

VOGEL'S

TEXTBOOK OF QUANTITATIVE CHEMICAL ANALYSIS

J MENDHAM · R C DENNEY·
J D BARNES · M J K THOMAS

6TH
EDITION

PRENTICE HALL

Contents

<i>Detailed contents</i>	vi
<i>Preface to the sixth edition</i>	xxi
<i>Preface to first edition</i>	xxiv
<i>Safety in the laboratory</i>	xxvi
<i>Units and reagent purity</i>	xxvii
1 Chemical analysis	1
2 Solution reactions: fundamental theory	12
3 Common apparatus and basic techniques	66
4 Statistics: introduction to chemometrics	117
5 Sampling	170
6 Separation	211
7 Thin-layer chromatography	251
8 Liquid chromatography	261
9 Gas chromatography	289
10 Titrimetric analysis	312
11 Gravimetric analysis	461
12 Thermal analysis	475
13 Direct electroanalytical methods	495
14 Nuclear magnetic resonance spectroscopy	571
15 Atomic absorption spectroscopy	584
16 Atomic emission spectroscopy	617
17 Molecular electronic spectroscopy	630
18 Vibrational spectroscopy	692
19 Mass spectrometry	717
<i>Appendices</i>	763
<i>Index</i>	786

Detailed contents

1	Chemical analysis	1
1.1	Introduction	1
1.2	Applications	1
1.3	Stages of analysis	2
1.4	Selecting the method	3
1.5	Searching the literature	4
1.6	Quantitative analysis	5
1.7	Special techniques	6
1.8	Instrumental methods	8
1.9	Factors affecting the choice of analytical method	8
1.10	Interferences	9
1.11	Data handling	10
1.12	Summary	11
2	Solution reactions: fundamental theory	12
2.1	The law of mass action	12
2.2	Activity and activity coefficient	13
2.3	Chemical equilibrium	14
2.4	Factors affecting chemical reactions in solution	16
2.5	The ionic product of water	17
2.6	Electrolytic dissociation	19
2.7	Acid-base equilibria in water	22
2.8	Strengths of acids and bases	23
2.9	Dissociation of polyprotic acids	24
2.10	The hydrogen ion exponent	26
2.11	Buffer solutions	28
2.12	The hydrolysis of salts	31
2.13	Degree of hydrolysis	33
2.14	Solubility product	37
2.15	Common ion effect	39
2.16	Common ion: quantitative effects	41
2.17	Fractional precipitation	43
2.18	Precipitate solubility: effect of acids	44
2.19	Precipitate solubility: effect of temperature	44

2.20	Precipitate solubility: effect of solvent	45
2.21	Complex ions	45
2.22	Complexation	46
2.23	Stability of complexes	47
2.24	Metal ion buffers	48
2.25	Complex stability: important factors	48
2.26	Complexones	50
2.27	Stability constants of EDTA complexes	53
2.28	Electrode potentials	54
2.29	Concentration cells	57
2.30	Calculating the e.m.f. of a voltaic cell	58
2.31	Oxidation-reduction cells	59
2.32	Calculating the standard reduction potential	61
2.33	Equilibrium constants of redox reactions	61
2.34	References	65
2.35	Bibliography	65
3	Common apparatus and basic techniques	66
3.1	Introduction	66
	Balances	68
3.2	The analytical balance	68
3.3	Other balances	70
3.4	Weights and reference masses	70
3.5	Care and use of analytical balances	71
3.6	Errors in weighing	72
	Graduated glassware	75
3.7	Units of volume	75
3.8	Graduated apparatus	75
3.9	Temperature standard	76
3.10	Graduated flasks	77
3.11	Pipettes	77
3.12	Burettes	79
3.13	Weight burettes	81
3.14	Piston burettes	82
3.15	Graduated (measuring) cylinders	82
3.16	Calibration of graduated apparatus	82
	Water for laboratory use	84
3.17	Purified water	84
3.18	Wash bottles	86
	General apparatus	87
3.19	Glassware, ceramics, plastic ware	87
3.20	Metal apparatus	89
3.21	Heating apparatus	92
3.22	Desiccators and dry boxes	93
3.23	Stirring apparatus	96
3.24	Filtration apparatus	97
3.25	Weighing bottles	99

Detailed contents

	Reagents and standard solutions	100
3.26	Reagents	100
3.27	Purification of substances	101
3.28	Standard solutions	102
	Some basic techniques	104
3.29	Preparing substances for analysis	104
3.30	Weighing the sample	104
3.31	Dissolving the sample	104
3.32	Decomposing organic compounds	107
3.33	Precipitation	108
3.34	Filtration	109
3.35	Filter papers	109
3.36	Crucibles with permanent porous plates	111
3.37	Washing precipitates	112
3.38	Drying and igniting precipitates	113
3.39	References	115
3.40	Bibliography	116
4	Statistics: introduction to chemometrics	117
4.1	Limitations of analytical methods	117
4.2	Classification of errors	117
4.3	Accuracy	118
4.4	Precision	119
4.5	How to reduce systematic errors	120
4.6	Significant figures	122
4.7	Calculators and computers	123
4.8	Mean and standard deviation	123
4.9	Distribution of random errors	125
4.10	Reliability of results	126
4.11	Confidence interval	127
4.12	Comparison of results	128
4.13	Comparing the means of two samples	130
4.14	Paired <i>t</i> -test	131
4.15	The number of replicate determinations	132
4.16	Correlation and regression	133
4.17	Linear regression	135
4.18	Errors in the slope and the intercept	136
4.19	Error in the estimate of concentration	137
4.20	Standard additions	138
4.21	Non-linear regression	140
4.22	Comparison of more than two means	140
4.23	Experimental design	143
4.24	Two-way analysis of variance	144
4.25	Chemometrics and experimental design	146
4.26	Factorial design	147
4.27	Yates' method	149
4.28	Interaction effect: alternative calculation	152
4.29	Factorial design: critical appraisal	152

4.30	Optimisation methods	154
4.31	Sequential simplex optimisation	154
4.32	Simplex optimisation: critical appraisal	159
4.33	Treatment of multivariate data	160
4.34	Factor analysis	165
4.35	Quick statistics	166
4.36	The value of chemometrics	169
4.37	References	169
4.38	Bibliography	169
5	Sampling	170
5.1	Introduction	170
5.2	Gases and vapours	173
5.3	Liquids	191
5.4	Solids	201
5.5	References	210
5.6	Bibliography	210
6	Separation	211
6.1	Introduction	211
6.2	Separation techniques	212
6.3	Solvent extraction	214
6.4	Crystallisation and precipitation	225
6.5	Ion exchange separations	226
6.6	Dialysis and lyophilisation	237
6.7	Instrumental separations	237
6.8	Solid phase extraction	241
6.9	Comparing separation efficiencies	242
6.10	Kinetic factors: rate theory	244
6.11	Separations by electrophoresis	246
6.12	The theory of electrophoresis	246
6.13	Instrumentation for capillary electrophoresis	247
6.14	Capillaries	248
6.15	The applied field	248
6.16	The detector	248
6.17	Applications	249
6.18	References	249
6.19	Bibliography	250
7	Thin-layer chromatography	251
7.1	Introduction	251
7.2	The technique of TLC	252
7.3	Stationary phases	255
7.4	Mobile phases	255
7.5	Two-dimensional TLC	255
7.6	High-performance thin-layer chromatography (HPTLC)	256
7.7	Experimental section	257
7.8	Separation and recovery of dyes	257
7.8	Separation of carbohydrates	258

Detailed contents

7.9	Separation of artificial colourants in confectionery	259
7.10	References	260
7.11	Bibliography	260
8	Liquid chromatography	261
8.1	Introduction	261
8.2	Types of liquid chromatography	262
8.3	Mobile phase, sample injection, column design	268
8.4	Choosing a detector	277
8.5	Column efficiency	282
8.6	Chiral chromatography	284
8.7	Derivatisation	285
8.8	Quantitative analysis	286
	Experimental section	287
8.9	Aspirin, phenacetin and caffeine in a mixture	287
8.10	References	288
8.11	Bibliography	288
9	Gas chromatography	289
9.1	Introduction	289
9.2	Apparatus	290
9.3	Programmed temperature	304
9.4	Quantitative analysis	304
9.5	Quantitative procedures	305
9.6	Elemental analysis	306
	Experimental section	308
9.7	Internal normalisation	308
9.8	Sucrose as its trimethylsilyl derivative	308
9.9	Determination of aluminium as its tris(acetylacetonato) complex	309
9.10	Derivatisation and quantitation of sugar alcohols	310
9.11	References	311
9.12	Bibliography	311
10	Titrimetric analysis	312
	Theoretical considerations	312
10.1	Introduction	312
10.2	Titrimetric analysis	312
10.3	Classification of reactions in titrimetric analysis	314
10.4	Standard solutions	314
10.5	Preparation of standard solutions	315
10.6	Primary and secondary standards	315
10.7	Principles of potentiometric titrations	316
10.8	General considerations	317
10.9	Location of end points	318
10.10	Automatic titrators	319
10.11	Advantages of potentiometric titrations	320

Coulometry at constant current	320
10.12 General	320
10.13 Principles	322
10.14 Instrumentation	322
10.15 External generation of titrant	324
10.16 Advantages	325
10.17 Applications	327
Amperometric titrations	327
10.18 Principles	327
10.19 Amperometric titration with a dropping mercury electrode (DME)	329
10.20 Apparatus	330
10.21 Biamperometric titrations	331
10.22 Advantages	332
10.23 Applications	332
Spectrophotometric titrations	333
10.24 General	333
10.25 Apparatus	334
10.26 Technique	334
10.27 Advantages	335
10.28 Applications	335
Neutralisation titrations	335
10.29 Neutralisation indicators	335
10.30 Preparation of indicator solutions	339
10.31 Mixed indicators	340
10.32 Neutralisation curves	341
10.33 Strong acid neutralised by strong base	341
10.34 Weak acid neutralised by strong base	344
10.35 Weak base neutralised by strong acid	346
10.36 Weak acid neutralised by weak base	347
10.37 Polyprotic acid neutralised by strong base	348
10.38 Anions of weak acids titrated with strong acids	349
10.39 Choice of indicators in neutralisation reactions	351
10.40 Titrations in non-aqueous solvents	353
10.41 Solvents for non-aqueous titrations	354
10.42 Indicators for non-aqueous titrations	355
10.43 Standard solutions of acids and alkalis	356
Neutralisation: titrimetric determinations	356
10.44 A mixture of carbonate and hydrogencarbonate	356
10.45 Boric acid	356
10.46 Ammonia in an ammonium salt	357
10.47 Organic nitrogen: the Kjeldahl procedure	359
10.48 Nitrates	359
10.49 Phosphate: precipitation as quinoline molybdophosphate	360
10.50 Relative molecular mass of an organic acid	362
10.51 Hydroxyl groups in carbohydrates	362
10.52 Saponification value of oils and fats	364

Detailed contents

10.53	Purity of acetylsalicylic acid (aspirin)	364
10.54	Amines using a non-aqueous titration	365
	Neutralisation: determinations using instruments	365
10.55	Potentiometry: general considerations	365
10.56	Potentiometric titration of ethanoic (acetic) acid with sodium hydroxide	366
10.57	Potentiometric titrations in non-aqueous solvents	367
10.58	Non-aqueous titration of a mixture of aniline and ethanolamine	368
10.59	Acids and bases by coulometry	369
10.60	Organic compounds by spectrophotometric titrations	370
	Complexation titrations	371
10.61	Introduction	371
10.62	Titration curves	371
10.63	Types of EDTA titration	372
10.64	Titration of mixtures	373
10.65	Metal ion indicators	375
10.66	Standard EDTA solutions	378
10.67	Some practical considerations	379
	Complexation: determining individual cations	380
10.68	Aluminium: back titration	380
10.69	Barium: direct titration	381
10.70	Bismuth: direct titration	381
10.71	Calcium: substitution titration	381
10.72	Iron(III): direct titration	383
10.73	Nickel: direct titration	383
10.74	A selection of metals by EDTA	383
10.75	Calcium in the presence of magnesium using EGTA	384
10.76	Total hardness of water: permanent and temporary	385
10.77	Calcium in the presence of barium using CDTA	386
10.78	Calcium and lead in a mixture	386
10.79	Chromium(III) and iron(III) in a mixture: kinetic masking	387
10.80	Manganese in the presence of iron: ferromanganese	387
10.81	Nickel in the presence of iron: nickel steel	388
10.82	Lead and tin in a mixture: solder	388
	Complexation: determining individual anions	389
10.83	Phosphates	389
10.84	Sulphates	390
	Other EDTA titrations	390
10.85	Potentiometric titrations	390
10.86	Coulometric titrations	392
10.87	Amperometric titrations	392
10.88	Zinc	392
10.89	Bismuth	393
	Spectrophotometric titrations	393
10.90	Copper(II)	393
10.91	Iron(III)	394

Precipitation titrations	395
10.92 Precipitation reactions	395
10.93 Determining end points in precipitation reactions	397
10.94 Standardising silver nitrate solution	401
10.95 Chlorides and bromides	402
10.96 Iodides	403
10.97 Preparing thiocyanate solutions: Volhard's method	403
10.98 Silver in a silver alloy	404
10.99 Chlorides by Volhard's method	404
10.100 Fluoride: Volhard titration of lead chlorofluoride	406
10.101 Potassium	406
Precipitation: determinations using instruments	407
10.102 Potentiometry: general considerations	408
10.103 Mixtures of halides by potentiometry	408
10.104 Chloride, bromide and iodide by coulometry	408
10.105 Lead by amperometry using potassium dichromate	410
10.106 Sulphate by amperometry using lead nitrate	411
10.107 Iodide by amperometry using mercury(II) nitrate	412
Oxidation-reduction titrations	412
10.108 Change of electrode potential	412
10.109 Formal potentials	416
10.110 Detecting the end point in oxidation-reduction titrations	417
Oxidations with potassium permanganate	419
10.111 Discussion	419
10.112 Preparing 0.02 M potassium permanganate	419
10.113 Standardising permanganate solutions	420
10.114 Hydrogen peroxide	420
10.115 Nitrites	421
10.116 Persulphates	422
Oxidations with potassium dichromate	422
10.117 Discussion	422
10.118 Preparing 0.02 M potassium dichromate	423
10.119 Iron in an ore	423
10.120 Chromium in a chromium(III) salt	424
10.121 Chemical oxygen demand	424
Oxidations with cerium(IV) sulphate solution	425
10.122 General discussion	425
10.123 Preparing 0.1 M cerium(IV) sulphate	426
10.124 Standardising cerium(IV) sulphate solutions	426
10.125 Copper	426
10.126 Molybdate	427
Iodometric titrations	428
10.127 General discussion	428
10.128 Detecting the end point	430
10.129 Preparing 0.05 M iodine solution	431
10.130 Standardising iodine solutions	432
10.131 Preparing 0.1 M sodium thiosulphate	432

10.132	Standardising sodium thiosulphate solutions	433
10.133	Copper in crystallised copper sulphate	434
10.134	Chlorates	435
10.135	Hydrogen peroxide	435
10.136	Dissolved oxygen	436
10.137	Available chlorine in hypochlorites	437
10.138	Hexacyanoferrates(III)	438
10.139	Vitamin C tablets	438
	Oxidations with potassium iodate	438
10.140	General discussion	439
10.141	Preparing 0.025 M potassium iodate	440
10.142	Arsenic or antimony	440
10.143	Hydrazine	441
10.144	Other ions	441
	Oxidations with potassium bromate	442
10.145	General discussion	442
10.146	Preparing 0.02 M potassium bromate	443
10.147	Metals: using 8-hydroxyquinoline (oxine)	443
10.148	Hydroxylamine	444
10.149	Phenol	445
	Reduction of higher oxidation states	445
10.150	General discussion	446
10.151	Reduction with amalgamated zinc: Jones reductor	446
10.152	The silver reductor	448
10.153	Other methods of reduction	449
	Redox reactions: determinations using instruments	450
10.154	Potentiometry: general considerations	450
10.155	Manganese by potentiometry	451
10.156	Copper by potentiometry	452
10.157	Coulometry: general considerations	452
10.158	Cyclohexene by coulometry	452
10.159	8-Hydroxyquinoline (oxine)	454
10.160	Amperometry: general considerations	454
10.161	Thiosulphate by amperometry with iodine	454
10.162	Antimony by amperometry with potassium bromate	455
10.163	Thiosulphate with iodine: dead-stop end point	455
10.164	Glucose by amperometry with an enzyme electrode	455
10.165	The Clark cell for oxygen determinations	456
10.166	Water by amperometry using the Karl Fischer reagent	456
10.167	Robotics	458
10.168	References	459
10.169	Bibliography	460
11	Gravimetric analysis	461
11.1	Introduction	461
11.2	Fundamentals	462
11.3	Precipitation reagents	464

11.4	Determination of chloride, sulphate and metal ions	466
11.5	Gravimetric experiments	466
11.6	Aluminium as the 8-hydroxyquinolate (oxinate)	467
11.7	Chloride as silver chloride	467
11.8	Lead as chromate	469
11.9	Nickel as the dimethylglyoximate	469
11.10	Sulphate as barium sulphate	470
11.11	Procedures for other ions	471
11.12	References	474
11.12	Bibliography	474
12	Thermal analysis	475
12.1	General discussion	475
12.2	Thermogravimetry	475
12.3	Instruments for thermogravimetry	478
12.4	Applications of thermogravimetry	482
	Experimental section	484
12.5	Thermogravimetric experiments	484
12.6	Differential techniques	486
12.7	Instruments for DTA and DSC	487
12.8	Experimental and instrumental factors	489
12.9	Applications of DTA and DSC	490
	Experimental section	491
12.10	DTA studies of hydrated copper sulphate and sodium tungstate	491
12.11	DSC determination of calcium sulphate hydrates in cement	492
12.12	Determining the purity of pharmaceuticals by DSC	492
12.13	References	494
12.14	Bibliography	494
13	Direct electroanalytical methods	495
13.1	Introduction	495
	Electrogravimetric analysis	495
13.2	Electrogravimetry: theory	495
13.3	Electrogravimetry: apparatus	496
13.4	Cell processes	497
13.5	Deposition and separation	500
13.6	Electrolytic separation of metals	500
13.7	Some metals which can be determined	501
	Coulometry	502
13.8	General discussion	502
13.9	Coulometry at controlled potential	503
13.10	Apparatus and general technique	504
	Experimental section	506
13.11	Separation of nickel and cobalt	506
13.12	Flowing stream coulometry	506
13.13	Evaluation of direct coulometry	507

Detailed contents

	Potentiometry	508
13.14	Fundamentals of potentiometry	508
	Reference electrodes	509
13.15	The hydrogen electrode	509
13.16	The calomel electrode	510
13.17	The silver–silver chloride electrode	511
	Indicator and ion selective electrodes	512
13.18	General discussion	512
13.19	The glass electrode	513
13.20	Solid-state ion selective detectors	521
13.21	Biochemical electrodes	522
	Instrumentation and measurement of cell e.m.f.	522
13.22	Using pH meters and selective ion meters	522
13.23	Determination of pH	524
13.24	Determining fluoride	527
13.25	Potentiometry in an oscillating reaction	529
	Voltammetry	530
13.26	Fundamentals of voltammetry	530
13.27	Conventional or d.c. polarography	531
13.28	Theoretical principles	533
13.29	Complex ions	537
13.30	Quantitative techniques	538
13.31	The effect of oxygen	540
13.32	Simple polarography and classical d.c. polarography	541
13.33	The three-electrode polarograph: potentiostatic control	543
13.34	Modified voltammetry	546
13.35	Quantitative applications of polarography	551
	Polarography experiments	555
13.36	Polarography experiments: introduction	555
13.37	Half-wave potential of the cadmium ion in 1M KCl	555
13.38	Investigating the influence of dissolved oxygen	557
13.39	Copper and zinc in tap water using DPP	558
13.40	Copper and zinc in tap water using square wave polarography	560
13.41	Ascorbic acid (vitamin C) in fruit juice	560
13.42	Indirect determination of nitrate via <i>o</i> -nitrophenol	561
	Stripping analysis	562
13.43	Stripping voltammetry: basic principles	562
13.44	Electrodes used for stripping analysis	565
13.45	Apparatus for stripping analysis	565
13.46	Determination of lead in tap water	567
13.47	References	568
13.48	Bibliography	569
14	Nuclear magnetic resonance spectroscopy	571
14.1	Introduction	571
14.2	Theory	571

14.3	The chemical shift	573
14.4	Coupling of magnetic nuclei	575
14.5	Instrumentation	576
14.6	Sample preparation	579
	Experimental determinations	580
14.7	Ethanol content of an alcoholic liquor	580
14.8	Ethanol content of a beer by standard addition	581
14.9	Aspirin, phenacetin and caffeine in an analgesic tablet	582
14.10	Keto–enol tautomerism in pentan-2,4-dione (acetylacetone)	583
14.11	References	583
14.12	Bibliography	583
15	Atomic absorption spectroscopy	584
15.1	Introduction	584
15.2	Elementary theory	584
15.3	Instrumentation	588
15.4	Flames	588
15.5	The nebuliser–burner system	589
15.6	Graphite furnace technique	591
15.7	Cold vapour technique and hydride generation	593
15.8	Resonance line sources	595
15.9	Monochromators	596
15.10	Detectors	596
15.11	Interferences	597
15.12	Chemical interferences	598
15.13	Background correction methods	600
15.14	Atomic absorption spectrophotometers	602
	Experimental preliminaries	603
15.15	Calibration curve procedure	603
15.16	Preparation of sample solutions	605
15.17	Preparation of standard solutions	606
15.18	Safety practices	606
15.19	Detection limits	607
	Selected determinations by atomic absorption spectroscopy	610
15.20	Magnesium and calcium in tap water	610
15.21	Vanadium in lubricating oil	612
15.22	Trace elements in contaminated soil	613
15.23	Tin in canned fruit juice	615
15.24	References	615
15.25	Bibliography	616
16	Atomic emission spectroscopy	617
16.1	Introduction	617
16.2	Emission spectra	617
16.3	Flame emission spectroscopy (FES)	619
16.4	Evaluation methods	621
16.5	Evaluating flame emission spectroscopy	621
16.6	Plasma emission spectroscopy	621
16.7	Direct current plasma (DCP)	621

Detailed contents

16.8	Inductively coupled plasma (ICP)	622
16.9	Sample introduction	623
16.10	ICP instrumentation	624
16.11	Evaluating ICP AES	627
16.12	Determining alkali metals by flame photometry	627
16.13	References	628
16.14	Bibliography	628
17	Molecular electronic spectroscopy	630
17.1	General discussion	630
17.2	Theory of spectrophotometry and colorimetry	632
17.3	Fluorimetry (theory)	636
17.4	Methods of 'colour' measurement	638
17.5	Photoelectric photometer method	639
17.6	Wavelength selection	641
17.7	Radiation sources	645
17.8	Standard cells	645
17.9	Data presentation	646
17.10	Instrumental layout	646
17.11	Instruments for fluorimetry	648
17.12	The origins of absorption spectra	649
17.13	Derivative spectrophotometry	650
	Colorimetry	653
17.14	General remarks	653
17.15	Choice of solvent	654
17.16	Colorimetric determinations: general procedure	655
17.17	Enzymatic analysis	658
17.18	Some applications of fluorimetry	659
17.19	Flow injection analysis	660
	Experimental section	
	Cations	663
17.20	Ammonia	663
17.21	Arsenic	664
17.22	Boron	666
17.23	Chromium	668
17.24	Titanium	669
17.25	Tungsten	670
	Anions	671
17.26	Chloride	671
17.27	Phosphate	672
17.28	Sulphate	673
	Organic compounds	674
17.29	Primary amines	674
17.30	Anionic detergents	675
	UV-visible spectrophotometry	676
17.31	Absorption curve and concentration of potassium nitrate	676
17.32	How substituents affect the absorption spectrum of benzoic acid	678

17.33	Simultaneous determinations (chromium and manganese)	679
17.34	Aromatic hydrocarbons and binary mixtures	682
17.35	Phenols in water	683
17.36	Active constituents in a medicine by derivative spectroscopy	684
17.37	Glycerol in fruit juice	685
17.38	Cholesterol in mayonnaise	686
	Fluorimetry	688
17.39	Quinine in tonic water	688
17.40	Codeine and morphine in a mixture	689
17.41	References	689
17.42	Bibliography	690
18	Vibrational spectroscopy	692
18.1	Infrared spectroscopy	692
18.2	Raman spectroscopy	695
18.3	The Raman effect	695
18.4	Correlation between IR and Raman	697
18.5	Infrared instruments	698
18.6	Dedicated process analysers	701
18.7	Infrared cells for liquid samples	703
18.8	Measuring cell path length	704
18.9	Raman instruments	705
18.10	Measuring IR absorption bands	707
18.11	Beer's law: quantitative IR spectra	707
18.12	Measurements using compressed discs	710
18.13	Reflectance methods	711
18.14	GC-FTIR systems	712
18.15	Near-infrared spectroscopy	713
	Experimental determinations	714
18.16	Purity of commercial benzoic acid by compressed discs	714
18.17	A calibration curve for cyclohexane	715
18.18	2-, 3-, 4-Methylphenols (cresols) in a mixture	715
18.19	Propanone (acetone) in propan-2-ol	715
18.20	References	716
18.21	Bibliography	716
19	Mass spectrometry	717
19.1	Introduction	717
19.2	Vacuum systems	719
19.3	Sample inlet systems	721
19.4	The ion source	723
19.5	Mass analysers	731
19.6	Detectors	737
19.7	Data handling	738
19.8	Inorganic mass spectrometry	739
19.9	Isotope ratio measurements	741
19.10	Interpretation of spectra	744
19.11	Hyphenated systems	753

Detailed contents

19.12	Future developments	761
19.13	References	761
19.14	Bibliography	762

Appendices 763

Appendix 1	Relative atomic masses 1994	765
Appendix 2	Aqueous concentrations: common acids and ammonia	766
Appendix 3	Saturated solutions of some reagents at 20 °C	766
Appendix 4	Sources of analysed samples	767
Appendix 5	Buffer solutions and secondary pH standards	767
Appendix 6a	Dissociation constants of some acids in water at 25 °C	769
Appendix 6b	Acidic dissociation constants of bases in water at 25 °C	770
Appendix 7	Polarographic half-wave potentials	771
Appendix 8	Resonance lines for atomic absorption	774
Appendix 9	Common chromophores: electronic absorption characteristics	775
Appendix 10	Characteristic infrared absorption bands	776
Appendix 11	Percentage points of the <i>t</i> -distribution	777
Appendix 12	<i>F</i> -distribution	778
Appendix 13	Critical values of <i>Q</i> (<i>P</i> = 0.05)	779
Appendix 14	Critical values of the correlation coefficient <i>p</i> (<i>P</i> = 0.05)	779
Appendix 15	Wilcoxon signed rank test: critical values (<i>P</i> = 0.05)	779
Appendix 16	Critical values for <i>T_d</i> (<i>P</i> = 0.05)	780
Appendix 17	Critical <i>F_R</i> values for one- and two-tailed tests (<i>P</i> = 0.05)	780
Appendix 18	Equivalents and normalities	780