

Table of Contents

Foreword

Preface

Acknowledgements	P-2
------------------------	-----

Glossary of Terms

G.1 LIST OF TERMS AND ABBREVIATIONS USED IN THIS BOOK	G-1
---	-----

Chapter 1: Introduction

1.1 INTRODUCTION	1-1
1.2 INSTRUMENTAL PARAMETERS OR VARIABLES	1-3
1.2.1 Sequential WDXRF spectrometers	1-3
1.2.2 Simultaneous WDXRF spectrometers	1-3
1.2.3 EDXRF spectrometers	1-5
1.3 WDXRF vs EDXRF	1-6
1.4 SELECTING THE BEST SET OF INSTRUMENTAL PARAMETERS.....	1-11
1.5 SAFETY	1-12
1.6 CONCLUSION.....	1-13

Chapter 2: Sample preparation

2.1 INTRODUCTION	2-1
2.2 CONTAMINATION.....	2-1
2.3 CRITICAL (ANALYSIS) DEPTH.....	2-2
2.4 PREPARATION OF GEOLOGICAL SAMPLES	2-6
2.4.1 Primary sample preparation.....	2-6
2.4.1.1 Crushing.....	2-6
2.4.1.2 Splitting	2-6
2.4.1.3 Pulverising (milling or grinding)	2-6
2.4.1.4 Sieving.....	2-8
2.4.1.5 Desliming	2-8
2.4.1.6 Removal of entrained metal prills	2-8
2.4.2 Preparation of pressed powder pellets.....	2-8
2.4.2.1 Binders.....	2-9
2.4.2.2 Mixing by milling.....	2-11
2.4.2.3 Pressing	2-11
2.4.2.4 Dilution with heavy absorbers.....	2-12
2.4.2.5 Pressed pellet supports.....	2-13
2.4.3 Particle size effects.....	2-13
2.4.4 Mineralogical effects.....	2-13
2.4.5 Preparation of fused beads	2-14
2.4.5.1 Pre-oxidation.....	2-14

2.4.5.2	Loss (LOI) and gain (GOI) on fusion	2-17
2.4.5.3	Fluxes	2-18
2.4.5.4	Additives	2-20
2.4.5.5	Nonwetting or Releasing agents	2-20
2.4.5.6	Mixing sample and flux	2-21
2.4.5.7	Fusion temperature	2-21
2.4.5.8	Casting and cooling	2-21
2.4.5.9	Handling and storage of fused beads	2-22
2.4.5.10	Problems with fused beads	2-22
2.4.5.11	Fully automated fused bead preparation systems	2-23
2.5	PREPARATION OF METAL SAMPLES	2-23
2.6	PREPARATION OF LIQUID SAMPLES	2-25
2.6.1	Precautions	2-26
2.6.2	Problems that can arise during the analysis of liquids.....	2-27
2.6.3	Alternative liquid sample preparation methods.....	2-27
2.7	OTHER SAMPLE PREPARATIONS.....	2-28
2.7.1	Preparation of air filter samples	2-28
2.7.2	Preparation of biological material	2-28
2.7.3	Preparation of small samples	2-28
2.8	REFERENCES	2-28

Chapter 3: Calibration Standards

3.1	INTRODUCTION	3-1
3.2	REFERENCE MATERIALS	3-1
3.3	SOURCES OF REFERENCE MATERIALS	3-2
3.3.1	Pre-treatment of standard materials.....	3-5
3.3.2	Most recent updates of analyte concentrations.....	3-5
3.4	MATRIX MATCHING.....	3-5
3.5	STANDARD ADDITIONS.....	3-6
3.6	FUSED BEADS USING REAGENTS	3-7
3.6.1	Sample mass and flux mass are fixed	3-10
3.6.2	Sample mass and flux mass are varied	3-10
3.7	SOLUTIONS	3-10

Chapter 4: Wavelength Dispersive XRF

4.1	INTRODUCTION	4-1
4.2	INSTRUMENTAL PARAMETERS OR VARIABLES FOR WDXRF.....	4-1
4.2.1	Sequential WDXRF spectrometers	4-1
4.2.2	Simultaneous WDXRF spectrometers	4-2
4.3	CHARACTERISTICS OF INDIVIDUAL INSTRUMENTAL PARAMETERS	4-2
4.3.1	X-ray tubes	4-2
4.3.1.1	End-window X-ray tubes	4-2
4.3.1.2	Side-window X-ray tubes	4-3
4.3.2	kV and mA.....	4-4
4.3.2.1	Effect of kV settings on excitation efficiency.....	4-6

4.3.3	Primary beam filters.....	4-10
4.3.4	Collimator masks.....	4-16
4.3.5	Primary collimators.....	4-17
4.3.6	Analysing crystals.....	4-19
4.3.6.1	Crystalline materials.....	4-19
4.3.6.2	Layered synthetic microstructures (LSMs).....	4-19
4.3.6.3	Available crystals and LSMs.....	4-21
4.3.7	Secondary collimators.....	4-23
4.3.8	Detectors.....	4-23
4.3.8.1	Scintillation detector.....	4-24
4.3.8.2	Ar-CH ₄ gas flow detector.....	4-25
4.3.8.3	Xe sealed gas detector.....	4-27
4.3.8.4	Detector resolution.....	4-27
4.3.8.5	General.....	4-28
4.3.8.6	Escape Peaks.....	4-28
4.3.8.7	Dead time.....	4-30
4.3.9	Pulse Height Selector.....	4-30
4.3.9.1	Escape Peaks and Pulse Height Selection.....	4-33
4.3.10	Fixed channels.....	4-33
4.3.11	X-ray path medium.....	4-35
4.3.12	Counting time and X-ray statistics.....	4-35
4.3.12.1	Counting error of a single measurement.....	4-35
4.3.12.2	Counting error of the net peak intensity.....	4-36
4.3.12.3	Confidence Limits.....	4-36
4.3.12.4	Lower Limit of Detection (LLD).....	4-36
4.4	RESOLUTION.....	4-37
4.4.1	Angular dispersion.....	4-37
4.4.2	High order peaks.....	4-37
4.4.3	Peak resolution.....	4-39
4.5	REFERENCE.....	4-40

Chapter 5: Energy Dispersive XRF

5.1	INTRODUCTION.....	5-1
5.2	INSTRUMENTAL PARAMETERS OR VARIABLES.....	5-1
5.2.1	Types of EDXRF spectrometer.....	5-1
5.2.2	Direct excitation spectrometer with primary beam filters.....	5-2
5.2.2.1	X-ray tube.....	5-2
5.2.2.2	kV and mA.....	5-5
5.2.2.3	Primary beam filters.....	5-5
5.2.2.4	Detectors.....	5-6
5.2.2.5	X-ray path medium.....	5-10
5.2.2.6	Counting times.....	5-10
5.2.2.7	Region of Interest (ROI).....	5-10
5.2.2.8	Processing an EDXRF spectrum.....	5-10
5.2.3	Secondary target excitation spectrometer with Cartesian geometry.....	5-12

5.2.3.1	X-ray tube	5-12
5.2.3.2	kV and mA.....	5-12
5.2.3.3	Secondary targets.....	5-12
5.2.3.4	Detectors.....	5-13
5.2.3.5	X-ray path medium	5-13
5.2.3.6	Counting times	5-13
5.2.4	Hand-held EDXRF spectrometers.....	5-14
5.2.4.1	X-ray tube	5-15
5.2.4.2	kV and mA.....	5-15
5.2.4.3	Detectors.....	5-15
5.2.4.4	X-ray path medium	5-15
5.2.4.5	Counting times	5-16
5.3	COMPARISON OF SPECTROMETER AND DETECTOR PERFORMANCE FOR DIFFERENT ENERGY RANGES	5-16
5.4	IN-STREAM ANALYSIS AND DOWN-HOLE BOREHOLE LOGGING.....	5-20
5.5	TOTAL REFLECTION XRF (TXRF)	5-21
5.6	REFERENCES	5-23

Chapter 6: Setting up an Analytical Programme

6.1	INTRODUCTION	6-1
6.2	WDXRF	6-1
6.2.1	Setting up an analytical programme for WDXRF	6-2
6.2.1.1	A suggested procedure for setting up an analytical programme	6-4
6.2.1.2	Figure of Merit (FOM)	6-7
6.3	METHODS FOR DETERMINING BACKGROUND AT A PEAK POSITION	6-10
6.3.1	Selecting a method for the determination of background at a peak position	6-10
6.3.2	Using one background position	6-10
6.3.3	Using two background positions	6-11
6.3.4	Using three or more background positions and a polynomial or spline fit	6-12
6.3.5	Estimating background intensity using the Feather and Willis method	6-13
6.3.6	Background determination on major and minor element peak "tails" and across absorption edges	6-14
6.4	CORRECTING FOR SPECTRAL INTERFERENCE FROM TUBE IMPURITIES	6-17
6.4.1	Introduction	6-17
6.4.2	Correction for spectral interference from impurities in the X-ray tube.....	6-17
6.4.3	Application of the Feather and Willis method to correct for spectral interference from tube impurities	6-19
6.5	CORRECTING FOR SPECTRAL OVERLAP	6-19
6.5.1	Spectral interference samples, blanks and samples.....	6-19

6.5.1.1	Spectral overlap corrections	6-21
6.6	CORRECTING FOR INTER-ELEMENT MATRIX EFFECTS	6-22
6.6.1	Using a ratio to background.....	6-22
6.6.2	Using MACs.....	6-23
6.6.3	Using influence coefficients	6-24
6.6.3.1	Using empirical influence coefficients.....	6-24
6.6.3.2	Using theoretical influence coefficients.....	6-24
6.6.3.3	General, Lachance-Traill and de Jongh theoretical binary influence coefficient equations, eliminated alphas, including loss eliminated alphas	6-25
6.6.3.4	"Loss-eliminated alphas"	6-27
6.6.3.5	Using theoretical intensities (Fundamental Parameter models)	6-27
6.7	CALIBRATION STRATEGIES.....	6-28
6.7.1	Calibration strategies for fused beads	6-28
6.7.1.1	Calibration using CRMs and/or analysed samples.....	6-29
6.7.1.2	Calibration using a single multi-element synthetic standard ("Line only" method).....	6-29
6.7.2	Calibration strategies for major elements in pressed powders	6-30
6.7.3	Calibration strategies for trace elements in pressed powders... ..	6-31
6.7.4	Calibration using internal standards.....	6-32
6.7.5	Drift correction	6-34
6.8	SETTING UP AN ANALYTICAL PROGRAMME FOR EDXRF.....	6-36
6.8.1	EDXRF operating conditions.....	6-37
6.9	CHECKING WDXRF SPECTROMETER DEAD TIME	6-38
6.10	REFERENCES	6-42

Chapter 7: X-ray Tube Compton peak

7.1	INTRODUCTION	7-1
7.2	INSTRUMENTAL PARAMETERS	7-1
7.2.1	Sequential WDXRF spectrometers	7-1
7.3	EDXRF	7-3
7.4	ADDITIONAL COMMENTS.....	7-3
7.5	LIMITATIONS ON THE USE OF COMPTON PEAKS	7-5
7.6	WORKED EXAMPLE	7-6
7.6.1	Estimating MACs using the RhK α Compton peak	7-6
7.7	REFERENCES	7-7

Chapter 8: I, Te, Sb, Sn, Cd, Ag

8.1	INTRODUCTION	8-1
8.1.1	Sample preparation.....	8-1
8.2	INSTRUMENTAL PARAMETERS	8-1
8.2.1	Sequential WDXRF spectrometers - K Series lines.....	8-1
8.2.1.1	kV and mA	8-3
8.2.1.2	Primary beam filter	8-3
8.2.1.3	Primary collimator.....	8-4

8.2.1.4	Analysing crystal.....	8-4
8.2.1.5	Background positions	8-5
8.2.1.6	Spectral overlap corrections	8-7
8.2.1.7	Detector	8-9
8.2.1.8	Pulse Height Selection	8-9
8.2.2	Sequential WDXRF spectrometers - L Series lines	8-9
8.2.2.1	Sample preparation.....	8-9
8.2.2.2	Instrumental parameters	8-10
8.2.3	EDXRF spectrometers	8-13
8.2.3.1	K series lines.....	8-13
8.2.3.2	L series lines	8-15
8.3	MEASUREMENT OF Ba AND Cs.....	8-15
8.3.1	Correction for inter-element matrix effects	8-18
8.4	FLOWCHART	8-18
8.5	REFERENCES	8-18

Chapter 9: Mo, Nb, Zr, Y, Sr, U, Rb, Th, Pb, (Br)

9.1	INTRODUCTION	9-1
9.1.1	Sample preparation.....	9-1
9.2	INSTRUMENTAL PARAMETERS	9-1
9.2.1	Sequential WDXRF spectrometers	9-1
9.2.1.1	kV and mA.....	9-2
9.2.1.2	Primary beam filter	9-2
9.2.1.3	Primary collimator.....	9-3
9.2.1.4	Analysing crystal.....	9-3
9.2.1.5	Background positions	9-4
9.2.1.6	Spectral overlap corrections	9-8
9.2.1.7	Detector	9-8
9.2.1.8	Pulse Height Selection	9-8
9.2.2	EDXRF spectrometers	9-9
9.3	ADDITIONAL COMMENTS.....	9-10
9.3.1	Choice of Th analyte line.....	9-10
9.3.2	Correction for inter-element matrix effects	9-12
9.4	FLOWCHART	9-13
9.5	REFERENCE	9-13

Chapter 10: Br, As, Se, Bi, Tl, (Pb, Ge)

10.1	INTRODUCTION	10-1
10.1.1	Sample preparation.....	10-1
10.2	INSTRUMENTAL PARAMETERS	10-1
10.2.1	Sequential WDXRF spectrometers	10-1
10.2.1.1	kV and mA.....	10-1
10.2.1.2	Primary beam filter	10-2
10.2.1.3	Primary collimator.....	10-3
10.2.1.4	Analysing crystal.....	10-5
10.2.1.5	Analyte line selection.....	10-7

10.2.1.6	Background positions	10-7
10.2.1.7	Spectral overlap corrections	10-9
10.2.1.8	Detector	10-9
10.2.1.9	Pulse Height Selection	10-10
10.2.2	EDXRF spectrometers	10-10
10.3	ADDITIONAL COMMENTS.....	10-10
10.3.1	Correction for inter-element matrix effects	10-12
10.4	FLOWCHART	10-13

Chapter 11: Ge, Ga, Zn, Cu, Ni, W, Ta, Hf, (Co)

11.1	INTRODUCTION	11-1
11.1.1	Sample preparation.....	11-1
11.2	INSTRUMENTAL PARAMETERS	11-1
11.2.1	Sequential WDXRF spectrometers	11-1
11.2.1.1	kV and mA.....	11-1
11.2.1.2	Primary beam filter	11-1
11.2.1.3	Primary collimator.....	11-2
11.2.1.4	Analysing crystal.....	11-8
11.2.1.5	Analyte line selection.....	11-8
11.2.1.6	Background positions	11-10
11.2.1.7	Spectral overlap corrections	11-13
11.2.1.8	Detector	11-13
11.2.1.9	Pulse Height Selection	11-13
11.2.2	EDXRF spectrometers	11-13
11.3	ADDITIONAL COMMENTS.....	11-16
11.3.1	Background correction for Zn, Cu and Ni	11-17
11.3.2	Correcting Zn, Cu and Ni for tube spectral impurities	11-18
11.3.3	Feather and Willis background method.....	11-18
11.3.4	Spectral interference samples, blanks and samples.....	11-19
11.3.5	Correction for inter-element matrix effects	11-21
11.4	FLOWCHART	11-21
11.5	REFERENCE	11-21

Chapter 12: Co, Mn, Cr, V

12.1	INTRODUCTION	12-1
12.1.1	Sample preparation.....	12-1
12.2	INSTRUMENTAL PARAMETERS	12-1
12.2.1	Sequential WDXRF spectrometers	12-1
12.2.1.1	kV and mA.....	12-1
12.2.1.2	Primary beam filter	12-3
12.2.1.3	Primary collimator.....	12-4
12.2.1.4	Analysing crystal.....	12-5
12.2.1.5	Detector	12-6
12.2.1.6	Pulse Height Selection	12-7
12.2.1.7	Analyte line selection.....	12-9
12.2.1.8	Background positions and background determination	12-11

12.2.1.9	Spectral overlap corrections	12-12
12.2.2	EDXRF spectrometers	12-14
12.3	CORRECTION FOR INTER-ELEMENT MATRIX EFFECTS	12-14
12.4	FLOWCHART	12-16
12.5	REFERENCE	12-16

Chapter 13: Ba, Sc, La, Ce, Pr, Nd, Sm, Cs, (Y)

13.1	INTRODUCTION	13-1
13.1.1	Sample preparation.....	13-1
13.2	INSTRUMENTAL PARAMETERS	13-2
13.2.1	Sequential WDXRF spectrometers	13-3
13.2.1.1	kV and mA.....	13-4
13.2.1.2	Primary beam filter	13-4
13.2.1.3	Primary collimator.....	13-4
13.2.1.4	Analysing crystal.....	13-4
13.2.1.5	Detector	13-6
13.2.1.6	Pulse Height Selection	13-7
13.2.1.7	Analyte line selection.....	13-7
13.2.1.8	Background positions and background determination....	13-11
13.2.1.9	Spectral overlap corrections	13-12
13.2.2	EDXRF.....	13-12
13.3	ADDITIONAL COMMENTS.....	13-14
13.3.1	Rare earth element abundance pattern	13-14
13.3.2	Correction for inter-element matrix effects	13-14
13.4	FLOWCHART	13-14
13.5	REFERENCE	13-14

Chapter 14: Cl, S, P and Phosphates

14.1	INTRODUCTION	14-1
14.1.1	Sample preparation.....	14-1
14.2	INSTRUMENTAL PARAMETERS	14-1
14.2.1	Sequential WDXRF spectrometers	14-1
14.2.1.1	kV and mA.....	14-2
14.2.1.2	Primary beam filter	14-2
14.2.1.3	Primary collimator.....	14-3
14.2.1.4	Analysing crystal.....	14-3
14.2.1.5	Detector	14-4
14.2.1.6	Pulse Height Selection	14-4
14.2.1.7	Analyte line selection.....	14-6
14.2.1.8	Background positions and background determination....	14-6
14.2.1.9	Possible spectral line overlaps.....	14-8
14.2.1.10	Correcting for spectral overlap.....	14-9
14.2.2	EDXRF spectrometers	14-11
14.3	CORRECTION FOR INTER-ELEMENT MATRIX EFFECTS	14-12
14.4	ADDITIONAL COMMENTS.....	14-13
14.4.1	Chemical effects (wavelength shift)	14-13

14.5	FLOWCHART	14-14
14.6	PHOSPHATE DEPOSITS.....	14-14
14.6.1	Sample preparation.....	14-16
14.6.2	Analyte elements	14-16
14.6.3	Instrumental Parameters	14-16
14.6.3.1	Sequential WDXRF spectrometers	14-16
14.6.3.2	Simultaneous WDXRF spectrometers	14-17
14.6.3.3	EDXRF spectrometers	14-17
14.6.4	Spectral Overlap Corrections	14-17
14.6.5	Matrix Corrections.....	14-17
14.6.6	Calibration and Recommended Standards.....	14-18
14.7	REFERENCES	14-18

Chapter 15: F and Fluorspar (CaF₂)

15.1	INTRODUCTION	15-1
15.1.1	Sample preparation.....	15-1
15.2	INSTRUMENTAL PARAMETERS	15-1
15.2.1	Sequential WDXRF spectrometers	15-1
15.2.1.1	kV and mA.....	15-3
15.2.1.2	Primary beam filter	15-3
15.2.1.3	Primary collimator.....	15-3
15.2.1.4	Analysing crystal.....	15-4
15.2.1.5	Detector	15-6
15.2.1.6	Pulse Height Selection	15-6
15.2.1.7	Analyte line selection.....	15-6
15.2.1.8	Background positions and background determination.....	15-6
15.2.1.9	Spectral overlap corrections	15-7
15.2.2	EDXRF spectrometers	15-7
15.3	FLUORSPAR.....	15-8
15.4	CORRECTION FOR INTER-ELEMENT MATRIX EFFECTS	15-8
15.5	CALIBRATION STANDARDS	15-11
15.6	ADDITIONAL COMMENTS.....	15-12
15.7	FLOWCHART	15-13
15.8	REFERENCES	15-13

Chapter 16: Major and minor element analysis

16.1	INTRODUCTION	16-1
16.2	INSTRUMENTAL PARAMETERS	16-1
16.2.1	WDXRF.....	16-1
16.2.2	EDXRF.....	16-2
16.3	STANDARDS	16-2
16.3.1	Fusions.....	16-3
16.3.2	Pressed powders.....	16-3
16.4	FUSION METHODS.....	16-3
16.4.1	Sample preparation (see also Chapter 2, Section 2.4.5)	16-3
16.4.2	Calibration strategies.....	16-4

16.5	PRESSED POWDER METHODS	16-4
16.5.1	Sample preparation (See also Chapter 2, Section 2.4.2).....	16-4
16.5.2	Calibration strategies.....	16-5
16.6	SILICATE ROCKS AND THE CIPW NORM.....	16-5

Chapter 17: Coal and Coke

17.1	COAL ASH AND METALLURGICAL COKE ASH.....	17-1
17.1.1	Sample preparation.....	17-1
17.1.2	Analyte elements	17-1
17.1.3	Analyte lines, measurement conditions, line overlaps.....	17-1
17.1.3.1	Sequential WDXRF spectrometers	17-1
17.1.3.2	Simultaneous WDXRF spectrometers	17-2
17.1.3.3	EDXRF spectrometers	17-2
17.1.4	Matrix corrections	17-3
17.2	UNASHED (WHOLE) COAL AND COKE.....	17-3
17.2.1	Sample preparation.....	17-3
17.2.2	Infinite thickness	17-3
17.2.2.1	Addition of a heavy absorber	17-4
17.2.2.2	Use of less than infinitely thick samples	17-5
17.2.3	Analyte elements	17-5
17.2.3.1	Analyte lines, line overlaps, and instrumental parameters.....	17-6
17.2.3.2	Sequential WDXRF spectrometers	17-6
17.2.3.3	Major element analysis	17-6
17.2.3.4	Trace element analysis.....	17-7
17.2.3.5	EDXRF spectrometers	17-8
17.2.4	Matrix Corrections.....	17-8
17.2.5	Comparison of infinite and non-infinite thickness methods.....	17-8
17.2.6	Recommended standards	17-9
17.3	ADDITIONAL COMMENTS.....	17-9
17.4	REFERENCES	17-12

Chapter 18: Mineral Sands and Heavy Minerals

18.1	INTRODUCTION	18-1
18.1.1	Instrumental parameters	18-1
18.1.2	WDXRF spectrometers	18-1
18.1.3	EDXRF spectrometers	18-1
18.1.4	Calibration standards.....	18-1
18.2	MINERAL SANDS	18-2
18.2.1	Introduction	18-2
18.2.2	Sample preparation.....	18-2
18.2.3	Analyte elements	18-3
18.2.4	Analyte lines and measurement conditions	18-3
18.2.5	Background correction	18-4
18.2.6	Spectral line overlaps and pulse height selection	18-5
18.2.7	"Line only" calibration and correction for background and line over-	

lap.....	18-6
18.2.8 Matrix correction.....	18-7
18.3 NIOBIUM-, TANTALUM-, TUNGSTEN- AND TIN-BEARING MINERALS	18-7
18.3.1 Introduction.....	18-7
18.3.2 Sample preparation.....	18-9
18.3.3 Analyte elements.....	18-10
18.3.4 Analyte lines and measurement conditions.....	18-10
18.3.5 Background correction.....	18-11
18.3.6 Spectral line overlaps and pulse height selection.....	18-11
18.3.7 Matrix correction.....	18-13
18.3.8 Internal standards.....	18-14
18.4. RARE EARTH ELEMENT MINERALS.....	18-15
18.4.1 Sample preparation.....	18-16
18.4.2 Analyte elements.....	18-17
18.4.3 Analyte lines.....	18-17
18.4.3.1 WDXRF spectrometers.....	18-17
18.4.3.2 EDXRF spectrometers.....	18-18
18.4.4 Background correction.....	18-18
18.4.5 Spectral line overlaps.....	18-18
18.4.6 Pulse height selection.....	18-20
18.4.7 Matrix correction.....	18-22
18.4.8 Calibration standards.....	18-24
18.5 REFERENCES.....	18-24

Chapter 19: Precious metals

19.1 INTRODUCTION.....	19-1
19.2 BULLION AND ALLOYS.....	19-1
19.2.1 Analyte elements.....	19-1
19.2.2 Sample preparation.....	19-1
19.2.3 Analyte lines and measurement conditions.....	19-1
19.2.3.1 WDXRF.....	19-1
19.2.3.2 Matrix correction.....	19-3
19.2.3.3 Background correction.....	19-3
19.2.3.4 EDXRF.....	19-3
19.3 GOLD IN CARBON.....	19-3
19.3.1 Analyte elements.....	19-3
19.3.2 Sample preparation.....	19-3
19.3.3 Analyte lines and measurement conditions.....	19-4
19.3.3.1 WDXRF.....	19-4
19.3.3.2 EDXRF.....	19-4
19.3.3.3 Matrix correction.....	19-5
19.4 GOLD AND PLATINUM IN ORES.....	19-5
19.4.1 WDXRF and EDXRF.....	19-5
19.5 PRECIOUS METALS IN SOLUTION.....	19-6
19.6 PRECIOUS METALS STANDARDS.....	19-6

19.7	Pt, Pd AND Rh IN USED AUTOMOBILE CATALYTIC CONVERTERS	19-6
19.7.1	Sample preparation	19-7
19.7.2	Analyte elements	19-7
19.7.3	Analyte lines and measurement conditions	19-9
19.7.3.1	Sequential WDXRF spectrometers	19-9
19.7.3.2	Simultaneous WDXRF spectrometers	19-9
19.7.3.3	EDXRF spectrometers	19-9
19.7.4	Spectral overlap.....	19-10
19.7.5	Matrix corrections	19-10
19.7.6	Calibration standards.....	19-10
19.8	REFERENCES	19-10

Chapter 20: Sulphide and Oxide ores

20.1	INTRODUCTION	20-1
20.1.1	Instrumental parameters	20-1
20.1.2	Sequential and simultaneous WDXRF spectrometers	20-1
20.1.3	EDXRF spectrometers	20-1
20.1.4	Calibration standards.....	20-2
20.2	BASE METAL SULPHIDE ORES	20-2
20.2.1	Introduction	20-2
20.2.2	Sample preparation.....	20-2
20.2.3	Analyte elements	20-3
20.2.4	Analyte lines and measurement conditions	20-4
20.2.5	Background correction	20-4
20.2.6	Line overlap correction	20-4
20.2.7	Matrix correction.....	20-5
20.3	FERROCHROME AND CHROME ORES	20-6
20.3.1	Sample preparation.....	20-6
20.3.2	Analyte elements	20-7
20.3.3	Analyte lines and measurement conditions	20-7
20.3.4	Line overlap correction	20-7
20.3.5	Matrix corrections	20-8
20.4	MANGANESE ORES, FERROMANGANESE METAL AND ASSOCIATED METALLURGICAL SLAGS	20-8
20.4.1	Introduction	20-8
20.4.2	Manganese ores.....	20-8
20.4.2.1	Sample preparation.....	20-8
20.4.2.2	Analyte elements	20-9
20.4.2.3	Analyte lines and measurement conditions	20-9
20.4.2.4	Spectral overlap correction	20-9
20.4.2.5	Matrix corrections	20-10
20.4.3	Ferromanganese (ferrosilicon and silicomanganese).....	20-10
20.4.3.1	Sample preparation.....	20-10
20.4.3.2	Analyte elements	20-11
20.4.3.3	Analyte lines and measurement conditions	20-11
20.4.3.4	Spectral overlap correction.....	20-12

20.4.3.5	Matrix corrections	20-12
20.4.4	Ferromanganese (ferrosilicon and silicomanganese) slags.....	20-12
20.4.4.1	Sample preparation.....	20-12
20.4.4.2	Analyte elements	20-13
20.4.4.3	Analyte lines and measurement conditions	20-13
20.4.4.4	Line overlap correction	20-13
20.4.4.5	Calibration standards.....	20-13
20.4.4.6	Matrix corrections	20-14
20.5	OXIDISED ZINC ORE	20-14
20.5.1	Sample preparation.....	20-14
20.5.2	Analyte elements	20-14
20.5.3	Analyte lines and measurement conditions	20-15
20.5.4	Line overlap correction	20-15
20.5.5	Matrix correction.....	20-15
20.6	REFERENCES	20-15

Chapter 21: Cements and Carbonates

21.1	INTRODUCTION	21-1
21.2	CEMENTS	21-1
21.2.1	Sample preparation.....	21-1
21.2.2	Analyte elements and comments.....	21-1
21.2.3	Instrumental Parameters	21-2
21.2.3.1	Sequential WDXRF spectrometers	21-2
21.2.3.2	Simultaneous WDXRF spectrometers	21-2
21.2.3.3	EDXRF spectrometers	21-3
21.2.4	Background and Spectral Overlap Corrections.....	21-3
21.2.5	Matrix Corrections.....	21-3
21.2.6	Calibration and Recommended Standards.....	21-3
21.3	CARBONATES.....	21-4
21.3.1	Dolomite.....	21-4
21.3.2	Magnesite	21-4
21.3.3	Limestone.....	21-4
21.3.4	Sample preparation.....	21-4
21.3.5	Analysis conditions.....	21-5
21.3.6	Calibration	21-5

Chapter 22: Aluminium ores and Alumina

22.1	INTRODUCTION	22-1
22.2	ALUMINIUM ORES	22-1
22.2.1	Sample preparation.....	22-1
22.2.2	Analyte elements	22-2
22.2.3	Analyte lines and measurement conditions	22-2
22.2.3.1	Sequential WDXRF spectrometers	22-2
22.2.3.2	Simultaneous WDXRF spectrometers	22-3
22.2.3.3	EDXRF spectrometers	22-4
22.2.4	Calibration and recommended standards.....	22-4

22.2.5	Spectral overlap corrections	22-4
22.2.6	Matrix corrections	22-4
22.3	ALUMINA.....	22-4
22.3.1	Sample preparation.....	22-4
22.3.2	Analyte elements	22-5
22.3.3	Analyte lines and measurement conditions	22-6
22.3.4	Spectral overlap corrections	22-6
22.3.5	Matrix corrections	22-6
22.3.6	Calibration and recommended standards.....	22-7
22.4	REFERENCES	22-7

Chapter 23: Iron Ore and associated Iron- and Steel-making Slags

23.1	INTRODUCTION	23-1
23.2	IRON ORES	23-1
23.2.1	Sample preparation.....	23-1
23.2.2	Analyte elements and lines.....	23-2
23.2.3	Instrumental parameters	23-3
23.2.3.1	Sequential WDXRF spectrometers	23-3
23.2.3.2	Simultaneous WDXRF spectrometers	23-3
23.2.3.3	EDXRF spectrometers	23-4
23.2.4	Calibration and recommended standards.....	23-4
23.2.5	Background and spectral overlap corrections	23-4
23.2.6	Matrix corrections	23-5
23.2.7	"Line only" ISO 9516-1 method	23-5
23.3	MAGNETITE ORES	23-6
23.3.1	Introduction	23-6
23.3.2	Sample preparation.....	23-6
23.3.3	Analyte elements – Vanadium-bearing magnetite iron ores	23-6
23.3.4	Analyte lines and measurement conditions	23-7
23.3.4.1	Sequential WDXRF spectrometers	23-7
23.3.4.2	Simultaneous WDXRF spectrometers	23-7
23.3.4.3	EDXRF spectrometers	23-8
23.3.5	Recommended standards	23-8
23.3.6	Matrix corrections	23-8
23.4	IRON- AND STEEL-MAKING SLAGS.....	23-8
23.4.1	Introduction	23-8
23.4.2	Sample preparation.....	23-9
23.4.3	Analyte elements and lines.....	23-9
23.4.4	Instrumental parameters	23-10
23.4.4.1	Sequential WDXRF spectrometers	23-10
23.4.4.2	Simultaneous WDXRF spectrometers	23-10
23.4.4.3	EDXRF spectrometers	23-11
23.4.5	Recommended standards	23-11
23.4.6	Background and spectral overlap corrections	23-11
23.4.7	Matrix corrections	23-12
23.5	REFERENCES	23-12

Chapter 24: Refractories and Ceramics

24.1	INTRODUCTION	24-1
24.2	SAMPLE PREPARATION	24-1
24.3	ANALYTE ELEMENTS.....	24-2
24.4	REFERENCE	24-2

Chapter 25: Nickel Ores, Laterites, Concentrates and Processing

25.1	INTRODUCTION	25-1
25.2	NICKEL ORES AND LATERITES	25-1
25.2.1	Sample preparation.....	25-1
25.2.1.1	Nickel laterites and sulphides.....	25-1
25.2.2	Analyte elements	25-2
25.2.3	Analyte lines and measurement conditions	25-2
25.2.3.1	Nickel laterites.....	25-3
25.2.3.2	Nickel sulphide ores	25-3
25.2.4	Background and spectral overlap corrections	25-3
25.2.5	Matrix corrections	25-4
25.2.6	Calibration and recommended standards.....	25-4

Chapter 26: Uranium and Thorium ores and "yellow cake"

26.1	INTRODUCTION	26-1
26.2	URANIUM AND THORIUM.....	26-1
26.2.1	Ores.....	26-1
26.2.1.1	Sample preparation.....	26-1
26.2.1.2	Analytes and spectral lines	26-3
26.2.1.3	WDXRF.....	26-3
26.2.1.4	EDXRF.....	26-3
26.2.1.5	Standards.....	26-4
26.2.2	Uranium in solution	26-4
26.2.2.1	Sample preparation.....	26-6
26.2.2.2	Analytes and spectral lines	26-6
26.2.2.3	EDXRF.....	26-7
26.2.2.4	Standards for the analysis of solutions.....	26-7
26.2.3	Uranium in ion exchange resins	26-7
26.2.4	Yellow cake	26-8
26.2.4.1	Sample preparation.....	26-8
26.2.4.2	Analyte elements	26-9
26.2.4.3	Analytes and spectral lines - WDXRF.....	26-9
26.2.4.4	Analytes and spectral lines - EDXRF.....	26-10
26.2.4.5	Standards.....	26-10
26.2.4.6	Matrix Correction	26-10
26.3	REFERENCE	26-12

Chapter 27: Geochemical trace elements

27.1	GEOCHEMICAL TRACE ELEMENTS	27-1
------	----------------------------------	------

27.1.1	Sample preparation	27-1
27.1.2	WDXRF	27-1
27.1.2.1	Analyte elements and lines	27-1
27.1.2.2	Background correction	27-3
27.1.2.3	Line overlap correction	27-3
27.1.2.4	Matrix correction	27-4
27.1.3	EDXRF	27-4
27.1.4	Standards	27-4

Chapter 28: Plastics and Polymers

28.1	INTRODUCTION	28-1
28.2	PLASTICS AND POLYMERS	28-2
28.2.1	Sample preparation	28-2
28.2.2	Analyte elements	28-5
28.2.3	Analyte lines and measurement conditions	28-5
28.2.4	Calibration standards	28-5
28.2.5	Matrix corrections	28-9
28.3	ANALYSIS (CRITICAL) DEPTH	28-11
28.4	REFERENCES	28-12

Chapter 29: Fuels, Oils and Wear metals

29.1	INTRODUCTION	29-1
29.2	OIL	29-1
29.3	DIESEL, MINERAL OIL, GASOLINE AND BIODIESEL	29-2
29.3.1	Sample preparation	29-2
29.3.2	Analyte elements and spectral lines	29-3
29.3.2.1	WDXRF	29-4
29.3.2.2	Analyte lines and measurement conditions	29-4
29.3.2.3	EDXRF	29-5
29.3.2.4	Matrix correction	29-6
29.4	LUBRICATING OILS	29-7
29.4.1	Sample preparation	29-8
29.4.2	Analyte elements	29-8
29.4.2.1	WDXRF	29-8
29.4.2.2	EDXRF	29-9
29.4.2.3	Matrix correction	29-10
29.5	WEAR METALS	29-10
29.5.1	Sample preparation	29-12
29.5.2	Analyte elements	29-12
29.5.3	Matrix correction	29-13
29.6	ADDITIONAL COMMENTS	29-13
29.7	REFERENCES	29-14

Chapter 30: Metals and Alloys

30.1	INTRODUCTION	30-1
------	--------------------	------

30.2	SAMPLE PREPARATION	30-1
30.3	ANALYTE ELEMENTS.....	30-2
30.3.1	Analyte Lines	30-3
30.3.2	Simultaneous WDXRF spectrometers	30-3
30.3.3	Detector saturation	30-3
30.3.4	EDXRF.....	30-5
30.3.5	Matrix correction.....	30-5
30.4	FERROUS METALS	30-6
30.4.1	Stainless steel	30-6
30.4.1.1	Sample preparation.....	30-6
30.4.1.2	Analyte elements	30-6
30.4.2	Low-alloy, ferritic and austenitic steels	30-6
30.4.2.1	Sample preparation.....	30-7
30.4.2.2	Analyte elements	30-7
30.4.3	Nickel - Iron - Cobalt alloys (NiFeCo)	30-8
30.4.3.1	Sample preparation.....	30-8
30.4.3.2	Analyte elements	30-8
30.5	ALUMINIUM	30-8
30.5.1	Sample preparation.....	30-8
30.5.2	Analyte elements	30-8
30.6	TITANIUM.....	30-9
30.6.1	Sample preparation.....	30-9
30.6.2	Analyte elements	30-9
30.7	COPPER-BASED ALLOYS	30-9
30.7.1	Sample preparation.....	30-10
30.7.2	Analyte elements	30-10
30.8	GOLD BULLION	30-10

Chapter 31: Environmental samples

31.1	INTRODUCTION	31-1
31.2	MONITORING AIR QUALITY.....	31-1
31.3	MONITORING OF WATER QUALITY	31-3
31.4	MONITORING OF CONTAMINATED SOILS	31-6
31.5	MONITORING OF BIOLOGICAL MATERIALS.....	31-7
31.6	XRF ANALYSIS OF THIN FILMS	31-7
31.6.1	Calibration	31-8
31.6.2	Line overlap correction	31-11
31.6.3	Analysis of samples.....	31-12
31.7	REFERENCES	31-15

Chapter 32: Tables

32.1	TABLES.....	32-1
32.2	COPYRIGHT PERMISSIONS	32-1
32.3	REFERENCE	32-1
Table 32-1. Wavelengths of the principal K, L and M Series X-ray lines (nm)...		32-2

Table 32-2. Photon energies of the principal K, L and M Series X-ray lines (keV). 32-4

Table 32-3. K, L and M Excitation Potentials (kV) or Binding Energies (keV).... 32-6

Table 32-4. Recommended WDXRF instrumental parameters for the determination of major, minor and trace elements, and 2θ angles for peak and background positions for different analysing crystals..... 32-8

Table 32-5. Analyte elements, analyte lines and possible spectral overlaps..... 32-15

Table 32-6. Energy values for the recommended peak and background positions in Table 32-4..... 32-19

Index

Chapter 28: Plastics and Polymers 28-1

Chapter 29: Fuels, Oils and Wear Metals 29-1

Chapter 30: Environmental Samples 30-1

Chapter 31: Environmental Samples 31-1

Chapter 32: Tables 32-1

Chapter 33: Tables 33-1

Chapter 34: Tables 34-1

Chapter 35: Tables 35-1

Chapter 36: Tables 36-1

Chapter 37: Tables 37-1

Chapter 38: Tables 38-1

Chapter 39: Tables 39-1

Chapter 40: Tables 40-1

Chapter 41: Tables 41-1

Chapter 42: Tables 42-1

Chapter 43: Tables 43-1

Chapter 44: Tables 44-1

Chapter 45: Tables 45-1

Chapter 46: Tables 46-1

Chapter 47: Tables 47-1

Chapter 48: Tables 48-1

Chapter 49: Tables 49-1

Chapter 50: Tables 50-1

Chapter 51: Tables 51-1

Chapter 52: Tables 52-1

Chapter 53: Tables 53-1

Chapter 54: Tables 54-1

Chapter 55: Tables 55-1

Chapter 56: Tables 56-1

Chapter 57: Tables 57-1

Chapter 58: Tables 58-1

Chapter 59: Tables 59-1

Chapter 60: Tables 60-1

Chapter 61: Tables 61-1

Chapter 62: Tables 62-1

Chapter 63: Tables 63-1

Chapter 64: Tables 64-1

Chapter 65: Tables 65-1

Chapter 66: Tables 66-1

Chapter 67: Tables 67-1

Chapter 68: Tables 68-1

Chapter 69: Tables 69-1

Chapter 70: Tables 70-1

Chapter 71: Tables 71-1

Chapter 72: Tables 72-1

Chapter 73: Tables 73-1

Chapter 74: Tables 74-1

Chapter 75: Tables 75-1

Chapter 76: Tables 76-1

Chapter 77: Tables 77-1

Chapter 78: Tables 78-1

Chapter 79: Tables 79-1

Chapter 80: Tables 80-1

Chapter 81: Tables 81-1

Chapter 82: Tables 82-1

Chapter 83: Tables 83-1

Chapter 84: Tables 84-1

Chapter 85: Tables 85-1

Chapter 86: Tables 86-1

Chapter 87: Tables 87-1

Chapter 88: Tables 88-1

Chapter 89: Tables 89-1

Chapter 90: Tables 90-1

Chapter 91: Tables 91-1

Chapter 92: Tables 92-1

Chapter 93: Tables 93-1

Chapter 94: Tables 94-1

Chapter 95: Tables 95-1

Chapter 96: Tables 96-1

Chapter 97: Tables 97-1

Chapter 98: Tables 98-1

Chapter 99: Tables 99-1

Chapter 100: Tables 100-1