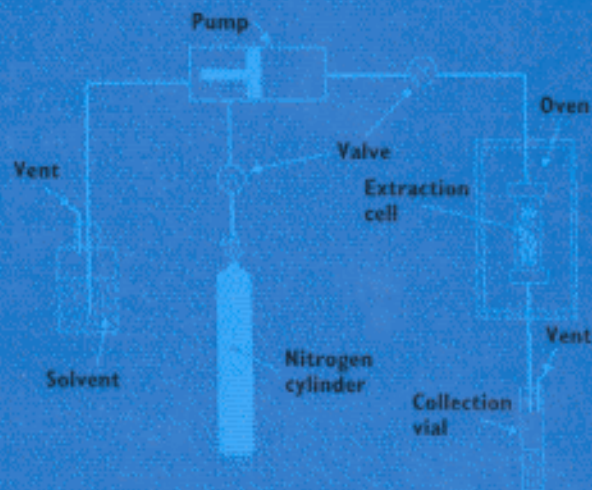


AnTs

Analytical Techniques in the Sciences

# METHODS FOR ENVIRONMENTAL TRACE ANALYSIS

John R. Dean



 WILEY

# Contents

<b>Series Preface</b>	<b>xiii</b>
<b>Preface</b>	<b>xv</b>
<b>Acronyms, Abbreviations and Symbols</b>	<b>xix</b>
<b>About the Author</b>	<b>xviii</b>
<b>1 Basic Laboratory Skills</b>	<b>1</b>
1.1 Introduction	1
1.2 Safety Aspects	1
1.3 Recording of Practical Results	3
1.4 Units	3
1.5 Sample Handling: Liquids	5
1.6 Sample Handling: Solids	5
1.7 Preparing Solutions for Quantitative Work	6
1.8 Presentation of Data: Tables	6
1.9 Presentation of Data: Graphs	7
1.10 Calculations: Dilution Factors	9
Further Reading	11
<b>2 Investigative Approach for Sample Preparation</b>	<b>13</b>
2.1 Introduction	13
2.2 Quality Assurance	14
References	25

<b>3</b>	<b>Sampling</b>	<b>27</b>
3.1	Introduction	27
3.2	Sampling Methods	29
3.3	Number of Samples	30
3.4	Sampling Soil and Sediment	31
3.5	Sampling Water	34
3.6	Sampling Air	37
	References	37
<b>4</b>	<b>Storage of Samples</b>	<b>39</b>
4.1	Introduction	39
4.2	Methods	40
	References	45
	<b>SAMPLE PREPARATION FOR INORGANIC ANALYSIS</b>	<b>47</b>
<b>5</b>	<b>Solids</b>	<b>49</b>
5.1	Introduction	50
5.2	Decomposition Techniques	50
5.3	Dry Ashing	50
5.4	Acid Digestion (including the Use of Microwaves)	51
5.4.1	Microwave Digestion	55
5.4.2	Microwave Digestion Procedure	61
5.4.3	Fusion	64
5.5	Speciation Studies	65
5.6	Selected Examples of Metal Speciation	66
5.6.1	Mercury	66
5.6.2	Tin	69
5.6.3	Arsenic	70
5.6.4	Chromium	75
5.7	Selective Extraction Methods	76
5.7.1	Plant Uptake Studies	76
5.7.2	Soil Pollution Studies	76
5.7.3	Single Extraction Procedures	78
5.7.4	Sequential Extraction Procedure	83
5.7.5	Food Studies	88
5.8	Case Studies on Total and Selective Methods of Metal Analysis	92
5.8.1	Example 5.1: Total Metal Analysis of Soil, followed by Flame Atomic Absorption Spectroscopy	92

5.8.2	Example 5.2: Total Metal Analysis of Soil Using X-Ray Fluorescence Spectroscopy – Comparison with Acid Digestion (Method 3050B), followed by Flame Atomic Absorption Spectroscopy	93
5.8.3	Example 5.3: Sequential Metal Analysis of Soils, followed by Flame Atomic Absorption Spectroscopy	94
	References	96
<b>6</b>	<b>Liquids – Natural and Waste Waters</b>	<b>99</b>
6.1	Introduction	99
6.2	Liquid–Liquid Extraction	100
6.3	Ion-Exchange	103
6.4	Co-Precipitation	104
	References	105
	<b>SAMPLE PREPARATION FOR ORGANIC ANALYSIS</b>	<b>107</b>
<b>7</b>	<b>Solids</b>	<b>109</b>
7.1	Introduction	109
7.2	Soxhlet Extraction	110
7.2.1	Example 7.1: Soxhlet Extraction of Polycyclic Aromatic Hydrocarbons from Contaminated Soil	113
7.3	Shake-Flask Extraction	114
7.3.1	Example 7.2: Shake-Flask Extraction of Phenols from Contaminated Soil	115
7.4	Ultrasonic Extraction	116
7.5	Supercritical Fluid Extraction	118
7.5.1	Instrumentation	120
7.5.2	Example 7.3: Supercritical Fluid Extraction of Organochlorine Pesticides from Contaminated Soil and ‘Celite’	122
7.6	Microwave-Assisted Extraction	124
7.6.1	Instrumentation	124
7.6.2	Example 7.4: Atmospheric Microwave-Assisted Extraction of Polycyclic Aromatic Hydrocarbons from Contaminated Soil	126
7.6.3	Example 7.5: Pressurized Microwave-Assisted Extraction of Polycyclic Aromatic Hydrocarbons from Contaminated Soil	128
7.7	Pressurized Fluid Extraction	129
7.7.1	Instrumentation	130

7.7.2	Example 7.6: Pressurized Fluid Extraction of DDT, DDD and DDE from Contaminated Soil	133
7.8	Matrix Solid-Phase Dispersion	135
7.8.1	Example 7.7: Matrix Solid-Phase Dispersion of an Alcohol Ethoxylate (Lutensol, C13 and C15, with an Average Ethoxy Chain of EO7), Spiked onto an Homogenized Fish Tissue	135
	References	139
<b>8</b>	<b>Liquids</b>	<b>141</b>
8.1	Liquid-Liquid Extraction	141
8.2	Solvent Extraction	142
8.2.1	Example 8.1: Liquid-Liquid Extraction of various Polycyclic Aromatic Hydrocarbons from Water	145
8.3	Solid-Phase Extraction	147
8.3.1	Types of SPE Media	148
8.3.2	Cartridge or Disc Format	149
8.3.3	Method of SPE Operation	152
8.3.4	Solvent Selection	154
8.3.5	Factors Affecting SPE	155
8.3.6	Example 8.2: Solid-Phase Extraction of various Phenols from Water	156
8.4	Solid-Phase Microextraction	158
8.4.1	Experimental	159
8.4.2	Example 8.3: Solid-Phase Microextraction of BTEX from Water	160
	References	164
<b>9</b>	<b>Volatile Compounds</b>	<b>165</b>
9.1	Introduction	165
9.2	Thermal Desorption	165
9.3	Purge-and-Trap	168
9.3.1	Example 9.1: Purge-and-Trap Extraction of BTEX from Water	169
	References	172
<b>10</b>	<b>Pre-Concentration Using Solvent Evaporation</b>	<b>173</b>
10.1	Introduction	173
10.2	Rotary Evaporation	174
10.3	Kuderna-Danish Evaporative Concentration	175

<i>Contents</i>	xi
10.4 Automated Evaporative Concentration System	176
10.5 Gas 'Blow-Down'	176
References	182
<b>11 Instrumental Techniques for Trace Analysis</b>	<b>183</b>
11.1 Introduction	184
11.2 Environmental Organic Analysis	185
11.2.1 Chromatographic Techniques	185
11.2.2 Other Techniques	191
11.3 Environmental Inorganic Analysis	192
11.3.1 Atomic Absorption Spectroscopy	192
11.3.2 Atomic Emission Spectroscopy	198
11.3.3 Inductively Coupled Plasma–Mass Spectrometry	201
11.3.4 Other Techniques	203
<b>12 Recording of Information in the Laboratory and Selected Resources</b>	<b>207</b>
12.1 Recording of Information	207
12.1.1 Introduction	207
12.1.2 Examples of Data Sheets	209
12.2 Selected Resources	219
12.2.1 Journals	219
12.2.2 Books	219
12.2.3 Software	225
12.2.4 CD-ROMs	226
12.2.5 Videos	226
12.2.6 Useful Websites	226
<b>Responses to Self-Assessment Questions</b>	<b>229</b>
<b>Glossary of Terms</b>	<b>243</b>
<b>SI Units and Physical Constants</b>	<b>251</b>
<b>Periodic Table</b>	<b>255</b>
<b>Index</b>	<b>257</b>