

Handbook of Exploration Geochemistry

# VOLUME 1

## Analytical Methods in Geochemical Prospecting

by

W.K. FLETCHER

*Associate Professor*

*Department of Geological Sciences*

*University of British Columbia, Vancouver, B.C., Canada*



ELSEVIER SCIENCE PUBLISHERS B.V.  
Amsterdam — Oxford — New York 1981



# CONTENTS

Editor's Foreword .....	V
Preface .....	IX
<b>CHAPTER 1. INTRODUCTION .....</b>	<b>1</b>
Analysis of exploration samples: choice of methods .....	5
Geochemical considerations .....	5
Analytical and organizational considerations .....	10
Reporting results .....	21
Safety .....	23
<b>CHAPTER 2. QUALITY CONTROL IN THE LABORATORY .....</b>	<b>25</b>
Introduction .....	25
Random errors .....	27
Precision .....	27
Detection limits .....	30
Sources of and reduction of random errors .....	32
Laboratory sampling .....	34
Systematic errors .....	41
Contamination .....	42
Drift .....	42
Physical and chemical interferences .....	42
Control and monitoring of systematic errors .....	42
Accuracy .....	45
A quality control programme .....	46
<b>CHAPTER 3. SAMPLE PREPARATION .....</b>	<b>47</b>
Introduction .....	47
Preparation of rock samples .....	48
Soils and sediments .....	50
Vegetation .....	52
Waters .....	52
<b>CHAPTER 4. SAMPLE DECOMPOSITION-SOLUTION TECHNIQUES .....</b>	<b>57</b>
Introduction .....	57
Strong decompositions .....	58

Acid digestion . . . . .	59
Fusions . . . . .	66
Partial extractions . . . . .	71
Application to bedrock samples . . . . .	71
Application to soils and sediments . . . . .	75
Summary . . . . .	94
 CHAPTER 5. COLORIMETRY AND RELATED TECHNIQUES . . . . .	 97
Introduction . . . . .	97
Colorimetry . . . . .	98
Fluorimetry . . . . .	106
Turbidimetric methods . . . . .	108
 CHAPTER 6. ATOMIC ABSORPTION SPECTROPHOTOMETRY . . . . .	 109
Introduction . . . . .	109
Theory . . . . .	109
Instrumentation . . . . .	112
The light source . . . . .	114
Production of atomic vapour . . . . .	116
Wavelength selection . . . . .	120
Detection and readout systems . . . . .	120
Applications . . . . .	121
Flame atomic absorption . . . . .	122
Electrothermal atomization . . . . .	131
Hydride generation . . . . .	132
Determination of mercury by flameless AAS . . . . .	134
Indirect determinations of elements by AAS . . . . .	136
 CHAPTER 7. EMISSION SPECTROSCOPY . . . . .	 139
Introduction . . . . .	139
Theory . . . . .	140
Flame emission spectroscopy . . . . .	141
Semi-quantitative DC-arc spectroscopy . . . . .	142
Equipment . . . . .	142
Operating conditions . . . . .	145
Standards for semi-quantitative DC-arc spectroscopy . . . . .	148
Analysis of unknowns . . . . .	151
Direct-reading DC-arc spectrometry . . . . .	152
Matrix effects . . . . .	154
Background . . . . .	156
Spectral interferences . . . . .	157
Plasma sources . . . . .	157
The inductively coupled plasma . . . . .	158
Analysis of geochemical samples . . . . .	163
 CHAPTER 8. X-RAY FLUORESCENCE . . . . .	 167
Introduction . . . . .	167
Theory . . . . .	168
Excitation of X-rays . . . . .	168

Interaction of X-rays with matter . . . . .	171
Instrumentation . . . . .	173
Primary radiation sources . . . . .	175
Dispersion of X-rays . . . . .	176
Operation of the X-ray fluorescence spectrometer . . . . .	182
Instrument conditions . . . . .	182
Counting strategy . . . . .	184
Analysis of geochemical samples . . . . .	186
Sample preparation . . . . .	188
Matrix corrections . . . . .	189
Line interferences . . . . .	196
Summary . . . . .	196
 CHAPTER 9. ELECTROCHEMICAL METHODS . . . . .	 199
Introduction . . . . .	199
Theory . . . . .	200
Determination of pH . . . . .	203
Specific ion electrodes . . . . .	205
Fluoride . . . . .	208
Chloride . . . . .	209
Iodide . . . . .	209
Copper . . . . .	210
Boron . . . . .	211
Appendix 1 — Colour photographs . . . . .	213
Appendix 2 — Preparation of standards . . . . .	214
 References . . . . .	 217
Subject index . . . . .	241