

CONTENTS

Author's Preface	5
Foreword to the English Edition	6
Part I GENERAL METHODS OF ANALYSIS	16
Chapter 1 Chemical Methods	17
1.1 Preparation of Test Sample and Separation of Polymer by <i>W. Czerwiński</i>	17
1.2 Identification Tests by <i>W. Czerwiński</i>	18
Identification by Thermal Decomposition	19
Solubility Tests	22
Qualitative Elemental Analysis	26
Identification by Colour Tests	28
References	33
1.3 Elemental Analysis by <i>J. Urbański</i>	35
Carbon and Hydrogen	35
Nitrogen	36
Chlorine	38
Fluorine	42
Sulphur	45
Phosphorus	46
References	46
1.4 Estimation of Chemical Characteristics by <i>J. Urbański</i>	48
Acid Number	48
Saponification Value	49
Iodine Value	51
Hydroxyl Value	52
References	54
1.5 Determination of Water by <i>J. Urbański</i>	54
Drying	54
Azeotropic Distillation	55
The Karl Fischer Method	56
Manometric Method	60
Gas Chromatography	61
Infrared Spectroscopy	61
NMR Spectroscopy	62
Other Methods	62
References	62
Chapter 2 Infrared and Ultraviolet Absorption Spectroscopy by <i>K. Janicka</i>	64
2.1 Introduction	64
2.2 Principles	65
Vibrations of Molecules	65
Absorption Laws	67
2.3 Sample Preparation	68
Capillary Films	68
Solutions	69

	Films	69
	Mulls	72
	Pellets	73
2.4	Ultraviolet Spectrophotometry	73
2.5	Infrared Absorption Spectrophotometry	75
	Qualitative Analysis	75
	Quantitative Analysis	79
	Analysis of Polymers in Polarised Light	93
	Attenuated Total Reflection (ATR) Technique	95
	References	96
Chapter 3	Chromatography by <i>H. Zowall</i>	101
3.1	Principles	101
	Supports	104
	Adsorbents	105
	Solvents	105
	Apparatus	106
3.2	Paper Chromatography	108
	Principles	108
	Paper and Solvents	108
	Reversed Phase Chromatography	108
	Other Procedures in Paper Chromatography	108
	Quantitative Chromatography	109
	Preparative Chromatography	109
	Apparatus	109
	Detection	110
	Interpretation of Chromatograms	110
3.3	Thin Layer Chromatography (TLC)	111
	Fundamental Procedure	111
	Adsorbents and Supports	112
	Quantitative Chromatography	112
	Apparatus	112
	Mobile Phase	113
	Analytical and Preparative Procedures	113
	Detection and Interpretation of Chromatograms	114
3.4	Gas Chromatography	114
	Supports	116
	Stationary Phase	117
	Columns	117
	Mobile Phase	118
	Apparatus	118
	Identification	119
	Quantitative Analysis	119
3.5	Application of Chromatography to Analysis of Polymers	122
	Fractionation	122
	Identification	123
	Quantitative Determinations	125
	References	126
Chapter 4	Polarography by <i>J. Urbański</i>	129
4.1	Introduction	129
4.2	Experimental Technique	132
	Apparatus	132
	Solvents	132
	Supporting Electrolytes	133

4.3	Polarography in the Study of Polymers	133
	Introduction	133
	Investigation of the Physical Properties of Polymers	134
	Qualitative Analysis	135
	Quantitative Analysis	135
	References	140
Chapter 5	Nuclear Magnetic Resonance (NMR) Spectroscopy by <i>J. Urbański</i>	142
5.1	Principles	142
5.2	Experimental Technique	145
	Solvents	145
	Sample Preparation	145
	Results	146
5.3	NMR Spectroscopy in Polymer Studies	147
	Introduction	147
	Broad-line NMR Spectroscopy	147
	Study of Chemical Reactions	149
	High-Resolution NMR Spectroscopy	153
	References	158
Part II	ANALYSIS OF POLYMERS	163
Chapter 6	Phenolic Resins by <i>J. Urbański</i>	165
6.1	Structure	166
6.2	Qualitative Analysis	168
	Direct Chemical Tests	168
	Chemical Tests after Pyrolysis	170
	Tests after Alkali Fusion	171
	Instrumental Methods	171
6.3	Determination of Additives and Contaminants	174
	Water	174
	Ash	174
	Unreacted Substrates	175
	Instrumental Methods	179
6.4	Chemical Composition of the Polymer	181
	Elemental Analysis	181
	Active Sites	182
	Ether Bridges	183
	Methylene Bridges	183
	Hydroxyl Groups	183
6.5	Analysis of Moulding Powders	183
	Resin	186
	Unreacted Substrates	187
	Moisture	187
	Hexamethylenetetramine	187
6.6	Cured Resins	189
	References	191
Chapter 7	Amino Resins by <i>J. Urbański</i>	194
7.1	Structure	194
7.2	Qualitative Analysis	194
	Chemical Methods	194
	Instrumental Methods	201
7.3	Determination of Additives and Contaminants	204
	Water	204
	Hexamethylenetetramine	204

	Ash	205
	Volatile Matter	205
	Free Formaldehyde	205
	Free Aniline	205
	Free Alcohols	205
7.4	Chemical Composition of the Polymer	206
	Elemental Analysis	206
	Structural Units Derived from Formaldehyde	206
7.5	Cured Resins	218
	Resin	219
	Urea	219
	Melamine	219
	Thiourea	220
	Fillers	220
	Water	220
	Free Formaldehyde	221
	References	221
Chapter 8	Polyester Resins by <i>J. Urbański</i>	223
8.1	Introduction	223
8.2	Structure	225
8.3	Qualitative Analysis	226
	Detection of Dicarboxylic Acids	227
	Detection of Polyhydric Alcohols	236
	Detection of Fatty Acids	244
	Detection of Styrene	245
8.4	Quantitative Analysis	245
	Determination of Structural Components	246
	Hydrolytic Decomposition of Polyesters and Separation of the Polymer Components	246
	Determination of Dicarboxylic Acids	247
	Determination of Fatty Acids	252
	Determination of Polyhydric Alcohols	253
	Determination of Functional Groups	259
8.5	Determination of Other Constituents	264
	Unreacted Anhydrides	264
	Free Monomer	265
	Unhydrolysable Matter	267
	Water	267
	Catalysts	267
	Miscellaneous Contaminants and Additives	268
8.6	Degree of Cure of Unsaturated Resins	268
	References	268
Chapter 9	Polyamides by <i>F. Majewska</i>	273
9.1	Introduction	273
9.2	Qualitative Analysis	275
	Chemical Methods	275
	Instrumental Methods	277
9.3	Chemical Composition of the Polymer	284
	Determination of Constituents	284
	References	292
Chapter 10	Uncured Epoxy Resins by <i>J. Urbański</i>	295
10.1	Structure	295
10.2	Qualitative Analysis	298
	Chemical Methods	298

Contents

11

10.3	Chemical Composition of the Polymer	301
	Determination of Epoxy Groups	301
	Determination of Hydroxyl Groups	308
	Determination of Phenolic Hydroxyl Groups	310
	Determination of α -Glycol Groups	311
	Determination of Chlorine Bound in Chlorohydrin Groups (Active Chlorine)	312
	Total Organically Bound Chlorine	312
	Inactive Chlorine	313
	Bisphenol Component	314
10.4	Determination of Contaminants	314
10.5	Analysis of Cured Epoxy Resins	316
	References	317
Chapter 11	Polyurethanes by <i>F. Majewska</i>	319
11.1	Qualitative Analysis	320
11.2	Quantitative Analysis	325
	References	330
Chapter 12	Polyformaldehyde by <i>J. Urbański</i>	332
12.1	Structure	332
12.2	Qualitative Analysis	333
12.3	Chemical Composition of the Polymer	333
	Acetate Groups	333
	Methoxyl Groups	334
	Formaldehyde	336
	Dioxolan Content	336
	Water	337
	Antioxidants	337
	References	338
Chapter 13	Polyolefins by <i>J. Urbański</i>	339
13.1	Structure	340
	Polyethylene	340
	Polymers of Ethylene Homologues	342
13.2	Qualitative Tests	343
	Chemical Methods	343
	Instrumental Methods	343
13.3	Chemical Composition of the Polymer	346
13.4	Determination of Contaminants and Additives	350
	Water and Carbon Black	350
	Ash	350
	Metals and Other Elements	351
	Antioxidants	352
	References	353
Chapter 14	Polymers of Chlorine-Containing Olefins by <i>J. Urbański</i>	356
14.1	Poly(vinyl chloride)	356
	Structure	357
	Qualitative Analysis	357
	Quantitative Analysis	359
14.2	Chlorinated Poly(vinyl chloride)	362
	Qualitative Analysis	362
14.3	Poly(vinylidene chloride)	363
	Qualitative Analysis	363

14.4	Vinyl Chloride-Vinyl Acetate Copolymers	363
	Qualitative Analysis	364
	Quantitative Analysis	364
14.5	Vinyl Chloride-Vinylidene Chloride Copolymers	366
	References	366
Chapter 15	Fluoroethylene Polymers by <i>J. Urbański</i>	369
15.1	Polytetrafluoroethylene	369
	Qualitative Analysis	369
	Chemical Composition of the Polymer	370
	Additives and Contaminants	370
15.2	Polychlorotrifluoroethylene	370
	Qualitative Analysis	370
	Chemical Composition of the Polymer	371
	References	371
Chapter 16	Polystyrene and Copolymers by <i>J. Urbański</i>	372
16.1	Polystyrene	372
	Structure	372
	Qualitative Analysis	373
	Structural Elements of the Polymer	374
	Composition of High-Impact Polystyrene	378
16.2	Styrene-Butadiene Copolymers	378
	Structure	379
	Qualitative Analysis	379
	Structural Elements of the Polymer	379
	Additives and Contaminants	381
16.3	Acrylonitrile-Butadiene-Styrene (ABS) Terpolymers	383
	Qualitative Analysis	383
	Structural Elements of the Polymer	383
	References	385
Chapter 17	Poly(vinyl alcohol) and Its Derivatives by <i>J. Urbański</i>	388
17.1	Poly(vinyl acetate)	388
	Structure	388
	Qualitative Analysis	389
17.2	Poly(vinyl alcohol)	391
	Structure	392
	Qualitative Analysis	392
	Chemical Composition of the Polymer	392
17.3	Poly(vinyl ethers)	395
	Qualitative Analysis	395
	Quantitative Analysis of Vinyl Ethyl Ether Monomer	396
17.4	Poly(vinyl acetals)	396
	Structure	397
	Qualitative Analysis	397
	Chemical Composition of the Polymer	398
	References	400
Chapter 18	Acrylic Polymers by <i>J. Urbański</i>	403
18.1	Polymethacrylates and Polyacrylates	403
	Structure	404
	Qualitative Analysis	404
	Quantitative Analysis	407
18.2	Polyacrylonitrile	410
	Structure	410

Contents

13

	Qualitative Analysis	411
	Chemical Composition of the Polymer	411
	References	413
Chapter 19	Cellulose Derivatives by <i>W. Czerwiński</i>	415
19.1	Separation and Determination of Plasticiser	415
	Extraction Method	415
	Precipitation Method	415
19.2	Cellulose Esters (except Cellulose Nitrate)	418
	Identification	419
	Quantitative Analysis	420
19.3	Cellulose Nitrate	423
	Identification	424
	Quantitative Analysis	424
19.4	Cellulose Ethers	427
	Identification	427
	Quantitative Analysis	429
	References	429
Chapter 20	Silicones by <i>J. Urbański</i>	431
20.1	Structure	432
20.2	Qualitative Analysis	433
20.3	Chemical Composition of the Polymer	436
	Silicon	436
	Carbon and Hydrogen	440
	Chlorine	442
	Hydrogen Bonded to Silicon	443
	Hydroxyl Groups Bonded to Silicon	446
	Alkoxyl and Aryloxyl Groups	449
	Organic Substituents Bonded to Silicon	451
	Olefinic Double Bonds	453
20.4	Additives and Contaminants	453
	References	454
Index		457