CONTENTS

Preface xi
Acknowledgments xiii

1 Introduction 1
1.1 Evolution of Gas Chromatographic Columns 1
1.2 Central Role Played by the Column 6
1.3 Justification for Column Selection and Care 8
1.4 Literature on Gas Chromatographic Columns 11
1.5 Gas Chromatographic Resources on the Internet 12
References 13

2 Packed Column Gas Chromatography 15
2.1 Introduction 15
2.2 Solid Supports and Adsorbents 15
   Supports for Gas–Liquid Chromatography 15
   Adsorbents for Gas–Solid Chromatography 22
2.3 Stationary Phases 33
   Requirements of a Stationary Phase 33
   USP Designation of Stationary Phases 36
   Kovats Retention Index 36
   McReynolds and Rohrschneider Classifications
   of Stationary Phases 41
   Evaluation of Column Operation 45
   Optimization of Packed Column Separations 53
2.4 Column Preparation 54
   Coating Methods 56
   Tubing Materials and Dimensions 56
   Glass Wool Plugs and Column Fittings 57
   Filling the Column 58
   Conditioning the Column and Column Care 59
2.5 United States Pharmacopeia and National Formulary
   Chromatographic Methods 60
References 91
3 Capillary Column Gas Chromatography

3.1 Introduction  
Significance and Impact of Capillary Gas Chromatography  94  
Chronology of Achievements in Capillary Gas Chromatography  95  
Comparison of Packed and Capillary Columns  98

3.2 Capillary Column Technology  100  
Capillary Column Materials  100  
Fused Silica and Other Glasses  100  
Extrusion of a Fused-Silica Capillary Column  103  
Aluminum-Clad Fused-Silica Capillary Columns  106  
Fused-Silica-Lined Stainless Steel Capillary Columns  106

3.3 Preparation of Fused-Silica Capillary Columns  110  
Silanol Deactivation Procedures  110  
Static Coating of Capillary Columns  116  
Capillary Cages  116  
Test Mixtures for Monitoring Column Performance  117  
Diagnostic Role Played by Components of Test Mixtures  119

3.4 Chromatographic Performance of Capillary Columns  122  
Golay Equation Versus the van Deemter Expression  122  
Choice of Carrier Gas  124  
Measurement of Linear Velocity and Flow Rate  126  
Effect of Carrier Gas Viscosity on Linear Velocity  127  
Phase Ratio  129  
Coating Efficiency  132

3.5 Stationary-Phase Selection for Capillary Gas Chromatography  132  
Requirements  132  
History  132  
Comparison of Columns from Manufacturers  134  
Polysiloxane Phases  140  
Polyethylene Glycol Phases  141  
Cross-Linked Versus Chemically Bonded Phase  142  
Chemical Bonding  149  
MS-Grade Phases Versus Polysilarylene or Polysilphenylene Phases  150  
Sol-Gel Stationary Phases  150  
Phenylpolycarborane–Siloxane Phases  151

3.6 Specialty Columns  153  
EPA Methods  153
3.7 Capillary Column Selection 159
   - Practical Considerations of Column Diameter, Film Thickness, and Column Length 159
   - Capillary Columns of 0.53 mm i.d.: Megabore Columns 165
   - Correlation of Column Dimensions and Film Thickness with Parameters in the Fundamental Resolution Equation 167
   - Column Selection for Gas Chromatography by Specifications 172

3.8 Column Installation and Care 186
   - Carrier Gas Purifiers 186
   - Ferrule Materials and Fittings 187
   - Column Installation 191
   - Column Conditioning 192
   - Column Bleed 194
   - Retention Gap and Guard Columns 196
   - Column Fatigue and Regeneration 200

3.9 Special Gas Chromatographic Techniques 200
   - Simulated Distillation 200
   - Multidimensional Gas Chromatography 201
   - Computer Modeling of Stationary Phases 203

References 204

4 Column Oven Temperature Control 210
4.1 Thermal Performance Variables and Electronic Considerations 210
4.2 Advantages of Temperature Programming over Isothermal Operation 211
4.3 Oven Temperature Profiles for Programmed-Temperature Gas Chromatography 212
4.4 Role of Computer Assistance in Optimizing Separations in Gas Chromatography 214
   - DryLab (LC Resources) 214
   - ProezGC (Restek Corporation) 215
   - GC-SOS (Chem SW) 216
4.5 Fast or High-Speed Gas Chromatography 217
   - Selectivity Tuning 219
   - Resistively Heated Columns and Column Jackets 223
4.6 Subambient Oven Temperature Control 228

References 229