

Contents

Preface	iii
List of Tables	xiii
Chapter 1. INTRODUCTION	
1.1 Objectives of Organic Elemental Analysis	1
1.2 Historical Background	2
1.3 Recent Developments	3
1.4 General Remarks on Organic Microanalysis	7
1.5 Experimental: Preparation of the Sample for Organic Elemental Analysis	9
1.6 Experimental: Techniques of Weighing	11
1.7 Experimental: Measurement of Liquid Volume	21
1.8 Experimental: Measurement of Gas Volume	27
References	30
Chapter 2. CARBON AND HYDROGEN	35
2.1 General Considerations	35
2.2 Review of Recent Developments	40
2.3 Experimental: Determination of Carbon, Hydrogen, and Nitrogen Using the Perkin-Elmer Model 240 Elemental Analyzer	51
2.4 Experimental: Determination of Carbon, Hydrogen, and Nitrogen Using the Hewlett-Packard (F&M) C, H, N Analyzer	59
2.5 Experimental: Determination of Carbon, Hydrogen, and Nitrogen Using the Carlo Erba Elemental Analyzer	63
2.6 Experimental: Determination of Carbon and Hydrogen by Gravimetry	69

2.7	Experimental: Rapid Multiple Gravimetric Determination of Carbon and Hydrogen	75
	References	80
Chapter 3.	NITROGEN	87
3.1	General Considerations	87
3.2	Review of Recent Developments	93
3.3	Experimental: Determination of Nitrogen by the Micro Kjeldahl Method	
3.4	Experimental: Determination of Nitrogen by the Micro Dumas Method	
3.5	Experimental: Determination of Nitrogen Using the Coleman Nitrogen Analyzer Model 29	
	References	122
Chapter 4.	OXYGEN	128
4.1	General Considerations	128
4.2	Current Practice	134
4.3	Survey of Recent Developments	141
4.4	Experimental: Determination of Oxygen by Pyrolytic Reduction and Iodometry	143
4.5	Experimental: Automated Determination of Oxygen Using Coulometry at Controlled Potential	146
4.6	Experimental: Determination of Oxygen Using a Modified Perkin-Elmer Elemental Analyzer	150
	References	153
Chapter 5.	THE HALOGENS	158
5.1	General Considerations	158
5.2	Review of Recent Developments	169
5.3	Experimental: Decomposition by Closed-flask Combustion	175
5.4	Experimental: Potentiometric Determination of Chlorine or Bromine	178
5.5	Experimental: Determination of Iodine by Titrimetry	182
5.6	Experimental: Determination of Fluorine Using an Ion-specific Electrode	185
5.7	Experimental: Determination of Fluorine by Alkali Fusion, Fluorosilicic Acid Distillation, and Thorium Nitrate Titration	188
5.8	Experimental: Determination of Fluorine by Automated Spectrophotometry Using the Zirconium-SPADNS Reagent	192
5.9	Experimental: Gravimetric Determination of Chlorine or Bromine	194
	References	198

CONTENTS

Chapter 6.	SULFUR, SELENIUM, AND TELLURIUM	207
6.1	General Considerations	207
6.2	Current Practice	209
6.3	Review of Recent Developments	212
6.4	Experimental: Determination of Sulfur by Titrating the Sulfate	218
6.5	Experimental: Determination of Sulfur by Gravimetry	220
6.6	Experimental: Determination of Sulfur by Acidimetry	223
6.7	Experimental: Determination of Selenium	225
6.8	Experimental: Determination of Tellurium	226
	References	228
Chapter 7.	PHOSPHORUS, ARSENIC, ANTIMONY, AND BISMUTH	235
7.1	General Considerations	235
7.2	Current Practice	235
7.3	Survey of Recent Developments	241
7.4	Experimental: Determination of Phosphorus by Acid Digestion and Phosphovanadomolybdate Colorimetry	244
7.5	Experimental: Determination of Phosphorus by the Molybdenum Blue Method	247
7.6	Experimental: Determination of Arsenic	248
7.7	Experimental: Determination of Antimony	250
7.8	Experimental: Determination of Bismuth	251
	References	252
Chapter 8.	BORON, SILICON, AND MERCURY	258
8.1	Introductory Remarks	258
8.2	Current Practice	259
8.3	Recent Developments	267
8.4	Experimental: Determination of Boron	270
8.5	Experimental: Determination of Silicon	272
8.6	Experimental: Determination of Mercury by Gravimetry	275
8.7	Experimental: Determination of Mercury by Automated Spectrophotometric Titration with Potassium Thiocyanate	276
	References	279
Chapter 9.	THE METALLIC ELEMENTS	285
9.1	General Considerations	285
9.2	Current Practice	286
9.3	Survey of Recent Literature	293
9.4	Experimental: Determination of Metallic Elements by Ashing in a Crucible	299
9.5	Experimental: Determination of Metallic Elements by Heating in the Combustion Tube	300

9.6	Experimental: Determination of Metallic Elements by Acid Digestion and Indirect Polarography	303
9.7	Experimental: Determination of Nickel, Cobalt, or Palladium in Organometallic Complexes by Indirect EDTA Titration	305
9.8	Experimental: Gravimetric Determination of Tin and Other Metals by Means of Cupferron	307
	References	309
Chapter 10.	SIMULTANEOUS DETERMINATION OF SEVERAL ELEMENTS	314
10.1	Introductory Remarks	314
10.2	Current Practice	316
10.3	Survey of Recent Developments	320
10.4	Experimental: Simultaneous Determination of Chlorine, Bromine, and Iodine with One Sample	
10.5	Experimental: Simultaneous Gravimetric Determination of Carbon, Hydrogen, and the Other Elements with One Sample	
10.6	Experimental: Simultaneous Determination of Nitrogen, Phosphorus, and Boron with One Sample	352
	References	355
Chapter 11.	ULTRAMICRO ANALYSIS	365
11.1	General Considerations	365
11.2	Current Practice	368
11.3	Review of Literature	382
11.4	Experimental: Determination of Chlorine Using Decimilligram Samples	387
11.5	Experimental: Determination of Chlorine Using Centimilligram Samples	392
11.6	Experimental: Ultramicro Kjeldahl Determination of Nitrogen Using the Aeration Technique	398
	References	401
Chapter 12.	TRACE ANALYSIS	409
12.1	General Remarks	409
12.2	Approaches for Trace Analysis of the Elements	411
12.3	Current Practice	418
12.4	Survey of Recent Literature	432
12.5	Experimental: Determination of Fluorine at the Parts-per-Million Level Using the Diffusion Technique	442
12.6	Experimental: Determination of Total Mercury in Fish by Pyrolysis and Ultraviolet Photometry	446

CONTENTS

xi

12.7	Experimental: Determination of Trace Mercury in Coal by Cold Vapor Atomic Absorption Spectrometry	449
12.8	Experimental: Determination of Trace Carbon in Inorganic Matrix	450
	References	456
Appendix.	NITROGEN DETERMINATIONS IN THE U. S. PHARMACOPEIA	473
	Author Index	477
	Subject Index	513