

Contents

Series Preface	xiii
Preface	xv
Acknowledgements	xix
Acronyms, Abbreviations and Symbols	xxi
About the Author	xxv
1 Pre- and Post-Extraction Considerations	1
1.1 Introduction	2
1.2 Organic Compounds of Interest	2
1.3 Pre-Sampling Issues	2
1.4 Sampling Strategies: Solid, Aqueous and Air Samples	8
1.4.1 Practical Aspects of Sampling Soil and Sediment	11
1.4.2 Practical Aspects of Sampling Water	13
1.4.3 Practical Aspects of Air Sampling	15
1.5 An Introduction to Practical Chromatographic Analysis	15
1.5.1 Gas Chromatography	18
1.5.2 High Performance Liquid Chromatography	22
1.5.3 Sample Pre-Concentration Methods	29
1.6 Quality Assurance Aspects	34
1.7 Health and Safety Considerations	35
References	36

AQUEOUS SAMPLES	37
2 Classical Approaches for Aqueous Extraction	39
2.1 Introduction	39
2.2 Liquid–Liquid Extraction	39
2.2.1 Theory of Liquid–Liquid Extraction	40
2.2.2 Selection of Solvents	41
2.2.3 Solvent Extraction	42
2.2.4 Problems with the LLE Process	44
2.3 Purge and Trap for Volatile Organics in Aqueous Samples	45
References	47
3 Solid Phase Extraction	49
3.1 Introduction	49
3.2 Types of SPE Media (Sorbent)	50
3.2.1 Multimodal and Mixed-Phase Extractions	51
3.2.2 Molecularly Imprinted Polymers (MIPs)	51
3.3 SPE Formats and Apparatus	53
3.4 Method of SPE Operation	55
3.5 Solvent Selection	58
3.6 Factors Affecting SPE	59
3.7 Selected Methods of Analysis for SPE	60
3.7.1 Applications of Normal Phase SPE	60
3.7.2 Applications of Reversed Phase SPE	63
3.7.3 Applications of Ion Exchange SPE	65
3.7.4 Applications of Molecularly Imprinted Polymers (MIPs)	67
3.8 Automation and On-Line SPE	76
3.8.1 Application of Automated On-Line SPE	78
References	84
4 Solid Phase Microextraction	85
4.1 Introduction	85
4.2 Theoretical Considerations	88
4.3 Experimental	89
4.4 Methods of Analysis: SPME–GC	92
4.4.1 Direct Immersion SPME: Semi-Volatile Organic Compounds in Water	92
4.4.2 Headspace SPME: Volatile Organic Compounds (VOCs) in Water	92
4.4.3 Analysis of Compounds from Solid Matrices	94

4.4.4	Other SPME–GC Applications	101
4.5	Methods of Analysis: SPME–HPLC–MS	105
4.5.1	Analysis of Abietic Acid and Dehydroabietic Acid in Food Samples	106
4.5.2	Analysis of Fungicides in Water Samples	107
4.6	Automation of SPME	109
4.6.1	Applications of Automated SPME	110
	References	114
5	New Developments in Microextraction	117
5.1	Introduction	117
5.2	Stir-Bar Sorptive Extraction (SBSE)	118
5.3	Liquid-Phase Microextraction	118
5.3.1	Single-Drop Microextraction (SDME)	118
5.4	Membrane Microextraction	119
5.4.1	Semipermeable Membrane Device (SPMD)	120
5.4.2	Polar Organic Chemical Integrative Sampler (POCIS)	120
5.4.3	‘Chemcatcher’	120
5.4.4	Ceramic Dosimeter	120
5.4.5	Membrane Enclosed-Sorptive Coating (MESCO) Device	120
5.5	Microextraction in a Packed Syringe (MEPS)	121
	References	123
	SOLID SAMPLES	125
6	Classical Approaches for Solid–Liquid Extraction	127
6.1	Introduction	127
6.2	Soxhlet Extraction	128
6.3	Automated Soxhlet Extraction or ‘Soxtec’	130
6.4	Other Approaches for Solid–Liquid Extraction	132
	References	139
7	Pressurized Fluid Extraction	141
7.1	Introduction	141
7.2	Theoretical Considerations Relating to the Extraction Process	142
7.2.1	Solubility and Mass Transfer Effects	144
7.2.2	Disruption of Surface Equilibria	144

7.3	Instrumentation for PFE	146
7.3.1	Dionex System	146
7.3.2	Applied Separations, Inc.	149
7.3.3	Fluid Management Systems, Inc.	149
7.4	Method Development for PFE	149
7.5	Applications of PFE	152
7.5.1	Parameter Optimization	152
7.5.2	<i>In situ</i> Clean-Up or Selective PFE	156
7.5.3	Shape-Selective, Fractionated PFE	158
7.6	Comparative Studies	160
7.7	Miscellaneous	160
	References	165
8	Microwave-Assisted Extraction	167
8.1	Introduction	167
8.2	Instrumentation	171
8.2.1	Anton-Parr	173
8.2.2	CEM Corporation	173
8.2.3	Milestone	174
8.3	Applications of MAE	174
	References	183
9	Matrix Solid Phase Dispersion	185
9.1	Introduction	185
9.2	Issues on the Comparison of MSPD and SPE	187
9.3	A Review of Selected Applications	188
	References	194
10	Supercritical Fluid Extraction	197
10.1	Introduction	197
10.2	Instrumentation for SFE	200
10.3	Applications of SFE	202
10.4	Selection of SFE Operating Parameters	202
	References	207
	GASEOUS SAMPLES	209
11	Air Sampling	211
11.1	Introduction	211
11.2	Techniques Used for Air Sampling	213

<i>Contents</i>	xi
11.2.1 Whole Air Collection	213
11.2.2 Enrichment into Solid Sorbents	214
11.2.3 Desorption Techniques	216
References	219
COMPARISON OF EXTRACTION METHODS	221
12 Comparison of Extraction Methods	223
12.1 Introduction	223
12.2 Role of Certified Reference Materials	225
12.3 Comparison of Extraction Techniques for (Semi)-Solid Samples	227
12.3.1 A Comparison of Extraction Techniques for Solid Samples: a Case Study	230
12.4 Comparison of Extraction Techniques for Liquid Samples	233
12.5 Comparison of Extraction Techniques for Air Sampling	233
References	240
RESOURCES	241
13 Resources for Extraction Techniques	243
13.1 Introduction	243
13.1.1 Sources of Data	244
13.2 Role of Worldwide Web	244
Responses to Self-Assessment Questions	249
Glossary of Terms	261
SI Units and Physical Constants	269
Periodic Table	273
General Index	275
Application Index	279