

Contents

<i>Preface</i>	<i>iii</i>
Part 1. THE CHROMATOGRAPHIC PROCESS	
1. The Chromatographic Separation	3
Introduction	3
History of Chromatography	4
The Principles of a Chromatographic Separation	10
The Progress of a Solute Through a Chromatographic System	12
The Development Process in Thin Layer Chromatography	19
Chromatography Nomenclature	21
References	24
2. The Mechanism of Retention	27
The Plate Theory	27
The Thermodynamic Explanation of Retention	35
Parameters that Control the Magnitude of the Distribution Coefficient (K)	39
Molecular Forces	39
Hydrophobic and Hydrophilic Interactions	47
Molecular Forces and Chromatographic Selectivity	51
Separations Based on Dispersive Interactions	52
Separations Based on Polar Interactions	55
Separations Based on Ionic Interactions	58

Parameters that Control the Chromatographically Available Stationary Phase (V_s)	59
The Effect of Stationary Phase Loading on the Performance of a Chromatographic System	60
Stationary Phase Limitation by Chiral Selectivity	62
Stationary Phase Limitation by Exclusion	64
References	66
3. Peak Dispersion	69
The Peak Width	69
Alternative Forms of Presenting Chromatographic Data	72
The Rate Theory	74
The Van Deemter Equation	75
Carrier Gas Compressibility: Its Effect on the Interpretation of Chromatographic Data	81
Effect of Mobile Phase Compressibility on the HETP Equation for a Packed GC Column	86
Mobile Phase Compressibility: Its Effect on the Interpretation of Chromatographic Data in LC	93
Effect of Mobile Phase Compressibility on the HETP Equation for a Packed LC Column	95
Extensions of the HETP Equation	96
The Golay Equation for Open Tubular Columns	99
The Efficiency of a TLC Plate	102
References	103
4. Qualitative Analysis	105
The Corrected Retention Volume	106
The Capacity Ratio of a Solute	107
The Separation Ratio	108
Solute Identification in TLC	109
Two-Dimensional TLC	111
Tandem Techniques	112
Gas Chromatography/Mass Spectrometry (GC/MS) Systems	113
Liquid Chromatography/Mass Spectrometry (LC/MS) Systems	116
Gas Chromatography/Infrared (GC/IR) Systems	125

Liquid Chromatography/Infrared (LC/IR) Systems	129
The Use of Spectroscopic Techniques with TLC	131
Scanning Densitometry	131
References	133
5. Quantitative Analysis	135
Chromatographic Resolution	136
The Efficiency Required to Achieve a Specific Resolution	137
Peak Deconvolution	140
The Quantitative Evaluation of the Chromatogram	144
Peak Area Measurements	144
Peak Height Measurements	150
Quantitative Analytical Methods for GC and LC	152
Quantitative Analysis Using Reference Standards	153
Quantitative Analysis by TLC	158
Comparative Spot Assessment by Visual Estimation	159
Scanning Densitometry for Quantitative Analysis	160
Reference	162
Part 2. GAS CHROMATOGRAPHY	
6. The Gas Chromatograph	165
The Contemporary Gas Chromatograph	168
The Gas Supplies	169
The Sampling System	169
The Column Oven	174
The GC Column	175
The Capillary, or Open Tubular, Column	179
Detection	184
Data Acquisition and Processing	185
Automatic Sample Processing for GC Analysis	186
References	189
7. Gas Chromatography Detectors	191
Detector Specifications	192
Detector Linearity and the Response Index (α)	192
Linear Dynamic Range	195
Detector Noise Level	196

Measurement of Detector Noise	197
Detector Sensitivity or the Minimum Detectable Concentration	198
Pressure Sensitivity	198
Flow Sensitivity	199
Temperature Sensitivity	199
The Flame Ionization Detector	199
The Electron Capture Detector	202
The Nitrogen Phosphorus Detector (NPD)	206
The Katherometer Detector	209
References	212
8. Gas Chromatography Columns	215
Stationary Phases	218
GSC Stationary Phases	219
GLC Stationary Phases	219
Preparation of the Column Packing	221
Column Packing	222
Coating Open Tubular Columns	223
Dynamic Coating	225
Static Coating	226
Chiral Stationary Phases	227
References	230
9. Gas Chromatography Applications	233
Gas Analysis	234
Pre-Columns	236
The Separation of the Alcohols and Metabolites Found in Blood	237
The Separation of the Free Fatty Acids from Milk	239
The Separation of the Components of a Fermentation Product of an Alcoholic Beverage	241
The Analysis of Gasoline	243
The Separation of Basic Drugs on an Open Tubular Column	245
The Separation of Neutral Drugs	247
The Use of GC to Identify Bacteria from the Analysis of Their Fatty Acids	248

Part 3. LIQUID CHROMATOGRAPHY

10. Liquid Chromatography Apparatus	253
The Gradient System	254
Liquid Chromatography Pumps	256
Sample Valves	264
Column Ovens	267
Detectors	268
Data Acquisition and Processing	269
The Modern Versatile Liquid Chromatograph	269
References	274
11. Liquid Chromatography Detectors	275
The UV Detectors	276
The Fixed Wavelength Detector	276
The Variable Wavelength Detector	282
The Diode Array Detector	284
The Fluorescence Detector	288
The Electrical Conductivity Detector	293
The Refractive Index Detector	298
References	301
12. Liquid Chromatography Columns	303
Silica Gel	303
Bonded Phases	307
Oligomeric Phases	308
Bulk Phases	309
Interactions on the Surface of Bonded Phases	310
Bi-Layer Adsorption	314
Solute-Solvent Interactions with Bonded Phases	316
Chiral Stationary Phases	317
Macro-Porous Polymers	319
Aqueous Solvent Mixtures	321
Column Packing	324
References	325
13. Liquid Chromatography Applications	327
Entropically Driven Separations	328

LC Separations by Size Exclusion	328
Chiral Separations	332
Enthalpically (Energy) Driven Separations	333
LC Separations Dominated by Dispersive Interactions	334
LC Separations Dominated by Polar Interactions	337
LC Separations Dominated by Ionic Interactions	342
Part 4. THIN LAYER CHROMATOGRAPHY	
14. Thin Layer Chromatography Apparatus	349
Thin Layer Chromatography Chambers	351
Continuous Plate Development	356
Forced-Flow Development	357
Sample Application	359
References	364
15. Thin Layer Chromatography Techniques	365
TLC Stationary Phases and Supports	365
Silica Gel	366
Reversed Phases	367
Alumina	368
Magnesia	370
Kieselguhr	370
Cellulose	371
Polyamides	372
Ion Exchange Materials	372
Coating the Thin Layer Plate	374
Choice of Phase System in TLC	376
Stationary Phase Selection	376
Selection of the Mobile Phase	381
Spot Detection in TLC	384
The Iodine Reagent	384
The Sulfuric Acid Spray	385
The Chromic-Sulfuric Acid Spray	385
Fluorescence	386
References	387
<i>Index</i>	389