

CONTENTS

PREFACE		xi
CONTRIBUTORS		xiii
CHAPTER 1 INTRODUCTION: BASIC PRINCIPLES OF ASSAYS TO BE COVERED, SAMPLE HANDLING, AND SAMPLE PROCESSING		1
	<i>Wanlong Zhou, Eugene Y. Chang, and Perry G. Wang</i>	
1.1	Introduction	1
1.1.1	Current Situation and Challenges of Food Safety and Regulations	1
1.1.2	Residues and Matrices of Food Analysis and High-Throughput Analysis	2
1.1.3	Food Safety Classifications	3
1.1.4	"High Throughput" Definition	3
1.1.5	Scope of the Book	4
1.2	Advanced Sample Preparation Techniques	5
1.2.1	Automation of Weighing and Preparing Standard Solutions	5
1.2.2	QuEChERS	6
1.2.3	Swedish Extraction Technique (SweEt) and Other Fast Sample Preparation Methods	6
1.2.4	Turbulent Flow Chromatography	7
1.2.5	Pressurized Liquid Extraction	7
1.2.6	Automated 96- and 384-Well Formatted Sample Preparation as well as Automated SPE Workstations	8
1.2.7	Solid-Phase Microextraction	8
1.2.8	Microextraction by Packed Sorbent	9
1.2.9	Liquid Extraction Surface Analysis	9
1.2.10	Headspace GC	10
1.2.11	Summary	10

1.3	Future Perspectives	10
	Acknowledgment	11
	References	11
CHAPTER 2	SURVEY OF MASS SPECTROMETRY-BASED HIGH-THROUGHPUT METHODS IN FOOD ANALYSIS	15
	<i>Lukas Vaclavik, Tomas Cajka, Wanlong Zhou, and Perry G. Wang</i>	
2.1	Introduction	15
2.2	Techniques Employing Chromatographic Separation	15
2.2.1	Gas Chromatography–Mass Spectrometry	15
2.2.2	Liquid Chromatography–Mass Spectrometry	21
2.3	Direct Techniques	30
2.3.1	Matrix-Assisted Laser Desorption/ Ionization–Mass Spectrometry	30
2.3.2	Headspace (Solid-Phase Microextraction)- Mass Spectrometry E-Nose	37
2.3.3	Ambient Desorption/Ionization–Mass Spectrometry	38
2.4	Concluding Remarks	62
	Acknowledgments	62
	References	63
CHAPTER 3	QUALITY SYSTEMS, QUALITY CONTROL GUIDELINES AND STANDARDS, METHOD VALIDATION, AND ONGOING ANALYTICAL QUALITY CONTROL	73
	<i>David Galsworthy and Stewart Reynolds</i>	
3.1	Introduction	73
3.1.1	Quality System Design	73
3.1.2	Procedures	74
3.1.3	Roles and Responsibilities	74
3.1.4	Quality Manual	74
3.1.5	Document Control	74
3.1.6	Control of Records	75
3.1.7	Audits	75
3.1.8	Validation of Methodology	75
3.1.9	Staff Competency	75

3.1.10	Internal Quality Control	76
3.1.11	Method Performance Criteria	76
3.2	Qualitative Screening Methods	76
3.2.1	Selectivity of Mass Spectrometry-Based Methods	78
3.2.2	Confirmatory Methods	78
3.2.3	Validation of Qualitative Screening Multiresidue Methods for Pesticide Residues in Foods	79
3.3	Elements of the Analytical Workflow	80
3.3.1	Sample Preparation	80
3.3.2	Effects of Sample Processing	81
3.3.3	Extraction Efficiency	81
3.4	Initial Method Validation	81
3.5	Ongoing Analytical Quality Control	86
3.5.1	Internal Quality Control	86
3.5.2	Proficiency Testing	86
3.6	Validation of Qualitative Screening Multiresidue Methods for Veterinary Drug Residues in Foods	87
3.6.1	EU Legislation Covering Method Validation for Veterinary Drug Screening	87
3.6.2	Determination of Specificity/Selectivity and Detection Capability ($CC\beta$) Using the Classical Approach	88
3.6.3	Establishment of a Cutoff Level and Calculation of $CC\beta$	88
3.6.4	Determination of the Applicability	89
3.7	Conclusions	90
	References	90

CHAPTER 4 DELIBERATE CHEMICAL CONTAMINATION AND PROCESSING CONTAMINATION 93

Stephen Lock

4.1	Introduction	93
4.2	Heat-Induced Food Processing Contaminants	97
4.3	Packaging Migrants	101
4.4	Malicious Contamination of Food	105
	References	111

CHAPTER 5	MULTIRESIDUAL DETERMINATION OF 295 PESTICIDES AND CHEMICAL POLLUTANTS IN ANIMAL FAT BY GEL PERMEATION CHROMATOGRAPHY (GPC) CLEANUP COUPLED WITH GC-MS/MS, GC-NCI-MS, AND LC-MS/MS	117
	<i>Yan-Zhong Cao, Yong-Ming Liu, Na Wang, Xin-Xin Ji, Cui-Cui Yao, Xiang Li, Li-Li Shi, Qiao-Ying Chang, Chun-Lin Fan, and Guo-Fang Pang</i>	
5.1	Introduction	117
5.1.1	Persistent Organic Pollutants	118
5.1.2	Polycyclic Aromatic Hydrocarbons	119
5.1.3	Polychlorinated Biphenyls	119
5.1.4	Phthalate Esters	120
5.1.5	Multiclass and Multiresidue Analyses	120
5.2	Experiment	122
5.2.1	Instruments	122
5.2.2	Reagents	122
5.2.3	Preparation of Standard Solutions	122
5.2.4	Sample Preparation	123
5.2.5	Analytical Methods	124
5.2.6	Qualitative and Quantitative Determination	136
5.3	Results and Discussion	136
5.3.1	Selection of GPC Cleanup Conditions	136
5.3.2	Selection of Extraction Solvent	138
5.3.3	Comparison of Sample Extraction Methods	150
5.3.4	Comparison of Sample Cleanup	151
5.3.5	Linear Range, LOD, and LOQ	152
5.3.6	Recoveries and Precisions	152
5.3.7	Actual Sample Analysis	157
5.4	Conclusions	161
	References	162
CHAPTER 6	ULTRAHIGH-PERFORMANCE LIQUID CHROMATOGRAPHY COUPLED WITH HIGH-RESOLUTION MASS SPECTROMETRY: A RELIABLE TOOL FOR ANALYSIS OF VETERINARY DRUGS IN FOOD	167
	<i>María del Mar Aguilera-Luiz, Roberto Romero-González, Patricia Plaza-Bolaños, José Luis Martínez Vidal, and Antonia Garrido Frenich</i>	
6.1	Introduction	167

6.2	Veterinary Drug Legislation	168
6.3	Analytical Techniques for VD Residue Analysis	172
6.3.1	Chromatographic Separation	174
6.3.2	High-Resolution Mass Spectrometers	175
6.4	Food Control Applications	181
6.4.1	Screening Applications	181
6.4.2	Confirmation and Quantification Methods	191
6.4.3	Comparison Studies	195
6.5	Conclusions and Future Trends	201
	Acknowledgments	202
	References	203

CHAPTER 7 A ROLE FOR HIGH-RESOLUTION MASS SPECTROMETRY IN THE HIGH-THROUGHPUT ANALYSIS AND IDENTIFICATION OF VETERINARY MEDICINAL PRODUCT RESIDUES AND OF THEIR METABOLITES IN FOODS OF ANIMAL ORIGIN 213

*Eric Verdon, Dominique Hurtaud-Pessel,
and Jagadeshwar-Reddy Thota*

7.1	Introduction	213
7.2	Issues Associated with Veterinary Drug Residues and European Regulations	215
7.3	Choosing a Strategy: Targeted or Nontargeted Analysis?	216
7.3.1	Targeted Analysis Using HRMS	218
7.3.2	Nontargeted Analysis Using HRMS: Screening for Unknown Compounds	219
7.4	Application Number 1: Identification of Brilliant Green and its Metabolites in Fish under High-Resolution Mass Spectral Conditions (Targeted and Nontargeted Approaches)	220
7.5	Application Number 2: Targeted and Nontargeted Screening Approaches for the Identification of Antimicrobial Residues in Meat	223
7.6	Conclusions	227
	References	227

CHAPTER 8 HIGH-THROUGHPUT ANALYSIS OF MYCOTOXINS 231

Marta Vaclavikova, Lukas Vaclavik, and Tomas Cajka

8.1	Introduction	231
8.1.1	Legislation and Regulatory Limits	231
8.1.2	Emerging Mycotoxins	237

8.1.3	Analysis of Mycotoxins in the High-Throughput Environment	238
8.2	Sample Preparation	239
8.2.1	Sampling	240
8.2.2	Matrices of Interest	240
8.2.3	Extraction of Mycotoxins	241
8.2.4	Purification of Sample Extracts	246
8.3	Separation and Detection of Mycotoxins	247
8.3.1	Liquid Chromatography–Mass Spectrometry-Based Methods	248
8.3.2	High-Resolution Mass Spectrometry in Mycotoxins Analysis	250
8.4	No-Separation Mass Spectrometry-Based Methods	252
8.4.1	Matrix-Assisted Laser Desorption Ionization–Mass Spectrometry	252
8.4.2	Ambient Ionization Mass Spectrometry	253
8.4.3	Ion Mobility Spectrometry	254
8.4.4	Immunochemical Methods	256
8.5	Conclusions	259
	Acknowledgments	259
	References	259
	INDEX	267