

Contents

Foreword *ix*

Preface *xi*

1	An Overview	1
1.1	Features of ICP-OES	1
1.2	Inductively Coupled Plasma Optical Emission Spectrometry – the Name Describes the Technique	2
1.3	Distribution of ICP-OES	4
1.4	Related Techniques for Elemental Analysis	4
1.5	Terms	8
2	Plasma	9
2.1	The Spectrometric Plasma	9
2.1.1	The Operating Gas	10
2.1.1.1	Argon	10
2.1.1.2	Addition of Air or Oxygen	11
2.1.2	Plasma Torch	12
2.1.3	Ignition of the Plasma	15
2.1.4	Orientation of the Plasma with Respect to the Torch	15
2.2	Excitation to Emit Electromagnetic Radiation (Light)	16
2.2.1	Emission Lines	16
2.2.2	Energy and Temperature	19
2.2.3	Spectroscopic Properties of the ICP	22
2.2.4	Plasma Viewing	28
2.2.4.1	Radial Viewing	29
2.2.4.2	Axial Viewing	30
2.2.4.3	Radial and Axial Viewing in One Instrument (“Dual View”)	31
2.3	Excitation Unit	35
2.3.1	Radio Frequency Generator	35
2.3.2	Induction Coil	38
2.4	Sample Introduction System	38
2.4.1	Nebulizer	40
2.4.1.1	Pneumatic Nebulizers	41
2.4.1.2	High-Pressure Nebulizer	45
2.4.1.3	Ultrasonic Nebulizer	46

2.4.2	Nebulizer Chamber	48
2.4.2.1	Tasks of the Nebulizer Chamber	48
2.4.2.2	Temperature of the Nebulizer Chamber	49
2.4.2.3	Materials and Surface Properties	52
2.4.2.4	Common Types of Nebulizer Chambers	54
2.4.2.5	Waste from the Nebulizer Chamber	55
2.4.3	Pump	56
2.4.4	Other Forms of Sample Introduction	59
2.4.4.1	Special Techniques for Liquid Samples	60
2.4.4.2	Gaseous Samples	60
2.4.4.3	Solid Sampling	63
3	Optics and Detector of the Spectrometer	67
3.1	Basic Principles of Optics	67
3.1.1	Resolution	67
3.1.2	Relevant Optical Terms	72
3.1.3	Optical Mounts	76
3.1.3.1	Paschen–Runge Mount	76
3.1.3.2	Czerny–Turner Mount	76
3.1.3.3	Echelle Mount	78
3.1.3.4	Littrow Mount	80
3.1.4	Light Transfer from the Plasma to the Optics	80
3.1.4.1	Separation of Plasma Compartment and Optics	80
3.1.4.2	Transparency of the Optics in the Vacuum-UV Range	82
3.2	Detectors	85
3.2.1	Photomultiplier Tube (PMT)	86
3.2.2	Solid-State Detectors	87
3.3	Types of Emission Spectrometer Mounts	95
3.3.1	Classical Spectrometers	96
3.3.1.1	Monochromators	96
3.3.1.2	Polychromators	96
3.3.2	Array Spectrometers	97
3.3.2.1	Scanning Array Spectrometers	97
3.3.2.2	Simultaneous Array Spectrometers	97
4	Method Development	99
4.1	Wavelength Selection	101
4.1.1	Working Range	101
4.1.1.1	Background Equivalent Concentration (BEC)	102
4.1.2	Freedom from Spectral Interference	103
4.1.2.1	Generation of Spectra for the Estimation of the Background	107
4.1.2.2	Generation of Spectra for Detecting Interfering Lines from the Matrix	111
4.1.2.3	Evaluating Spectra	114
4.2	Processing and Correction Techniques	117
4.2.1	Signal Processing	117
4.2.1.1	Calculation of the Peak Height	117
4.2.1.2	Calculation of the Peak Area or of the Partial Peak Area	119

4.2.1.3	Calibration of the Peak Position	122
4.2.2	Background Correction	123
4.2.2.1	Calculation of the Background Correction	126
4.2.2.2	Number the Background Correction Points	128
4.2.3	Impact of Peak Processing and Background Correction on Detection Limits	132
4.2.4	Correction of Spectral Interference	137
4.2.4.1	Inter-element Correction	137
4.2.4.2	Correction Using Multivariate Regression	138
4.2.4.3	Multivariate Regression and Inter-element Correction	147
4.3	Non-spectral Interference	147
4.3.1	Correction of Non-spectral Interference	148
4.3.1.1	Matrix Matching	148
4.3.1.2	Internal Standard	149
4.3.1.3	Calibration with Analyte Addition (Standard Addition)	151
4.3.1.4	Further Measures to Compensate for Non-spectral Interference	153
4.4	Optimization	153
4.4.1	Optimization Goals	154
4.4.2	Optimization Parameters	155
4.4.3	Optimization Algorithms	155
4.5	Validation	157
4.5.1	Accuracy and Specificity	157
4.5.2	Reproducibility	159
4.5.3	Limit of Detection	160
4.5.4	Working Range	164
4.5.5	Robustness	166
5	Routine Analysis	169
5.1	Preparation	169
5.1.1	Sample Preparation	169
5.1.2	Warm-up Time	170
5.1.3	Delay and Rinse Times	171
5.2	Calibration	173
5.2.1	Calibration Solutions	173
5.2.1.1	Number of Calibration Solutions	173
5.2.1.2	Concentrations in Calibration Solutions	174
5.2.1.3	Multielement Calibration Solutions	176
5.2.1.4	Multi-bottle Calibration	176
5.2.1.5	Stability of Calibration Solutions	177
5.2.2	Calibration Functions	177
5.2.2.1	External Calibration	178
5.2.2.2	Calibration by Analyte Addition (Standard Addition)	179
5.2.2.3	Bracketing Calibration	179
5.2.3	Examination of the Calibration Data	181
5.3	Quality Assurance	182
5.4	Software and Data Processing	184
6	Troubleshooting and Maintenance	187

7	Applications	197
7.1	General Notes	197
7.1.1	Material of Containers	197
7.1.2	Stability of Solutions	197
7.1.3	Matrix Effects	198
7.1.4	Contaminations	198
7.2	Comments on Selected Elements	198
7.3	Selected Applications	200
7.3.1	Environment	201
7.3.1.1	Drinking, Ground, and Surface Water	202
7.3.1.2	Wastewater, Leachates	202
7.3.1.3	Sludges	204
7.3.1.4	Soil Samples, Sediments	204
7.3.1.5	Airborne Particles, Fly Ashes	205
7.3.2	Samples of Biological Origin	206
7.3.2.1	Plant and Animal Samples	206
7.3.2.2	Clinical and Forensic Materials	207
7.3.2.3	Food and Animal Feeds	208
7.3.3	Geological Materials	208
7.3.4	Metallurgy	210
7.3.4.1	Steel and Iron Matrices	210
7.3.4.2	Nonferrous Metals	212
7.3.4.3	Noble Metals	212
7.3.4.4	Special Alloys	212
7.3.5	Material Sciences	213
7.3.5.1	Semiconductors	213
7.3.5.2	Ceramics	215
7.3.6	Industrial Applications	215
7.3.6.1	Industrial Chemicals and Fertilizers	215
7.3.6.2	Galvanizing/Electroplating Baths	216
7.3.6.3	Brines and Salts	216
7.3.6.4	Cement, Gypsum, Calcium Matrix	216
7.3.6.5	Glass	217
7.3.6.6	Other Industrial Applications	218
7.3.7	Organic Solvents	218
7.3.7.1	Wear Metals and Contamination in Oil	221
7.3.7.2	Additives	223
7.3.7.3	Tar	223
7.3.7.4	Edible Oils	223
8	Procurement of Equipment and Preparation of the Laboratory	225
8.1	Which Atomic Spectrometric Technique Is the Most Suitable?	225
8.2	Which ICP Emission Spectrometer Is the Most Suitable?	227
8.3	Preparation of the Laboratory	230
	References	233
	Index	269