

Contents

Preface — V

Contributors — XIII

Henryk Matusiewicz and Ewa Bulska

Trace Elements in Environmental, Biological and Industrial Samples — 1

Part I: Methodology in Trace Element Determination

Henryk Matusiewicz

1 Sample Preparation for Inorganic Trace Element Analysis — 5

1.1 Introduction — 5

1.2 Aspects of sampling and sample preservation — 5

1.2.1 Sample — 7

1.2.2 Specimen — 7

1.2.3 Random sampling — 7

1.2.4 Systematic sampling — 8

1.2.5 Representative sampling — 8

1.2.6 Composite sampling — 8

1.2.7 Subsampling — 9

1.3 Error sources during the analytical procedure — 11

1.3.1 Blank — 13

1.3.2 Contamination — 13

1.3.3 Reagents — 15

1.3.4 Materials — 18

1.3.5 Contamination by sample handling — 20

1.3.6 Losses — 20

1.4 Sample treatment after the sampling process — 22

1.5 Decomposition as a sample preparation method for elemental analysis: an analytical perspective — 25

1.5.1 Sample decomposition techniques — 26

1.6 Conclusions and future trends — 54

Funding — 57

References — 57

Ewa Bulska and Anna Ruszczyńska

2 Analytical Techniques for Trace Element Determination — 69

2.1 Introduction — 69

2.2 Spectroscopic techniques — 70

2.2.1 Atomic absorption spectrometry — 70

2.2.2 Inductively coupled plasma atomic emission spectrometry — 75

2.2.3 Inductively coupled plasma mass spectrometry — 76

2.2.4 Laser ablation ICPMS — 78

- 2.3 Electrochemical techniques — **80**
- 2.4 Other techniques used for trace analysis — **83**
- 2.4.1 Techniques uses X-ray — **83**
- 2.4.2 Activation analysis — **84**
- References — **84**

Part II: Matrices – Selected applications

Emilia Vassileva and Petko Mandjukov

- 3 Trace Elements in the Environment — 95**
- 3.1 General aspects of environmental analysis — **95**
- 3.2 Trace elements (general information, ecological importance, common species in the nature) — **97**
- 3.2.1 General information on trace elements — **100**
- 3.3 Environmental monitoring — **115**
- 3.4 Determination of trace elements in environmental samples — **118**
- 3.5 Environmental matrices – general characteristics and analytical aspects — **119**
- 3.5.1 Natural waters — **119**
- 3.5.2 Soils — **124**
- 3.5.3 Sediments — **126**
- 3.5.4 Air, airborne particles — **130**
- 3.6 Speciation analysis in the environment samples — **132**
- 3.6.1 Types of chromatographic techniques used in speciation analysis — **133**
- 3.7 Isotopic analysis — **145**
- 3.7.1 Use the isotope ratios as a tool for investigation of environmental pollution — **145**
- 3.8 Metrological aspects of environmental monitoring of trace elements — **147**
- 3.8.1 Use of reference materials — **148**
- 3.8.2 Measurement uncertainty in the environment analysis: study case — **149**
- 3.8.3 Practical aspects on the traceability of trace elements in environmental samples — **158**
- References — **159**

Katarzyna Wrobel, Kazimierz Wrobel, Alma Rosa Corrales Escobosa and Armando Gómez Ojeda

- 4 Food Analysis and Speciation — 178**
- 4.1 Topics, elements and species of interest — **178**
- 4.2 Determination of total elements — **181**
- 4.3 Speciation schemes and analysis of selected food matrices — **203**
- 4.3.1 Mercury — **207**
- 4.3.2 Arsenic — **240**

- 4.3.3 Selenium — 242
- 4.3.4 Other elements — 244
- 4.4 Future perspectives in food analysis and speciation — 245
- Acknowledgments — 246
- References — 247

Anna Ruszczyńska and Eliza Kurek

- 5 Trace Element and Speciation Analysis of Biological Samples — 261**
 - 5.1 Trace element analysis of biological samples — 261
 - 5.1.1 Sampling, sample storage and sampling treatment — 262
 - 5.2 Element speciation of biological samples — 265
 - 5.2.1 Sampling, sample storage and sample treatment — 266
 - 5.3 Elements — 267
 - References — 270

Wiktor Dmitruk and Zuzanna Brożek-Mucha

- 6 Forensic Analysis of Microtraces — 276**
 - 6.1 Characterisation of forensic microtraces — 276
 - 6.2 Analytical methodology — 276
 - 6.3 Evidence materials — 278
 - 6.3.1 GSR – inorganic gunshot residue — 278
 - 6.3.2 Airbag deployment residues — 285
 - 6.3.3 Paint — 286
 - 6.3.4 Glass — 288
 - 6.3.5 Evidential materials – summary — 298
 - References — 299

Andrzej Wyciślik

- 7 Industrial Analysis and Speciation — 302**
 - 7.1 Introduction — 302
 - 7.2 A brief historical overview of the development of metallurgical analytics — 304
 - 7.3 Fields of application of the analytical techniques in metallurgy — 307
 - 7.4 Range of materials — 307
 - 7.5 Developments in metallurgical analytics — 308
 - 7.6 Determination of chemical composition of metal alloys and other industrial materials — 310
 - 7.7 The concept of speciation in relation to metallurgy and materials engineering — 311
 - 7.8 Chemical and electrochemical phase extraction — 312
 - 7.8.1 Chemical phase extraction — 312
 - 7.8.2 Electrochemical phase extraction — 313

- 7.9 Techniques of testing isolates — **316**
- 7.10 Determination of chemical composition of isolates – speciation analysis — **318**
- 7.11 AAS in metallurgy — **319**
- 7.12 Flow injection technique combined with flame atomic absorption method — **320**
- 7.13 Application of alternative analytical lines — **324**
- 7.14 Calibration methods used in FAAS, including methods based on one reference standard — **327**
- 7.15 Determination of aluminium in steel as a typical example of speciation and speciation analysis in metallurgy — **330**
- 7.16 Tool alloys – speciation and speciation analysis – determination of chemical composition of carbide phase isolates — **332**
- 7.17 Speciation and speciation analysis in multicomponent nickel-based alloys — **343**
- 7.18 Studies of speciation in creep-resistant Fe–Ni alloys — **352**
- 7.19 Introduction to speciation and speciation analysis of chromium in welding dust — **358**
- 7.19.1 Description and characteristic of laboratory stands — **362**
- 7.19.2 Methodology of total dust emission determination — **362**
- 7.19.3 Methodology of drawing of dust samples for chemical analysis — **365**
- 7.20 Summary — **366**
- References — **367**

Part III: Inorganic and Bioinorganic Speciation Analysis at Trace Level

Ewa Bulska

- 8 Quality of Results in Trace Element and Speciation Analysis — 377**
- 8.1 Introduction — **377**
- 8.2 General aspects of QA and QC — **378**
- 8.3 Validation of analytical procedure — **379**
- 8.4 Traceability of analytical results — **380**
- 8.4.1 Reference materials — **381**
- 8.5 Monitoring of the quality of analytical results — **385**
- 8.6 Conclusions — **388**
- References — **389**

Beata Krasnodębska-Ostręga, Monika Sadowska and Ewa Biaduń

- 9 Sample Pretreatment for Trace Speciation Analysis — 392**
- 9.1 Introduction — **392**
- 9.2 Sampling and sample transport — **394**
- 9.2.1 Selection of vessels — **395**

9.2.2	Contamination of the sample with various substances —	396
9.2.3	Elimination of UV-Vis irradiation —	396
9.2.4	Oxidation and desorption of carbon dioxide —	397
9.2.5	Solid sample for fractionation study —	398
9.2.6	Temperature lowering just after sampling —	398
9.3	Sampling with some pretreatment on sampling site —	399
9.3.1	Suspended matter separation – fractionation in water —	400
9.3.2	Chemical modification of the sample —	401
9.4	Short- and long-term storage —	402
9.4.1	Dehydration of samples —	402
9.5	Extraction as a method of sample pretreatment for speciation analysis —	404
9.5.1	SPE – water analysis —	406
9.5.2	Extraction in fractionation study —	406
9.6	Conclusion —	408
	References —	408

Pawel Pohl, Anna Szymczycha-Madeja, Maja Welna and Piotr Jamroz

10	Solid-Phase Extraction in Fractionation of Trace Elements —	419
10.1	Background —	419
10.2	Operationally defined fractionation of elements through SPE —	421
10.2.1	Unary and binary fractionations using one-column SPE approaches —	421
10.2.2	Binary and tertiary fractionations using two-column SPE approaches —	427
10.2.3	Characterization of sorbents —	432
10.3	QA and QC of the chemical fractionation analysis by the SPE —	433
10.4	Conclusions —	433
	Acknowledgments —	434
	References —	434

Index —	437
----------------	------------