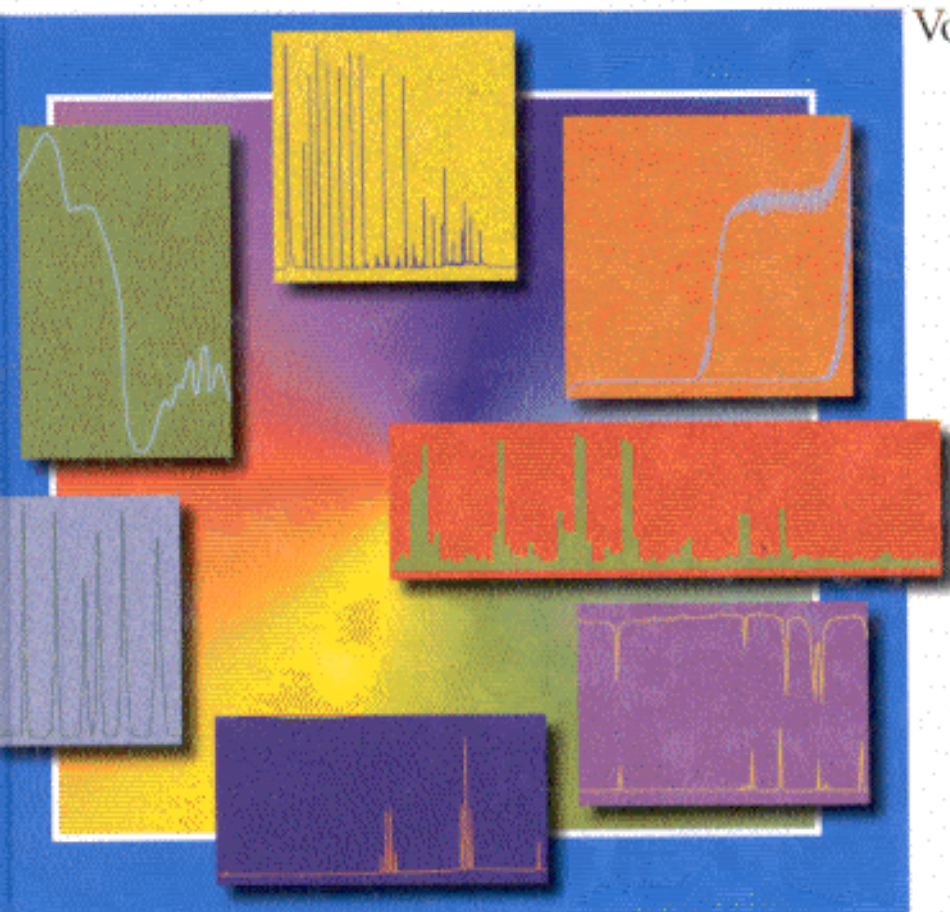


Handbook of Analytical Techniques

Edited by
Helmut Günzler and Alex Williams

Volume II



Contents

Volume I

1. Analytical Chemistry: Purpose and Procedures	1
1.1. The Evolution of Analytical Chemistry.	1
1.2. The Functional Organization of Analytical Chemistry.	4
1.3. Analysis Today.	5
1.4. Computers.	7
1.5. Analytical Tasks and Structures . . .	8
1.6. Definitions and Important Concepts.	13
1.7. "Legally Binding Analytical Results"	20
1.8. References.	20
2. Quality Assurance in Instrumentation	23
2.1. Introduction.	23
2.2. Selecting a Vendor.	24
2.3. Installation and Operation of Equipment.	25
2.4. Qualification of Software and Computer Systems.	29
2.5. Routine Maintenance and Ongoing Performance Control.	30
2.6. Handling of Defective Instruments.	34
2.7. References.	35
3. Chemometrics	37
3.1. Introduction.	37
3.2. Measurements and Statistical Distributions.	38
3.3. Statistical Tests.	40
3.4. Comparison of Several Measurement Series.	44
3.5. Regression and Calibration.	45
3.6. Characterization of Analytical Procedures.	47
3.7. Signal Processing.	49
3.8. Basic Concepts of Multivariate Methods.	51
3.9. Factorial Methods.	53
3.10. Classification Methods.	56
3.11. Multivariate Regression.	58
3.12. Multidimensional Arrays.	59
3.13. References.	61
4. Weighing	63
4.1. Introduction.	63
4.2. The Principle of Magnetic Force Compensation.	63
4.3. Automatic and Semiautomatic Calibration.	65
4.4. Processing and Computing Functions.	66
4.5. Balance Performance.	66
4.6. Fitness of a Balance for Its Application.	67
4.7. Gravity and Air Buoyancy.	67
4.8. The Distinction Between Mass and Weight.	68
4.9. Qualitative Factors in Weighing.	68
4.10. Governmental Regulations and Standardization.	69
4.11. References.	69

5. Sampling	71
5.1. Introduction and Terminology.....	71
5.2. Probability Sampling.....	72
5.3. Basic Sampling Statistics.....	73
5.4. Acceptance Sampling.....	74
5.5. Conclusions.....	76
5.6. References.....	76
6. Sample Preparation for Trace Analysis	77
6.1. Introduction.....	78
6.2. Sample Preparation and Digestion in Inorganic Analysis.....	80
6.3. Sample Preparation in Organic Analysis.....	96
6.4. References.....	104
7. Trace Analysis	109
7.1. Subject and Scope.....	110
7.2. Fields of Work.....	110
7.3. Methods of Modern Trace Analysis.....	111
7.4. Calibration and Validation.....	113
7.5. Environmental Analysis.....	117
7.6. References.....	125
8. Radionuclides in Analytical Chemistry	127
8.1. Introduction.....	127
8.2. Requirements for Analytical Use of Radionuclides.....	131
8.3. Radiotracers in Methodological Studies.....	134
8.4. Isotope Dilution Analysis.....	136
8.5. Radioreagent Methods.....	140
8.6. References.....	145
9. Enzyme and Immunoassays	147
9.1. Enzymatic Analysis Methods.....	147
9.2. Immunoassays in Analytical Chemistry.....	158
9.3. References.....	171
10. Basic Principles of Chromatography	173
10.1. Introduction.....	174
10.2. Historical Development.....	175
10.3. Chromatographic Systems.....	176
10.4. Theory of Linear Chromatography.....	177
10.5. Flow Rate of the Mobile Phase.....	182
10.6. The Thermodynamics of Phase Equilibria and Retention.....	183
10.7. Band Broadening.....	186
10.8. Qualitative Analysis.....	189
10.9. Quantitative Analysis.....	192
10.10. Theory of Nonlinear Chromatography.....	194
10.11. Reference Material.....	196
10.12. References.....	197

11. Gas Chromatography	199
11.1. Introduction	200
11.2. Instrumental Modules	201
11.3. The Separation System	201
11.4. Choice of Conditions of Analysis ..	212
11.5. Sample Inlet Systems	215
11.6. Detectors	231
11.7. Practical Considerations in Qualitative and Quantitative Analysis	242
11.8. Coupled Systems	244
11.9. Applicability	250
11.10. Recent and Future Developments ..	254
11.11. References	258
12. Liquid Chromatography	261
12.1. General	262
12.2. Equipment	266
12.3. Solvents (Mobile Phase)	283
12.4. Column Packing (Stationary Phase) ..	285
12.5. Separation Processes	288
12.6. Gradient Elution Technique	297
12.7. Quantitative Analysis	298
12.8. Sample Preparation and Derivatization	301
12.9. Coupling Techniques	305
12.10. Supercritical Fluid Chromatography	308
12.11. Affinity Chromatography	316
12.12. References	323
13. Thin Layer Chromatography	327
13.1. Introduction	327
13.2. Choice of the Sorbent Layer	327
13.3. Sample Cleanup	330
13.4. Sample Application	332
13.5. The Mobile Phase	334
13.6. Development	337
13.7. Visualization	339
13.8. Quantitation	341
13.9. References	344
14. Electrophoresis	345
14.1. Introduction	345
14.2. Basic Principles	346
14.3. Electrophoretic Matrices	346
14.4. Discontinuous Electrophoresis	350
14.5. Isoelectric Focusing	351
14.6. Sodium Dodecyl Sulfate Electrophoresis	355
14.7. Porosity Gradient Gels	355
14.8. Two-Dimensional Maps (Proteome Analysis)	356
14.9. Isotachopheresis	358
14.10. Immunoelectrophoresis	360
14.11. Staining Techniques and Blotting ..	362
14.12. Immobilized pH Gradients	362
14.13. Capillary Zone Electrophoresis	363
14.14. Preparative Electrophoresis	364
14.15. References	369
15. Structure Analysis by Diffraction	373
15.1. General Principles	373
15.2. Structure Analysis of Solids	374
15.3. Synchrotron Radiation	412
15.4. Neutron Diffraction	412
15.5. Electron Diffraction	413
15.6. Future Developments	413
15.7. References	414

16. Ultraviolet and Visible Spectroscopy	419
16.1. Introduction	420
16.2. Theoretical Principles	421
16.3. Optical Components and Spectrometers	430
16.4. Uses of UV – VIS Spectroscopy in Absorption, Fluorescence, and Reflection	443
16.5. Special Methods	452
16.6. References	459
17. Infrared and Raman Spectroscopy	465
17.1. Introduction	466
17.2. Techniques	466
17.3. Basic Principles of Vibrational Spectroscopy	470
17.4. Interpretation of Infrared and Raman Spectra of Organic Compounds. . .	474
17.5. Applications of Vibrational Spectroscopy	489
17.6. Near-Infrared Spectroscopy	502
17.7. References	504
18. Nuclear Magnetic Resonance and Electron Spin Resonance Spectroscopy	509
18.1. Introduction	510
18.2. Principles of Magnetic Resonance. .	511
18.3. High-Resolution Solution NMR Spectroscopy	514
18.4. NMR of Solids and Heterogeneous Systems	546
18.5. NMR Imaging	547
18.6. ESR Spectroscopy	548
18.7. References	557

Volume II

19. Mössbauer Spectroscopy	561
19.1. Introduction	561
19.2. Principle and Experimental Conditions of Recoil-free Nuclear Resonance Fluorescence.	561
19.3. Mössbauer Experiment.	564
19.4. Preparation of Mössbauer Source and Absorber	567
19.5. Hyperfine Interactions	568
19.6. Evaluation of Mössbauer Spectra ..	573
19.7. Selected Applications	574
19.8. References	577
20. Mass Spectrometry	579
20.1. Introduction	580
20.2. General Techniques and Definitions	580
20.3. Sample Inlets and Interfaces.	585
20.4. Ion Generation	590
20.6. Analyzers.	597
20.7. Metastable Ions and Linked Scans. .	603
20.8. MS/MS Instrumentation.	604
20.9. Detectors and Signals	607
20.10. Computer and Data Systems.	610
20.11. Applications	613
20.12. References	622

21. Atomic Spectroscopy	627
21.1. Introduction	628
21.2. Basic Principles	629
21.3. Spectrometric Instrumentation.	642
21.4. Sample Introduction Devices	660
21.5. Atomic Absorption Spectrometry ..	673
21.6. Atomic Emission Spectrometry....	688
21.7. Plasma Mass Spectrometry.....	704
21.8. Atomic Fluorescence Spectrometry .	713
21.9. Laser-Enhanced Ionization Spectrometry	716
21.10. Comparison With Other Methods ..	718
21.11. References	721
22. Laser Analytical Spectroscopy	727
22.1. Introduction	727
22.2. Tunable Lasers	730
22.3. Laser Techniques for Elemental Analysis.....	732
22.4. Laser Techniques for Molecular Analysis	744
22.5. Laser Ablation	750
22.6. References	751
23. X-Ray Fluorescence Spectrometry	753
23.1. Introduction	753
23.2. Historical Development of X-ray Spectrometry	755
23.3. Relationship Between Wavelength and Atomic Number	755
23.4. Instrumentation.....	757
23.5. Accuracy	760
23.6. Quantitative Analysis.....	761
23.7. Trace Analysis	762
23.8. New developments in Instrumentation and Techniques ...	763
23.9. References	765
24. Activation Analysis	767
24.1. Introduction	767
24.2. Neutron Activation Analysis.....	768
24.3. Photon Activation Analysis	779
24.4. Charged-Particle Activation Analysis	780
24.5. Applications	781
24.6. Evaluation of Activation Analysis..	783
24.7. References	783
25. Analytical Voltammetry and Polarography	785
25.1. Introduction	785
25.2. Techniques.....	788
25.3. Instrumentation.....	803
25.4. Evaluation and Calculation.....	808
25.5. Sample Preparation	810
25.6. Supporting Electrolyte Solution ...	812
25.7. Application to Inorganic and Organic Trace Analysis	814
25.8. References	823
26. Thermal Analysis and Calorimetry	827
26.1. Thermal Analysis	827
26.2. Calorimetry	836
26.3. References	849

27. Surface Analysis	851
27.1. Introduction	852
27.2. X-Ray Photoelectron Spectroscopy (XPS)	854
27.3. Auger Electron Spectroscopy (AES)	874
27.4. Static Secondary Ion Mass Spectrometry (SSIMS)	889
27.5. Ion Scattering Spectroscopies (ISS and RBS)	898
27.6. Scanning Tunneling Methods (STM, STS, AFM)	910
27.7. Other Surface Analytical Methods ..	917
27.8. Summary and Comparison of Techniques	940
27.9. Surface Analytical Equipment Suppliers	940
27.10. References	944
 28. Chemical and Biochemical Sensors	 951
28.1. Introduction to the Field of Sensors and Actuators	952
28.2. Chemical Sensors	953
28.3. Biochemical Sensors (Biosensors) ..	1032
28.4. Actuators and Instrumentation	1051
28.5. Future Trends and Outlook	1052
28.6. References	1053
 29. Microscopy	 1058
29.1. Modern Optical Microscopy	1061
29.2. Electron Microscopy	1077
29.3. References	1125
 30. Techniques for DNA Analysis	 1131
30.1. Introduction	1131
30.2. Primary Molecular Tools for DNA Analysis	1133
30.3. Methods of DNA Detection	1135
30.4. Applications of DNA Analysis	1144
30.5. References	1150
 Subject Index	 1151