

Contents

Chapter 1	Basic Concepts of High-performance Liquid Chromatography	1
1	Physical Parameters for High-speed Separations	1
2	Physical Considerations	4
3	Chemical Influences on the Separation Factor	5
4	Basic Considerations of Liquid Chromatography	6
5	Bibliography	9
Chapter 2	Instrumentation	11
1	Chromatographic Systems	11
2	Injectors	11
3	Eluent Delivery Systems	12
	Composition Gradient Delivery Systems	13
	Pressure Drop Changes due to Changes in the Eluent Composition	13
	Flow Rate Gradients	15
	Step-wise Elution	15
	Recycle Elution	15
	Column Switching Separation	17
4	Degassing Methods	17
5	Column Temperature Control	18
6	Detectors	18
	Spectroscopic Detectors	18
	Electrochemical Detectors	22
	Mass Spectrometer	22
	Other Detectors	22
	Measurement of Detector Sensitivity	23
7	Recording Systems	23
	Selection of Detector Time Constant	23
8	Columns and Connectors	25
9	Flow Cell Volume and Connecting Tube Dimensions for High Efficiency Operation	25
10	Other Components in a Liquid Chromatograph	26
11	Trouble-shooting	26

12	References	29
	Reference Books	30
Chapter 3	Preparation, Testing, and Selectivity of Stationary Phase Materials	31
1	Synthesis of Stationary Phase Materials	32
	Silica Gel	32
	Surface Modification of Silica Gel	35
	Modification Using a Halogenated Silica Surface	35
	Modification Using a Chlorosilane	35
	Synthesis of Ion-exchanger Stationary Phases	36
	Cation-exchanger	36
	Anion-exchanger	36
	Organic Stationary Phase Materials	37
2	Sieving of Stationary Phase Materials	37
3	Column Packing Methods	37
4	Column Evaluation	39
	Column Efficiency and Asymmetry	39
	Column Test for Reversed-phase Liquid Chromatography	40
	Inertness Test for Basic Compounds	40
	Inertness Test for Acidic Compounds	41
	Inertness Test for Heavy Metals	41
	Physicochemical Tests of the Stationary Phase	43
5	Measurement of Void Volume	43
	Elution Volume of Proposed Void Volume Markers	44
6	Selectivity of Stationary Phase Materials	46
	Silica Gel-based Stationary Phase Materials	46
	Organic Polymer-based Stationary Phase Materials	49
	Other Stationary Phase Materials	54
7	References	56
Chapter 4	Selection of the Eluent	57
1	Reversed-phase Liquid Chromatography	57
	Selectivity of Organic Modifiers in the Eluent	57
	Comparison of the Effect of Organic Modifiers on Analytes with $\log P = 3$	60
	Chromatographic Behaviour of $\log P = 3$ Compounds in Aqueous Tetrahydrofuran	60
	Chromatographic Behaviour of $\log P = 3$ Compounds in Aqueous Methanol	62
	Chromatographic Behaviour of $\log P = 3$ Compounds in Aqueous Acetone	62
	Chromatographic Behaviour of $\log P = 3$ Compounds in Aqueous Dimethylformamide (DMF)	62

	Comparison of Effects of Modifiers on a Mixture of Analytes	62
2	Chromatography of Ionic Compounds	65
	pH Effects	65
	Ion-pair Separations	70
	Theory of Ion-pair Liquid Chromatography	70
	Normal Phase Ion-pair Partition Liquid Chromatography	71
	Reversed-phase Ion-pair Liquid Chromatography	72
	pH Effects	75
	Effect of the Ionic Strength of the Buffer Solution	80
	Selectivity of Organic Modifier and the Concentration Effect	80
	Column Temperature Effects	80
	Detection	80
	Trouble-shooting in Ion-pair Liquid Chromatography	81
3	Normal-phase Liquid Chromatography	81
	Classification of Solvents	85
	Preparation of the Eluent	89
4	Size-exclusion Liquid Chromatography (SEC)	90
	Aqueous Phase Size-exclusion	91
	Organic Phase Size-exclusion	92
5	References	93
	Bibliography of additional references on theoretical approaches in ion-pair liquid chromatography	95
Chapter 5	Separation Based on an Improved Column Efficiency	96
1	Improving Separation by Changing the Selectivity	99
2	Improving Separation by Increasing the Column Efficiency	100
	Reducing the Eddy Diffusion Term	102
	Reducing the Effect of Longitudinal Diffusion	103
	Reducing the Effect of Resistance to Mass Transport in the Stationary and Mobile Phases	103
3	Bibliography	108
Chapter 6	Influence of Physical Chemistry on Separations in Liquid Chromatography	109
1	Quantitative Structure–Retention Relationships in Reversed-phase Liquid Chromatography	109
	Prediction of Retention Times from $\log P$ in Reversed-phase Liquid Chromatography	110
	Prediction of Partition Coefficient ($\log P$)	110
	Correlation between $\log P$ and $\log k$ Values	110
	Prediction of Retention Times from $\log P$	111

	Prediction of Retention Time of Ionic Compounds from log P and pK_a	113
	Calculation of Dissociation Constant pK_a from Hammett's Equation	113
2	Van der Waals Volume as the Basic Property	115
	Calculation of van der Waals Volume	115
	Prediction of Retention Times Based on van der Waals Volumes	115
3	Enthalpy as a Thermodynamic Property in Retention Studies	128
	Measurement of Enthalpy by Liquid Chromatography	129
	Enthalpy Related to Retention	129
4	Conclusion	131
5	References	131

Subject Index	133
----------------------	------------