) Software *108*

7 Safety in the Laboratory *111*
7.1 Introduction *111*
7.2 Personal Protection *112*
7.3 Fixed Laboratory Safety Equipment *115*
7.4 Dangerous Properties of Reagents and Solvents *118*
References *121*

8 Other Useful Information *123*
8.1 General Considerations *123*
8.2 Vacuum Sources *123*
8.3 Heat Sources *131*
8.4 Stirring Equipment *132*
8.5 Balances *133*
8.6 Labware *134*
8.7 Storage of Chemicals *139*
8.8 Setting Up an Organic Chemistry Lab: Equipment and Analytical Instruments *143*
References *145*

9 Multistep Organic Synthesis *147*
References *148*

Index *151*