

contents

preface	ix
----------------------	----

symbols and terminology	xi
--------------------------------------	----

CHAPTER 1 introduction

1.1 THE PLACE OF THE SPECTROMETER IN THE MODERN LABORATORY	1
1.2 X-RADIATION AND X-RAY SPECTROMETRY	2
1.3 EARLY DEVELOPMENT OF X-RAY SPECTROSCOPIC ANALYSIS...	3
1.4 THE MODERN X-RAY SPECTROMETER	6

CHAPTER 2 X-ray spectra

2.1 BASIC UNITS	8
2.2 ELECTRON CONFIGURATIONS OF THE ELEMENTS	10
2.3 PRODUCTION OF CONTINUOUS RADIATION	14
2.4 EXCITATION AND DE-EXCITATION	16
2.5 IONIZATION AND EXCITATION POTENTIALS	18
2.6 TRANSITION AND DIAGRAM LINES	20
2.7 SATELLITE LINES	21
2.8 CHARACTERISTIC LINE SPECTRA	21
2.9 K SERIES	21
2.9.1 The tin K spectrum.....	21

2.9.2 The copper K spectrum	23
2.9.3 The calcium K spectrum	23
2.9.4 The aluminium K spectrum.....	24
2.9.5 The oxygen K spectrum.....	26
2.10 L SPECTRA	27
2.10.1 The gold L spectrum	27
2.10.2 The strontium L spectrum.....	30
2.11 M SPECTRA	31
2.12 RELATIVE LINE INTENSITIES	33

CHAPTER 3 physics of X-rays

3.1 ABSORPTION OF X-RADIATION	37
3.1.1 Mass absorption coefficient	39
3.2 EXCITATION OF CHARACTERISTIC RADIATION.....	40
3.2.1 Primary and secondary absorption.....	41
3.2.2 Excitation efficiency	43
3.2.3 Enhancement and third element effects	43
3.3 INTERFERENCE AND DIFFRACTION PHENOMENA	44
3.3.1 Diffraction of X-rays	46
3.3.2 The crystalline state	47
3.3.3 Miller indices	47
3.3.4 Conditions for the diffraction of X-rays	48
3.3.5 Efficiency of X-ray diffraction	49

CHAPTER 4 instrumentation

4.1 EXCITATION SOURCES FOR X-RADIATION.....	52
4.1.1 Requirements of an X-ray source.....	54
4.1.2 Other configurations of X-ray tubes	57
4.1.3 Direct electron excitation.....	58
4.1.4 Proton excitation	59
4.1.5 Excitation by radioisotopes	59
4.2 DETECTION OF X-RAYS	60
4.2.1 The gas flow proportional detector	61
4.2.2 The scintillation detector	68
4.2.3 The semiconductor detector	70
4.2.4 Pulse height selection	73

4.3 SEPARATION OF A POLYCHROMATIC BEAM OF RADIATION	75
4.3.1 The crystal spectrometer	76
4.3.2 Geometry of the crystal spectrometer	78
4.3.3 Resolution of the spectrometer	81
4.3.4 Choice of analysing crystal	87
4.3.5 Angular reproducibility of the spectrometer	88
4.3.6 Use of primary beam filters	89
4.3.7 Typical instrumentation for analysis with a crystal spectrometer	91
4.3.8 The energy dispersion spectrometer	92
4.3.9 Sensitivity of the energy dispersion spectrometer	93
4.3.10 Data handling in the energy dispersion spectrometer	96

CHAPTER 5 qualitative analysis

5.1 QUALITATIVE ANALYSIS	99
5.1.1 Qualitative and semi-quantitative analysis with the crystal spectrometer	100
5.1.2 Qualitative and semi-quantitative analysis with the energy dispersion spectrometer	103

CHAPTER 6 errors in X-ray analysis

6.1 TYPES OF ERROR IN X-RAY ANALYSIS	107
6.2 SOURCES OF RANDOM ERROR IN QUANTITATIVE X-RAY SPECTROMETRY	109
6.3 THE LOWER LIMIT OF DETECTION	111
6.4 SYSTEMATIC ERRORS IN QUANTITATIVE X-RAY SPECTROMETRY	114
6.5 SAMPLE PREPARATION	115
6.6 ESTIMATION OF ERRORS IN QUANTITATIVE ANALYSIS	117

CHAPTER 7 quantitative analysis

7.1 BASIS OF QUANTITATIVE ANALYSIS	120
7.2 RELATIONSHIP BETWEEN X-RAY INTENSITY AND THE SAMPLE MATRIX	121
7.3 THE FUNDAMENTAL PARAMETERS APPROACH	123
7.4 THE CONCEPT OF EFFECTIVE WAVELENGTH	125

7.5	ALGORITHMS BASED ON THE USE OF EFFECTIVE WAVELENGTH	128
7.6	FURTHER SIMPLIFICATIONS OF THE INTENSITY/CONCENTRATION ALGORITHM	128
7.7	SECONDARY ABSORPTION CORRECTION	130
7.8	EMPIRICAL CORRECTION PROCEDURES.....	132
7.9	SEMI-EMPIRICAL METHODS.....	132
7.10	APPLICATION OF SEMI-EMPIRICAL CORRECTIONS IN MULTI-ELEMENT ANALYSES.....	136
7.11	EMPIRICAL POLYNOMIALS	138

CHAPTER 8 the study of chemical bonding

8.1	INTRODUCTION TO THE USE OF X-RAY METHODS FOR THE STUDY OF CHEMICAL BONDING	141
8.2	BASIS OF METHODS FOR THE STUDY OF CHEMICAL BONDING...	143
8.3	INSTRUMENTATION FOR THE STUDY OF CHEMICAL BONDING..	144
8.4	USE OF X-RAY EMISSION WAVELENGTHS	146
8.5	ESCA METHODS.....	149
8.5.1	Different forms of orbital coupling	151
8.5.2	Application of ESCA methods	152
8.6	ABSORPTION EDGE FINE STRUCTURE	153
index	159

appendices I, II, III, principal emission lines of X-ray spectra; K series, M series, L series, respectively *pocket at back*