

Contents

| | |
|---|-----|
| Preface | xix |
| Foreword by Herbert Muntau | xxi |
| Abbreviations | xxv |
| CHAPTER 1. GENERAL INTRODUCTION | |
| 1.1 The analyst and his duty | 1 |
| 1.2 The necessity to deliver good answers | 3 |
| 1.3 The necessity to produce reliable data | 3 |
| 1.4 The regulatory background of quality | 5 |
| 1.4.1 GLP | 6 |
| 1.4.2 Certification ISO 9000 | 6 |
| 1.4.3 Certification ISO 14000 | 7 |
| 1.4.4 Accreditation ISO 25/EN 45000 | 8 |
| 1.4.5 International bodies for the harmonisation of standards | 8 |
| 1.4.6 Conclusions | 9 |
| 1.5 References | 10 |
| CHAPTER 2. QUALITY IN CHEMICAL AND BIOLOGICAL ANALYSIS | |
| 2.1 Tools to achieve quality | 13 |
| 2.1.1 Terms and definitions | 13 |
| 2.1.1.1 Basic definitions | 13 |
| 2.1.1.2 Trivial terms | 14 |
| 2.1.1.2 Hierarchy of methods | 15 |
| 2.1.1.3 Calibration linked classification of methods | 16 |
| 2.1.1.4 Performance criteria of methods | 17 |
| 2.2 Tools for QA and QC of biological and chemical measurements | 18 |
| 2.2.1 First line checks | 19 |
| 2.2.2 Second line checks | 19 |
| 2.2.3 Third line checks | 20 |
| 2.3 Validation of methods | 20 |
| 2.3.1 Validation of instruments, computers and software | 21 |
| 2.3.2 Validation of the analytical method | 21 |
| 2.3.2.1 Full development of a method | 22 |
| 2.3.2.2 Validating a standard procedure | 22 |
| 2.3.2.3 Practical examples of method development and validation | 25 |
| 2.3.3 Quality control of microbiological determinations | 42 |
| 2.3.3.1 Microbiological methods | 42 |
| 2.3.3.2 Accuracy and precision of microbiological tests | 47 |

| | | | |
|-----|---------|---|----|
| | 2.3.3.3 | Validation of microbiological methods | 48 |
| | 2.3.3.4 | Quality assurance and standardisation | 48 |
| 2.4 | | Control charts | 49 |
| | 2.4.1 | Shewart charts | 50 |
| | 2.4.2 | Other control charts | 50 |
| | 2.4.3 | Microbiology | 51 |
| | 2.4.3.1 | High levels counts | 52 |
| | 2.4.3.2 | Presence-absence reference materials | 56 |
| 2.5 | | Where and when to use RMs and CRMs | 57 |
| | 2.5.1 | Definitions by ISO | 60 |
| | 2.5.2 | Calibration and traceability: | 60 |
| | 2.5.3 | Method validation | 62 |
| | 2.5.4 | Statistical control | 66 |
| | 2.5.5 | Microbiology | 66 |
| | 2.5.6 | Conclusions | 68 |
| 2.6 | | References | 68 |

CHAPTER 3. USE OF CERTIFIED REFERENCE MATERIALS

| | | | |
|-----|---------|---|----|
| 3.1 | | General aspects | 71 |
| | 3.1.1 | Structural role | 71 |
| | 3.1.2 | Practical role | 72 |
| 3.2 | | Types of CRMs | 73 |
| | 3.2.1 | Calibration materials | 73 |
| | 3.2.1.1 | Pure substances | 73 |
| | 3.2.1.2 | Synthetic mixtures | 76 |
| | 3.2.1.3 | Natural materials | 77 |
| | 3.2.1.4 | When using certified calibrants | 77 |
| | 3.2.2 | Method validation materials | 78 |
| | 3.2.2.1 | Choice of the CRM | 78 |
| | 3.2.2.2 | Validation of the precision of a method | 79 |
| | 3.2.2.3 | Validation of the trueness of a method | 82 |
| | 3.2.3 | Frequency of use | 84 |
| 3.3 | | Validation of standardised regulatory methods | 85 |
| 3.4 | | Comparison of methods and instruments | 85 |
| 3.5 | | Evaluation of non-certified reference materials | 86 |
| 3.6 | | CRM for microbiological methods | 86 |
| | 3.6.1 | Types of microbiology RM | 86 |
| | 3.6.2 | Use of BCR-CRMs in microbiology | 87 |
| | 3.6.3 | CRMs with high levels of cfp | 87 |
| | 3.6.3.1 | The number of capsules to be used | 89 |
| | 3.6.3.2 | The number of replicates to be used | 90 |
| | 3.6.3.3 | Practical results | 91 |
| | 3.6.4 | Low level CRMs | 93 |
| | 3.6.4.1 | Problem description | 93 |
| | 3.6.4.2 | Theory of testing | 94 |
| | 3.6.4.3 | The number of capsules to be used | 95 |
| | 3.6.4.4 | Practical results | 96 |

| | | |
|-------|------------------------|----|
| 3.6.5 | Frequency of use | 97 |
| 3.6.6 | Conclusions | 97 |
| 3.7 | References | 98 |

CHAPTER 4. PRODUCTION OF MATRIX CRMs

| | | |
|---------|--|-----|
| 4.1 | General considerations and warnings | 115 |
| 4.2 | Preparation of the material | 115 |
| 4.2.1 | Choice of the material | 115 |
| 4.2.1.1 | Analytes and their concentrations | 116 |
| 4.2.1.2 | The matrix of the material | 117 |
| 4.2.1.3 | The physical status | 117 |
| 4.2.1.4 | Limits to representativeness | 118 |
| 4.2.1.5 | Artificial matrix materials | 118 |
| 4.2.1.6 | Fresh materials | 118 |
| 4.2.1.7 | Series of materials | 119 |
| 4.2.2 | Stabilisation of the materials | 119 |
| 4.2.2.1 | Physical and chemical effects | 120 |
| 4.2.2.2 | Microbial activity | 120 |
| 4.2.2.3 | When to stabilise the material | 122 |
| 4.2.2.4 | Limits to stabilisation | 124 |
| 4.2.3 | Homogenisation | 124 |
| 4.2.3.1 | Necessity for homogeneous samples | 124 |
| 4.2.3.2 | Homogenisation of fluids | 125 |
| 4.2.3.3 | Homogenisation of solids | 125 |
| 4.2.4 | Packaging and storage | 128 |
| 4.2.4.1 | Packaging material and vial | 128 |
| 4.2.4.2 | Temperature of storage | 134 |
| 4.2.4.3 | Safety and information | 134 |
| 4.2.5 | Size of batch | 137 |
| 4.2.6 | Transport | 137 |
| 4.3 | Study of the homogeneity | 138 |
| 4.3.1 | Principle | 138 |
| 4.3.1.1 | The test method | 138 |
| 4.3.1.2 | Conclusions and limits of the study | 141 |
| 4.3.1.3 | Extrapolations | 142 |
| 4.3.2 | Within-vial homogeneity study | 142 |
| 4.3.2.1 | Objectives and limits | 142 |
| 4.3.2.2 | Testing for the within-vial homogeneity | 143 |
| 4.3.3 | Between-vial homogeneity study | 145 |
| 4.3.3.1 | Principle and objective | 145 |
| 4.3.3.2 | Testing for the between-vial homogeneity | 145 |
| 4.3.3.3 | Practical implementation | 146 |
| 4.3.4 | Between-vial check through interlaboratory study | 151 |
| 4.3.5 | Conclusions | 152 |
| 4.4 | Study of the stability | 154 |
| 4.4.1 | Objectives | 154 |
| 4.4.2 | Multiple temperature approach | 155 |
| 4.4.3 | Analytical methods used for the study of stability | 156 |

| | | |
|-------|--|-----|
| 4.4.4 | Isochronous studies | 157 |
| 4.4.5 | Extrapolations through Arrhenius law | 159 |
| 4.4.6 | Expiry dates | 161 |
| 4.4.7 | Short-term studies and conclusions | 163 |
| 4.5 | References | 164 |

CHAPTER 5. PROCEDURES TO CERTIFY REFERENCE MATERIALS

| | | |
|---------|--|-----|
| 5.1 | Absolute amounts | 168 |
| 5.1.1 | Pure substances | 168 |
| 5.1.1.1 | Inorganic materials | 168 |
| 5.1.1.2 | Organic and organo-metallic substances | 169 |
| 5.1.2 | Mixtures of substances | 171 |
| 5.1.3 | Calibration solutions | 171 |
| 5.1.4 | Matrix materials | 171 |
| 5.1.4.1 | Single laboratory approach | 172 |
| 5.1.4.2 | Interlaboratory certification study | 173 |
| 5.1.4.3 | The BCR approach | 173 |
| 5.2 | Method-dependent parameters | 181 |
| 5.2.1 | Chemical status | 182 |
| 5.2.2 | Physical status | 182 |
| 5.2.3 | Activity-linked fractions | 182 |
| 5.2.4 | CRM for testing | 183 |
| 5.3 | Certification of CRM for microbiology | 183 |
| 5.3.1 | Types of materials | 183 |
| 5.3.2 | Stabilisation of microbes | 184 |
| 5.3.3 | Homogeneity tests | 185 |
| 5.3.4 | Stability tests | 185 |
| 5.3.5 | Certification by interlaboratory studies | 187 |
| 5.4 | Certificate and certification report | 189 |
| 5.4.1 | Information content | 189 |
| 5.4.2 | Responsibility of the producer | 190 |
| 5.5 | Producers and production systems | 190 |
| 5.5.1 | Producers | 190 |
| 5.5.2 | Production systems | 191 |
| 5.6 | Driving forces for the future | 191 |
| 5.6.1 | Salient features of the inquiry | 198 |
| 5.6.2 | Needs and recommendations | 200 |
| 5.6.2.1 | Needs for reference materials | 200 |
| 5.6.2.2 | Representativeness | 205 |
| 5.6.2.3 | Development and CRM production | 206 |
| 5.6.2.4 | Interlaboratory trials | 206 |
| 5.6.2.5 | Information/Education/Training | 207 |
| 5.6.2.6 | Networking institutes/Databases | 207 |
| 5.6.3 | Summary of needs | 208 |
| 5.6.4 | Expected trends | 209 |
| 5.6.5 | Conclusions | 210 |
| 5.7 | References | 210 |

CHAPTER 6. CRMs FOR PLANT ANALYSIS

| | | |
|-------|---|-----|
| 6.1 | Trace elements in aquatic plants | 235 |
| 6.1.1 | Introduction | 235 |
| 6.1.2 | Production of the materials | 235 |
| 6.1.3 | Certification | 236 |
| 6.1.4 | Participating laboratories | 237 |
| 6.2 | Trace elements in sea lettuce | 239 |
| 6.2.1 | Introduction | 239 |
| 6.2.2 | Production of the material | 239 |
| 6.2.3 | Certification | 240 |
| 6.2.4 | Participating laboratories | 242 |
| 6.3 | Trace elements in rye grass | 243 |
| 6.3.1 | Introduction | 243 |
| 6.3.2 | Production of the material | 243 |
| 6.3.3 | Certification | 245 |
| 6.3.4 | Participating laboratories | 246 |
| 6.4 | Trace elements in hay powder | 248 |
| 6.4.1 | Introduction | 248 |
| 6.4.2 | Production of the material | 248 |
| 6.4.3 | Certification | 249 |
| 6.4.4 | Participating laboratories | 250 |
| 6.5 | Trace elements in white clover | 251 |
| 6.5.1 | Introduction | 251 |
| 6.5.2 | Production of the material | 252 |
| 6.5.3 | Certification | 252 |
| 6.5.4 | Participating laboratories | 253 |
| 6.6 | Trace elements in beech leaves and spruce needles | 255 |
| 6.6.1 | Introduction | 255 |
| 6.6.2 | Production of the materials | 255 |
| 6.6.3 | Certification | 256 |
| 6.6.4 | Participating laboratories | 257 |
| 6.7 | Trace elements in lichen | 258 |
| 6.7.1 | Introduction | 258 |
| 6.7.2 | Interlaboratory study | 259 |
| 6.7.3 | Production of the material | 259 |
| 6.7.4 | Certification | 260 |
| 6.7.5 | Participating laboratories | 262 |
| 6.8 | References | 264 |

CHAPTER 7. CRMs FOR BIOLOGICAL MATERIAL ANALYSIS

| | | |
|-------|-----------------------------------|-----|
| 7.1 | Trace elements in fish | 265 |
| 7.1.1 | Introduction | 265 |
| 7.1.2 | Production of the material | 265 |
| 7.1.3 | Certification | 266 |
| 7.1.4 | Participating laboratories | 267 |
| 7.2 | Methyl mercury in fish | 269 |
| 7.2.1 | Introduction | 269 |
| 7.2.2 | Production of the materials | 270 |

| | | | |
|------|--------|-----------------------------------|-----|
| | 7.2.3 | Certification | 271 |
| | 7.2.4 | Participating laboratories | 272 |
| 7.3 | | As-species in fish | 273 |
| | 7.3.1 | Introduction | 273 |
| | 7.3.2 | Production of the material | 273 |
| | 7.3.3 | Certification | 274 |
| | 7.3.4 | Participating laboratories | 275 |
| 7.4 | | CBs in fish oil | 275 |
| | 7.4.1 | Introduction | 275 |
| | 7.4.2 | Interlaboratory studies | 276 |
| | 7.4.3 | Production of the materials | 277 |
| | 7.4.4 | Certification | 278 |
| | 7.4.5 | Participating laboratories | 279 |
| 7.5 | | OCPs in cod liver oil | 280 |
| | 7.5.1 | Introduction | 280 |
| | 7.5.2 | Production of the material | 280 |
| | 7.5.3 | Certification | 282 |
| | 7.5.4 | Participating laboratories | 283 |
| 7.6 | | Trace elements in mussel | 284 |
| | 7.6.1 | Introduction | 284 |
| | 7.6.2 | Production of the material | 284 |
| | 7.6.3 | Certification | 285 |
| | 7.6.4 | Participating laboratories | 286 |
| 7.7 | | Organotins in mussel | 286 |
| | 7.7.1 | Introduction | 286 |
| | 7.7.2 | Production of the material | 288 |
| | 7.7.3 | Certification | 290 |
| | 7.7.4 | Participating laboratories | 292 |
| 7.8 | | Trace elements in plankton | 292 |
| | 7.8.1 | Introduction | 292 |
| | 7.8.2 | Production of the material | 292 |
| | 7.8.3 | Certification | 294 |
| | 7.8.4 | Participating laboratories | 295 |
| 7.9 | | Pesticides in animal fat | 296 |
| | 7.9.1 | Introduction | 296 |
| | 7.9.2 | Production of the material | 296 |
| | 7.9.3 | Certification | 297 |
| | 7.9.4 | Participating laboratories | 298 |
| 7.10 | | Trace elements in milk | 299 |
| | 7.10.1 | Introduction | 299 |
| | 7.10.2 | Production of the material | 299 |
| | 7.10.3 | Certification | 300 |
| | 7.10.4 | Participating laboratories | 302 |
| 7.11 | | CBs in milk | 303 |
| | 7.11.1 | Introduction | 303 |
| | 7.11.2 | Production of the material | 304 |
| | 7.11.3 | Certification | 304 |
| | 7.11.4 | Participating laboratories | 306 |
| 7.12 | | Dioxins in milk | 307 |

| | | |
|--------|--|-----|
| 7.12.1 | Introduction | 307 |
| 7.12.2 | Production of the material | 307 |
| 7.12.3 | Certification | 308 |
| 7.12.4 | Participating laboratories | 310 |
| 7.13 | Microbiological parameters in milk | 310 |
| 7.13.1 | Introduction | 310 |
| 7.13.2 | Production of the materials | 310 |
| | 7.13.2.1 Milk containing <i>Salmonella</i> | 310 |
| | 7.13.2.2 Milk containing <i>Enterococcus</i> | 311 |
| 7.13.3 | Certification | 313 |
| | 7.13.3.1 Milk containing <i>Salmonella</i> | 313 |
| | 7.13.3.2 Milk containing <i>Enterococcus</i> | 314 |
| 7.13.4 | Participating laboratories | 315 |
| 7.14 | Trace elements in human hair | 316 |
| 7.14.1 | Introduction | 316 |
| 7.14.2 | Production of the material | 317 |
| 7.14.3 | Certification | 317 |
| 7.14.4 | Participating laboratories | 319 |
| 7.15 | References | 319 |

CHAPTER 8. CRMs FOR WATER ANALYSIS

| | | |
|-------|-------------------------------------|-----|
| 8.1 | Major elements in freshwater | 321 |
| 8.1.1 | Introduction | 321 |
| 8.1.2 | Production of the materials | 321 |
| 8.1.3 | Certification | 322 |
| 8.1.4 | Participating laboratories | 323 |
| 8.2 | Nitrate in freshwater | 324 |
| 8.2.1 | Introduction | 324 |
| 8.2.2 | Feasibility study | 324 |
| 8.2.3 | Production of the materials | 327 |
| 8.2.4 | Certification | 328 |
| 8.2.5 | Participating laboratories | 329 |
| 8.3 | Major elements in rainwater | 329 |
| 8.3.1 | Introduction | 329 |
| 8.3.2 | Production of the materials | 329 |
| 8.3.3 | Certification | 331 |
| 8.3.4 | Participating laboratories | 332 |
| 8.4 | Trimethyllead in rainwater | 333 |
| 8.4.1 | Introduction | 333 |
| 8.4.2 | Feasibility study | 334 |
| 8.4.3 | Interlaboratory studies | 335 |
| 8.4.4 | Production of the materials | 336 |
| 8.4.5 | Certification | 338 |
| 8.4.6 | Participating laboratories | 340 |
| 8.5 | Major elements in groundwater | 340 |
| 8.5.1 | Introduction | 340 |

| | | |
|--------|--|-----|
| 8.5.2 | Feasibility study | 340 |
| 8.5.3 | Production of the materials | 341 |
| 8.5.4 | Certification | 342 |
| 8.5.5 | Participating laboratories | 343 |
| 8.6 | Trace elements in groundwater | 344 |
| 8.6.1 | Introduction | 344 |
| 8.6.2 | Interlaboratory study | 345 |
| 8.6.3 | Production of the materials | 346 |
| | 8.6.3.1 CRMs for trace elements | 346 |
| | 8.6.3.2 CRMs for bromide | 348 |
| 8.6.4 | Certification | 348 |
| 8.6.5 | Participating laboratories | 350 |
| 8.7 | Trace elements in estuarine water | 351 |
| 8.7.1 | Introduction | 351 |
| 8.7.2 | Feasibility study | 352 |
| 8.7.3 | Interlaboratory studies | 352 |
| 8.7.4 | Production of the material | 356 |
| 8.7.5 | Certification | 357 |
| 8.7.6 | Participating laboratories | 359 |
| 8.8 | Trace elements in seawater | 360 |
| 8.8.1 | Introduction | 360 |
| 8.8.2 | Interlaboratory studies | 360 |
| 8.8.3 | Production of the material | 361 |
| 8.8.4 | Certification | 363 |
| 8.8.5 | Some remarks on the analysis of the material | 363 |
| 8.8.6 | Participating laboratories | 364 |
| 8.9 | Mercury in seawater | 365 |
| 8.9.1 | Introduction | 365 |
| 8.9.2 | Feasibility study | 365 |
| 8.9.3 | Interlaboratory studies | 366 |
| 8.9.4 | Preliminary work | 366 |
| 8.9.5 | Production of the material | 367 |
| 8.9.6 | Certification | 369 |
| 8.9.7 | Participating laboratories | 370 |
| 8.10 | Cr-species in lyophilised water | 371 |
| 8.10.1 | Introduction | 371 |
| 8.10.2 | Preliminary investigations | 371 |
| 8.10.3 | Interlaboratory study | 372 |
| 8.10.4 | Production of the material | 373 |
| 8.10.5 | Certification | 374 |
| 8.10.6 | Participating laboratories | 375 |
| 8.11 | Se-species in solution | 375 |
| 8.11.1 | Introduction | 375 |
| 8.11.2 | Preliminary investigations | 376 |
| 8.11.3 | Interlaboratory study | 378 |
| 8.11.4 | Production of the materials | 378 |
| 8.11.5 | Certification | 380 |
| 8.11.6 | Participating laboratories | 381 |
| 8.12 | Unstable pesticides in lyophilised water | 382 |

| | | |
|--------|----------------------------------|-----|
| 8.12.1 | Introduction | 382 |
| 8.12.2 | Production of the material | 383 |
| 8.12.3 | Certification | 384 |
| 8.12.4 | Participating laboratories | 385 |
| 8.13 | References | 385 |

CHAPTER 9. CRMs FOR SEDIMENT ANALYSIS

| | | |
|-------|--|-----|
| 9.1 | Trace elements in river and lake sediment | 389 |
| 9.1.1 | Introduction | 389 |
| 9.1.2 | Production of the materials | 389 |
| 9.1.3 | Certification | 390 |
| 9.1.4 | Participating laboratories | 393 |
| 9.2 | Fluoride in clay | 393 |
| 9.2.1 | Introduction | 393 |
| 9.2.2 | Production of the material | 395 |
| 9.2.3 | Certification | 395 |
| 9.2.4 | Participating laboratories | 396 |
| 9.3 | Organotins in estuarine sediment | 396 |
| 9.3.1 | Introduction | 396 |
| 9.3.2 | Preliminary investigations | 396 |
| 9.3.3 | Production of the material | 397 |
| 9.3.4 | Certification | 399 |
| 9.3.5 | Further developments | 402 |
| 9.3.6 | Participating laboratories | 402 |
| 9.4 | Methyl mercury in estuarine sediment | 402 |
| 9.4.1 | Introduction | 402 |
| 9.4.2 | Interlaboratory study | 403 |
| 9.4.3 | Production of the material | 403 |
| 9.4.4 | Certification | 404 |
| 9.4.5 | Participating laboratories | 407 |
| 9.5 | Extractable trace elements in river sediment | 408 |
| 9.5.1 | Introduction | 408 |
| 9.5.2 | Interlaboratory study | 408 |
| 9.5.3 | Production of the material | 410 |
| 9.5.4 | Certification | 410 |
| 9.5.5 | Participating laboratories | 411 |
| 9.6 | PCBs in sediment | 412 |
| 9.6.1 | Introduction | 412 |
| 9.6.2 | Production of the material | 413 |
| 9.6.3 | Certification | 414 |
| 9.6.4 | Participating laboratories | 415 |
| 9.7 | PAHs in sediment | 416 |
| 9.7.1 | Introduction | 416 |
| 9.7.2 | Production of the material | 416 |
| 9.7.3 | Certification | 417 |
| 9.7.4 | Participating laboratories | 418 |
| 9.8 | References | 419 |

CHAPTER 10. CRMs FOR SOIL AND SLUDGE ANALYSIS

| | | |
|--------|---|-----|
| 10.1 | Introduction | 421 |
| 10.2 | Trace elements in soils | 421 |
| 10.2.1 | Introduction | 421 |
| 10.2.2 | Production of the material | 422 |
| 10.2.3 | Certification | 425 |
| 10.2.4 | Participating laboratories | 426 |
| 10.3 | Extractable trace elements in sewage sludge amended soils | 427 |
| 10.3.1 | Introduction | 427 |
| 10.3.2 | Production of the material | 429 |
| 10.3.3 | Certification | 431 |
| 10.3.4 | Participating laboratories | 432 |
| 10.4 | CBs in sewage sludge | 434 |
| 10.4.1 | Introduction | 434 |
| 10.4.2 | Production of the material | 435 |
| 10.4.3 | Certification | 436 |
| 10.4.4 | Participating laboratories | 437 |
| 10.5 | PAHs in sewage sludge | 438 |
| 10.5.1 | Introduction | 438 |
| 10.5.2 | Production of the material | 439 |
| 10.5.3 | Certification | 440 |
| 10.5.4 | Participating laboratories | 441 |
| 10.6 | References | 442 |

CHAPTER 11. OTHER TYPES OF CRMs

| | | |
|----------|---|-----|
| 11.1 | Coal materials | 445 |
| 11.1.1 | Introduction | 445 |
| 11.1.2 | Trace elements in coal | 445 |
| 11.1.2.1 | Production of the materials | 445 |
| 11.1.2.2 | Certification | 446 |
| 11.1.2.3 | Participating laboratories | 446 |
| 11.1.3 | Minor and major elements in three coals | 448 |
| 11.1.3.1 | Introduction | 448 |
| 11.1.3.2 | Production of the reference materials | 448 |
| 11.1.3.3 | Certification | 449 |
| 11.1.3.4 | Participating laboratories | 450 |
| 11.1.4 | Sulphur in six coals | 451 |
| 11.1.4.1 | Introduction | 451 |
| 11.1.4.2 | Production of the reference material | 452 |
| 11.1.4.3 | Certification | 452 |
| 11.1.4.4 | Participating laboratories | 454 |
| 11.1.5 | Fluorine in coal | 454 |
| 11.1.5.1 | Introduction | 454 |
| 11.1.5.2 | Production of the reference material | 454 |
| 11.1.5.3 | Certification | 455 |
| 11.1.5.4 | Participating laboratories | 455 |
| 11.2 | Ash materials | 456 |
| 11.2.1 | Introduction | 456 |

| | | |
|----------|--|-----|
| 11.2.2 | Trace elements, fluorine and chlorine in ash | 456 |
| 11.2.2.1 | Introduction | 457 |
| 11.2.2.2 | Production of the reference material | 457 |
| 11.2.2.3 | Certification | 458 |
| 11.2.2.4 | Participating laboratories | 461 |
| 11.2.3 | Dioxins in fly ash extract | 462 |
| 11.2.3.1 | Introduction | 462 |
| 11.2.3.2 | Preparation of the reference material | 462 |
| 11.2.3.3 | Certification | 464 |
| 11.2.3.4 | Participating laboratories | 465 |
| 11.3 | Dust materials | 466 |
| 11.3.1 | Trimethyllead in urban dust | 466 |
| 11.3.1.1 | Introduction | 466 |
| 11.3.1.2 | Feasibility study | 466 |
| 11.3.1.3 | Interlaboratory study | 467 |
| 11.3.1.4 | Production of the material | 467 |
| 11.3.1.5 | Certification | 469 |
| 11.3.1.6 | Participating laboratories | 470 |
| 11.3.2 | Cr(VI) in welding dust | 470 |
| 11.3.2.1 | Introduction | 470 |
| 11.3.2.2 | Preliminary investigations | 471 |
| 11.3.2.3 | Production of the material | 472 |
| 11.3.2.4 | Certification | 473 |
| 11.3.2.5 | Participating laboratories | 473 |
| 11.4 | PCBs in mineral oil | 474 |
| 11.4.1 | Introduction | 474 |
| 11.4.2 | Production of the materials | 475 |
| 11.4.3 | Certification | 476 |
| 11.4.4 | Participating laboratories | 477 |
| 11.5 | References | 478 |

CHAPTER 12. INTERLABORATORY STUDIES

| | | |
|----------|--|-----|
| 12.1 | Introduction | 481 |
| 12.2 | Definitions | 481 |
| 12.3 | General principles | 481 |
| 12.3.1 | Participants | 482 |
| 12.3.2 | Organiser | 482 |
| 12.3.3 | Objective of the study | 482 |
| 12.3.4 | Choice and preparation of the test materials | 483 |
| 12.3.4.1 | Selection of the test material | 483 |
| 12.3.4.2 | Representativeness | 483 |
| 12.3.4.3 | Preparation | 484 |
| 12.3.4.4 | Assigned values | 484 |
| 12.3.5 | Analytical protocol | 486 |
| 12.3.6 | Collection and evaluation of data | 486 |
| 12.3.6.1 | Collection of results | 486 |
| 12.3.6.2 | Technical evaluation | 487 |
| 12.3.6.3 | Statistical evaluation | 488 |

| | | |
|------------------|--|-----|
| 12.3.6.4 | Conclusions and actions | 488 |
| 12.4 | Method performance studies | 490 |
| 12.4.1 | Definition | 490 |
| 12.4.2 | Fields of application | 490 |
| 12.4.3 | Method and protocol | 491 |
| 12.4.4 | Participants | 491 |
| 12.4.5 | Materials | 491 |
| 12.4.6 | Reporting and exploitation of the data | 492 |
| 12.4.7 | Examples of method performance study | 492 |
| 12.4.7.1 | Extraction tests for soil and sediment analysis | 492 |
| 12.4.7.2 | Evaluation of methods of microbiological parameters | 498 |
| 12.5 | Laboratory performance studies | 498 |
| 12.5.1 | Definition | 498 |
| 12.5.2 | Field of application | 499 |
| 12.5.3 | Objective and organisation | 499 |
| 12.5.4 | Evaluation of performance | 500 |
| 12.5.5 | Relation to accreditation | 501 |
| 12.5.6 | Example of laboratory performance study: Proficiency testing scheme for marine monitoring | 503 |
| 12.5.6.1 | Introduction | 503 |
| 12.5.6.2 | Framework | 504 |
| 12.5.6.3 | Test materials | 505 |
| 12.5.6.4 | Performance evaluation | 505 |
| 12.5.6.5 | Developments | 506 |
| 12.5.7 | Conclusion | 507 |
| 12.6 | Material certification studies | 507 |
| 12.7 | Improvement schemes | 508 |
| 12.7.1 | Aims and principles | 508 |
| 12.7.2 | Organisation | 508 |
| 12.7.3 | General principles | 509 |
| 12.7.4 | Example of improvement scheme: Methyl mercury in environmental matrices | 510 |
| 12.7.4.1 | Aim of the project | 510 |
| 12.7.4.2 | The interlaboratory programme | 510 |
| 12.7.5 | Example of improvement scheme: Seawater microbiology | 515 |
| 12.8 | Conclusions | 516 |
| 12.9 | References | 516 |
| Index | | 537 |
| Laboratory Index | | 551 |