

COMPREHENSIVE ORGANOMETALLIC ANALYSIS

T. R. Crompton

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PREFACE

It is now some sixteen years since the author's first series of books on the analysis of organometallic compounds. Many developments in the subject have occurred since that time and a new book on the subject is now overdue.

The present book aims to provide a comprehensive review of the subject. It covers not only all aspects of the analysis of organometallic compounds but also contains two additional chapters, dealing with environmental analysis and the use of chelates of metals in the determination of very low concentrations of organic metals.

Whilst reviewing the literature for the present book, it was observed that whereas papers published prior to 1973 dealt almost exclusively with various forms of analysis, a high proportion of those published during the past ten years were concerned with the application of proven or newly developed methods to the determination of organometallic compounds in environmental samples such as water, air, soil, river and ocean sediments, fish life and biota samples. An increasing range of elements including mercury, lead, arsenic, tin, antimony, selenium and manganese are now being found in organically bound forms in the environment, some resulting from pollution, others formed in nature by bacterial processes. As many of these substances have appreciable implications to human and animal health and the ecosystem as a whole, it was considered that it would be timely to include a separate chapter in the book devoted entirely to this subject.

Many elements, upon reaction with particular chelating agents, produce chelates which are amenable to chromatographic analysis and, in many cases, are sufficiently volatile to be gas chromatographed. This has opened up a whole new area of analysis of mixtures of metals at very low concentrations.

In many instances chelate formation-gas chromatographic methods have absolute detection limits several orders of magnitude lower than those achieved by competing techniques such as atomic absorption, neutron activation analysis, emission spectrography and spark-source mass spectrometry.

The purpose of this separate chapter is to gather together the world literature on this new subject so as to enable analytical chemists to take

a strong interest in and develop further, the technique which, in the opinion of the author, has ever greater potential in the future.

The first seven chapters of the book cover each of the major analytical techniques that can be applied to the analysis of organometallic compounds. Within each chapter, the elements are discussed in alphabetical order. The first two chapters cover the determination of elements and functional groups. Succeeding chapters, respectively, cover the applications of titration techniques, visible and ultra-violet spectroscopic techniques, other spectroscopic techniques particularly infrared, NMR, PMR, etc., polarography, gas chromatography and finally a chapter covering other chromatographic techniques.

Errors are inevitable in a work of this size; the author would be grateful to receive notification of any errors so that they can be rectified in future editions.

It is hoped that this book will serve its aim of being a source-book of all aspects of the analysis of organometallic compounds, their occurrence in the environment and their uses in analytical chemistry.

The volumes will interest workers in a wide variety of fields both industrial and academic, at both the pure and the applied ends of the subject. In addition to analytical chemists the book will be of interest to organic chemists and those concerned with the environment and public health. Organometallic chemistry is a field which continues to grow and to which students should direct their interest and it is hoped that they will find much to interest them.

T. R. Crompton

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FIGURE

- 1,2,5,6,7 T.R. Crompton, Chemical Analysis of Organometallic Compounds, Vol. V, Academic Press, London, New York, San Francisco, 1977, Chapter 20, pp. 115-235.
- 3 R. Dijkstra and E.A.M. Dahmen, Z. Anal. Chem., 181, 399 (1961).
- 4 B.J. Phillip, W.L. Mundry, and S.C. Watson, Anal. Chem., 45, 2298 (1973).
- 8 I. Dunstan and J.V. Griffiths, Anal. Chem., 33, 1598 (1961).
- 9 H. Allen and S. Tannenbaum, Anal. Chem., 31, 265 (1959).
- 10 S.A. Greene and H. Pust, Anal. Chem., 30, 1039 (1958).
- 11 J.C. Boaker and T.L. Isenhour, Anal. Chem., 41, 1705 (1969).
- 12 H. Pieters and W.J. Buis, Mikrochem. J., 8, 383 (1964).
- 13,14 R.A. Mostyn and A.E. Cunningham, J. Inst. Pet., 53, 101 (1967).
- 15 B.C. Southworth, J.H. Hodecker, and K.D. Fleischer, Anal. Chem., 30, 1152 (1958).
- 16 I. Lysyj and J.E. Zarembo, Microchem. J., 2, 245 (1958).
- 17,18 B.D. Holt, Anal. Chem., 37, 751 (1965).
- 19 O. Meier and N. Shaltiel, Mikrochim Acta 580 (1960).
- 20 K. Ziegler H. Gellert and Justus Liebigs, Ann. Chem., 629, 20 (1960).
- 21 T.R. Crompton, Analyst (London), 91, 374 (1966).
- 22 D.F. Hagen and W.D. Leslie, Anal. Chem., 35, 814 (1963).
- 23 E. Bonitz, Chem. Ber., 88, 742 (1955).
- 24 M. Farina, M. Donati and H. Ragzzini, Ann. Chim., (Rome), 48, 501 (1958).
- 25 L. Nebbia and B. Pagani, Chim. Ind. (Milan), 44, 383 (1962).
- 26 E.H. Hoffman and W. Tornau, Z. Anal. Chem. 186, 231 (1962).
- 27,28 M. Dimbat and G.A. Harlow, Anal. Chem., 34, 450 (1962).
- 29,30,34 W.L. Everson, Anal. Chem., 36, 854 (1964).
- 31 E.G. Hoffman and W. Tornau, Z., Anal. Chem., 188, 321 (1962).
- 32 G. Pilloni and G. Plazzogna, Anal. Chim. Acta, 35, 325 (1966).
- 33 A.F. Clifford and R.R. Olsen, Anal. Chem., 32, 544 (1960).
- 35 S.C. Watson and J.F. Eastham, Anal. Chem., 39, 171 (1967).

ACKNOWLEDGEMENTS

- 36 M. Dimbat and G.R. Harlow, Anal. Chem., 34, 450 (1962).
- 37 C. Jolibois, R. Acad. Sci., 155, 213 (1912).
- 38,39 G. Tagliavini, Anal. Chim. Acta, 34, 24 (1966).
- 40,41 W.L. Everson and E.M. Ramirez, Anal. Chem., 37, 812 (1965).
- 42,43 E. Bonitz, Chem. Ber., 88, 742 (1955).
- 44 W.P. Neumann, Angew. Chem., 69, 730 (1957).
- 45 J.H. Mitchen, Anal. Chem., 33, 1331 (1961).
- 46,47,48 B.T. Commins and P.J. Lawther, Br. J. Ind. Med., 22, 139 (1965).
- 49 J. Stary, K. Kratzer and K.J. Prasilova, Anal. Chim. Acta, 100, 627 (1978).
- 50 S. Sasse, W.D. Ludemann, B. Witten, V. Fisien, A.J. Sisti, and J.I. Miller, Anal. Chem., 29, 1346 (1957).
- 51 K.S. Pitzer and R.J. Sheline, J. Chem. Phys., 16, 552 (1948).
- 52,53,61 F.G. Hoffman, Z. Elektrochem., 64, 616 (1960).
- 54,155 E.J. Hoffman and G. Schomberg, Z. fur Elecktrochemie 61, 1101 (1957).
- 56 E.J. Hoffman and G. Schomberg, Z. fur Elecktrochemie, 61, 1110 (1957).
- 57 J.V. Pell, J. Heisler, H. Tannenbaum, and J. Goldenson, Anal. Chem., 25, 1720 (1953).
- 58 D.V. Guertin, S.E. Wiberley, W.H. Bauer, and J. Golderson, J. Phys. Chem., 60, 1018 (1956).
- 59 V. Yamamoto, Bull. Chem. Soc. Japan, 35, 619 (1962).
- 60 J. Smidt, M.P. Gruenewage, and H. de Vries, Rec. Trav. Chim., 81, 729 (1962).
- 62 J.J. Kaufman, W.S. Koski, L.T. Kuhns, and S.S. Wright, J. Amer. Chem. Soc., 85, 1369 (1963).
- 63,64 W.S. Koski, J.J. Kaufman, and P.C. Lantebur, J. Amer. Chem. Soc., 79, 2382 (1957).
- 65 A.F. Reid, D.Z. Scaife, and P.C. Wailes, Spectrochim. Acta, 20, 1257 (1964).
- 66 J.K. Urwin and T.J. Reed, J. Organometal Chem., 15, 1 (1968).
- 67,68 H.Susi and H.E. Rector, Anal. Chem., 30, 1933 (1958).
- 69 J.H. Lowry, R.B. Smart, and K.H. Nancy, Anal. Chim., 50, 1303 (1978).
- 70 J.E. de Vries, A. Laauw-Zecha, and A. Pellecer, Anal. Chem., 31, 1995, (1959).
- 71 M. Mehner, H. Jehring, and H. Kriegsmann, 3rd Analytical Conference, Budapest, 24-29 August, 1970.
- 72,73 R. Guyer and H.T. Seidlitz, Z. Chem., 4, 468 (1964).
- 74 L.M. Brown and K.S. Mazdiyansi, Anal. Chem., 41, 1243 (1969).
- 75,76,79 B. Gudzinowicz and J.L. Driscoll, J. Gas Chromatogr., 1, 25 (1963).
- 77,78 G.E. Perris, W.R. Blair, and J.E. Brinkman, Anal. Chem., 49, 2215 (1977).
- 80,81,82 C. Feldman and D.A. Batistoni, Anal. Chem. 49, 2215 (1977).
- 83 G. Schomberg, R. Koster, and D. Henneberg, Z. Anal. Chem., 170, 285 (1959).

- 84 G.R. Seely, J.P. Oliver, and D.M. Ritter, Anal. Chem., 31, 1993 (1959).
- 85 T.D. Parsons, M.B. Silverman, and D.M. Ritter, J. Am. Chem. Soc., 79, 5091 (1957).
- 86 J.A. Semlyen and C.S.G. Phillips, J. Chromatography, 18, 1 (1965).
- 87,88 J.J. Kaufman, J.E. Todd, and W.S. Koski, Anal. Chem., 29, 1032 (1957).
- 89,90 H.W. Myers and R.F. Putman, Anal. Chem., 34, 664 (1962).
- 91,92 H. Veening, J. Graver, D.B. Clark, and B.R. Willeford, Anal. Chem., 41, 1655 (1969).
- 93 H. Veening, J.S. Keller, and B.R. Willeford, Anal. Chem., 43, 1516 (1971).
- 94 W.J.A. Van der Heuvel, J.S. Keller, H. Veening, and B.R. Willeford, Analyt. Lett., 3, 279 (1970).
- 95 C.S.G. Phillips and P.L. Timms, Anal. Chem., 35, 505 (1963).
- 96 B. Iatridis and G. Parissakis, Anal. Chem., 49, 909 (1977).
- 97 O.E. Ayers, T.C. Smith, J.D. Burnett, and B.W. Pouder, Anal. Chem., 38, 1606 (1966).
- 98,99,100 J.E. Lovelock and A. Zlatkis, Anal. Chem. 33, 1958 (1961).
- 101,102 H.J. Dawson, Anal. Chem., 35, 542 (1963).
- 103 E. Barrall and P. Ballinger, J. Gas Chromatography, 1, 7 (1963).
- 104 E. Bonnelli and H. Hartmann, Anal. Chem., 35, 1980 (1963).
- 105,106 N.L. Soulages, Anal. Chem., 38, 28 (1966).
- 107 W.S. Leonhardt, R.C. Morrison, and W.C. Kamicnski, Anal. Chem., 38, 466 (1966).
- 108 L.V. Giold, C.A. Hollingsworth, D.H. McDaniel, and J.H. Wotiz, Anal. Chem., 33, 1156 (1961).
- 109 A. Wowk and S. Di. Giovanni, Anal. Chem., 38, 742 (1966).
- 110,111 P.C. Uden, R.M. Barnes, and P. Di. Sanzo, Anal. Chem., 50, 852, (1978).
- 112,113 B.D. Quimby and P.C. Uden, Anal. Chem., 50, 2112 (1978).
- 114 A. Apelblat and A. Hornik, J. Chromatography, 24, 175 (1966).
- 115 A. Apelblat and A. Hornik, Trans Faraday Soc. No., 529, 63, 185 (1967).
- 116 A. Apelblat, J. Inorg. Nucl. Chem., 31, 483 (1969).
- 117 R.L. Grob and G.L. McCrea, Anal. Lett., 1, 55 (1967).
- 118 K.P. Berlin, T.H. Austin, M.E. Nagahushanam, J. Peterson, J. Calvert, W.A. Wilson, and D. Hopper, J. Gas Chromatogr., 3, 256 (1965).
- 119 A. Davis, A. Roadi, J.G. Michalovic, and A.M. Joseph, J. Gas Chromatography, 1, 23 (1963).
- 120 C.S. Evans and C.M. Johnson, J. Chromatography, 21, 202 (1966).
- 121,122 C.S.G. Phillips, P.L. Timms, Anal. Chem., 35, 505 (1963).
- 123 F.H. Pollard, G. Nickless, and P.C. Uden, J. Chromatogr., 19, 28 (1965).
- 124 H. Rotzsche, Z. Anorg. Chem., 324, 197 (1963).

- 125 H. Rotzsche, Z. fur Anorg. und Allgemeine. Chemie., 328, 79 (1964).
- 126 M. Wurst, Coll. Czech. Chem. Commun., 30, 2038 (1965).
- 127 J.B. Carmichael, D.J. Gordon, and C.E. Ferguson, J. Gas Chromatography, 4, 347 (1966).
- 128,129,130 M.A. Osman, H.H. Hill, M.W. Holdren, and H.H. Wetberg, Anal. Chem., 51, 1286 (1979).
- 131,132,133 G. Garzo and F. Till, Talanta, 10, 583 (1963).
- 134 D. Thrasiv L. Viosinet and K.E. William, J. Gas Chromatography July 248 (1965).
- 135,136 F.H. Pollard, G. Nickless, and D.B. Thomas, J. Chromatogr., 22, 286 (1966).
- 137 D.D. Schleuter and S. Siggia, Anal. Chem., 49, 2343 (1977).
- 138 D.D. Schleuter, Ph.D. Dissertation, University of Massachusetts, Amherst, Massachusetts (1976).
- 139,140 F.H. Pollard, G. Nicklett, and D.J. Cooke, J. Chromatography, 12, 48 (1964).
- 141 R.L. Putnam and F. Put, J. Gas Chromatography, 2, 2 (1965) and 3, 2 (1965).
- 142 H. Geissler and H. Kriegsmann, Z. Chemie, Lpz., 2, 354 (1964).
- 143,144 P.L. Tonge, J. Chromatography, 19, 182 (1965).
- 145,146,147 S.G. Perry, J. Gas Chromatography, 93 March (1964).
- 148 J.E. Schwarberg, R.W. Moshier, and J.H. Walsh, Talanta, 11, 1213 (1964).
- 149 J.E. Schwarberg, Master's thesis, University of Dayton, 1964.
- 150 P. Jacquelin and C. Thomas, Bull Soc. Chim. Fr. 702 (1971).
- 151 H. Veening and J.F.K. Huber, J. Gas Chromatog., 6, 326 (1968).
- 152,153 J.A. Stokeley, Master's thesis, Oak Ridge University, Diss. Abstr., 27, 1388 B (1966).
- 154 K.J. Eisentraut and R.E. Sievers, J. Am. Chem. Soc., 87, 5254 (1965).
- 155 Anonymous, Chem. Eng. News, 43, November 22, 39 (1965).
- 156 T. Shigematsu, M. Matsui, and K. Utsunomiya, Bull. Inst. Chem. Res., Kyoto Univ., 46, 256 (1968).
- 157 W.I. Stephen, I.J. Thompson and P.C. Uden, Chem. Commun., pp. 269-270 (1969).
- 158 A. Khalique, W.I. Stephen, P.E. Henderson, and P.C. Uden, Anal. Chim., Acta, 101, 117 (1978).
- 159 T. Fujinaga and Y. Ogino, Bull. Chem. Soc., Japan, 40, 434 (1967).
- 160 J.K. Foreman, T.A. Gough, and E.A. Walker, Analyst (London), 95, 797 (1970).
- 161 R. Belcher, C.R. Jenkins, W.I. Stephen, and P.C. Uden, Talanta, 17, 455 (1970).
- 162,163 T.J. Cardwell, D.J. Resarro, and P.C. Uden, Anal. Chim. Acta, 85, 415 (1976).
- 164 Y. Shimoishi and K. Toei, Anal. Chim. Acta, 100, 65 (1978).

- 165 E.G. Gaetani, C.F. Laureri, A. Magnia, and G. Parolari, *Anal. Chem.*, 48, 1725 (1976).
- 166,167 T.J. Cardwell, D. Caridi, and M.S. Loa, *J. Chromatography*, 351, 331 (1986).
- 168 M. Saitoh, R. Kurada, and M. Shibukawa, *Anal. Chem.*, 55, 1025 (1983).
- 169 K. Saitoh, M. Kabayashi, and N. Suzuki, *Anal. Chem.* 53, 2309 (1981).
- 170 C.R. Ricci, L.S. Shepard, G. Colovos, and N.H. Hester, *Anal. Chem.*, 53, 610 (1981).
- 171 J.D. Messman and T.C. Rains, *Anal. Chem.*, 53, 1632 (1981).
- 172 C. Misson, *Chem. Ztg.*, 32, 633 (1908).
- 173 R.B. Lew and F. Jakob, *Talanta*, 10, 322 (1963).
- 174 Department of the Environment and National Water Council (U.K.)
H.M. Stationary Office, London, 23 pp (pt 23 Abenv) (1978).
- 175 K. Minagawa, Y. Takizawa, and I. Kufune, *Anal. Chim. Acta*, 103, 115 (1980).
- 176,177 K. Chiba, K. Yoshida, K. Tanabe, H. Horaguchi, and K. Fuwa, *Anal. Chem.*, 55, 450 (1983).
- 178 G.A. Hambrick, P.N. Froebich, O.A. Meirate, and B.L. Lewis, *Anal. Chem.*, 56, 421 (1984).
- 179 Y.K. Chau, P.T.S. Wong, and G.A. Bengert, *Anal. Chem.*, 54, 246 (1982).
- 180 C.I. Soderquist and D.G. Crosby, *Anal. Chem.* 50, 1435 (1978).
- 181,182,183 R.S. Raman and M.A. Tomkins, *Anal. Chem.*, 51, 12 (1979).
- 184 Y.K. Chau, P.T.B. Wong, and U. Kramar, *Anal. Chem. Acta*, 146, 211 (1983).
- 185,186 M.O. Andreea, *Anal. Chem.*, 49, 820 (1977).
- 187 A.A. Grabinski, *Anal. Chem.*, 53, 966 (1981).
- 188,189 J. Aggett, R. Kadwani, *Analyst (London)*, 108, 1495 (1983).
- 190 C.E. Stringer and M. Attrep, *Anal. Chem.*, 51, 731 (1979).
- 191,192 L. Brown, S.J. Haswell, H.M. Rhead, P.O'Neill, and C.C. Bancroft, *Analyst*, 108, 1511 (1983).
- 193,194,195 Y.K. Chau, P.T.S. Wong, and H. Saitoh, *J. Chromatogr. Sci.*, 14, 162 (1976).
- 196,197,198 S. Hanamura, B.W. Smith, and J.D. Winefordner, *Anal. Chem.*, 55, 2026 (1983).
- 199,200,201 J.G. Gonzales, and R.T. Ross, *Anal. Lett.*, 5, 683 (1972).
- 202 W.A. MacCrehan, R.A. Durst, and J.M. Bellama, *Anal. Lett.*, 10, 1175 (1977).
- 203,204 C.J. Cappon and V. Crispin Smith, *J. Anal. Chem.*, 49, 365 (1977).
- 205 M. Morita, T. Uehiro, K. Fuwa, *Anal. Chem.*, 53, 1806 (1981).
- 206 M. Loe, R. Cruz, and J.C. Van Loon, *Anal. Chim. Acta*, 120, 171 (1980).
- 207 G. Torsi, F. Palmisano, *Analyst (London)*, 108, 1318 (1983).
- 208 G. Torsi, E. Desimoni, F. Palmisano, *Analyst (London)*, 107, 96 (1982).
- 209 J.W. Robinson, E.L. Kicsel, J.P. Goodbread, R. Bliss, and R. Marshall, *Anal. Chim. Acta*, 92, 321 (1977).

- 210,211 A.J. McCormack, S.C. Tong, and W.D. Cooke, *Anal. Chem.*, 37,
1470 (1965).
- 212 W.R.A. De Jonghe, D. Chakraborti, and F.C. Adams, *Anal. Chem.*, 52,
1974 (1980).
- 213 H. Koizumi, R.D. McLaughlin, and T. Hadeishi, *Anal. Chem.*, 51,
387 (1979).
- 214,215 R. Moss and E.V. Browett, *Analyst (London)*, 91, 428 (1966).

CONTENTS

CHAPTER 1 DETERMINATION OF ELEMENTS AND FUNCTIONAL GROUPS

Organaluminium Compounds	1
A. Determination of Aluminium (1)	
B. Determination of Carbon and Hydrogen (3)	
C. Determination of Aluminium-bound Halogens (4)	
D. Determination of Aluminium-bound Alkyl Groups up to Butyl and Hydride Groups (5)	
E. Determination of Higher Aluminium-bound Alkyl Groups (21)	
F. Determination of Higher Alkyl and Alkoxide Groups (22)	
G. Determination of Aluminium-bound Alkoxide Groups up to Butoxy (23)	
H. Determination of Aluminium-bound Amino Groups (26)	
I. Determination of Aluminium-bound Thioalkoxide Groups (26)	
Organoantimony Compounds	27
A. Determination of Antimony (27)	
Organotin Compounds	28
A. Determination of Arsenic (28)	
B. Determination of Carbon (33)	
C. Determination of Fluorine (33)	
D. Determination of Sulphur (33)	
Organoberyllium Compounds	33
A. Determination of Carbon and Hydrogen (33)	
Organobismuth Compounds	34
A. Determination of Bismuth (34)	
Organoboron Compounds	34
A. Determination of Boron, Carbon and Hydrogen (34)	
B. Determination of Chlorine (44)	
C. Determination of Nitrogen and Boron (46)	
D. Determination of Hydride and Active Hydrogen (48)	
Organocalcium Compounds	48
A. Determination of Carbon and Hydrogen (48)	
Organochromium Compounds	50
A. Determination of Chromium (50)	
Organocobalt Compounds	51
A. Determination of Cobalt (51)	
Organocupper Compounds	53
A. Determination of Copper (53)	

Organogermanium Compounds	53
A. Determination of Germanium (5)	
B. Determination of Carbon and Hydrogen (54)	
C. Determination of Alkoxy Germanes and Methylcyclogermanes (54)	
Organoiron Compounds	55
A. Determination of Iron (.8)	
Organolead Compounds	59
A. Determination of Lead (59)	
B. Determination of Organolead Compounds in Petroleum in the determination of Lead (59)	
C. Determination of Carbon and Hydrogen (69)	
D. Determination of Halogen (69)	
Organolithium Compounds	69
A. Determination of Lithium, Carbon, Hydrogen and Oxygen (69)	
Organomagnesium Compounds	70
A. Determination of Carbon and Hydrogen (70)	
B. Determination of Alkyl Groups (70)	
Organomanganese Compounds	70
A. Determination of Manganese (70)	
Organomercury Compounds	70
A. Determination of Mercury (70)	
B. Determination of Carbon, Sulphur, Halogens and Oxygen (75)	
C. Determination of Carbon, Hydrogen and Mercury (78)	
D. Determination of Oxygen (8)	
Organonickel Compounds	82
A. Determination of Nickel (82)	
Organopalladium Compounds	82
A. Determination of Palladium and Chlorine (82)	
Organophosphorus Compounds	83
A. Determination of Phosphorus (83)	
B. Determination of Iron, Silicon, Titanium, Arsenic and Phosphorus (98)	
C. Determination of Carbon and Hydrogen (98)	
D. Determination of Nitrogen (101)	
E. Determination of Oxygen (101)	
F. Determination of Halogens (102)	
G. Determination of Sulphur (108)	
Organoplatinum Compounds	108
Organopotassium Compounds	108
Organoselenium Compounds	109
A. Determination of Selenium (109)	
B. Determination of Carbon and Hydrogen (111)	
Organosilicon Compounds	111
Determination of Elements (111)	
A. Determination of Silicon (111)	
B. Determination of Phosphorus and Silicon (119)	

CONTENTS

C. Determination of Aluminium and Silicon	122
D. Determination of Titanium, Silicon, Phosphorus and Aluminium (124)	
E. Determination of Tin, Silicon and Titanium (125)	
F. Determination of Hydrogen, Silicon and Halogen (125)	
G. Determination of Halogens (125)	
Determination of Functional Groups (127)	
H. Determination of Alkoxy and Aryloxy Groups (131)	
I. Determination of Acetoxy Groups (133)	
J. Determination of Silicon-bound Hydroxyl Groups (134)	
K. Determination of Mercaptosilanes (137)	
L. Determination of Silylidyne Groups (138)	
M. Determination of Unsaturation (140)	
N. Determination of Phenyl and Vinyl Groups (140)	
O. Determination of Hydrocarbon Constituents (141)	
P. Non-Aqueous Titrimetry (141)	
Organothallium Compounds	142
A. Determination of Carbon, Hydrogen and Thallium (142)	
Organotin Compounds	143
A. Determination of Tin (143)	
B. Determination of Carbon and Hydrogen (144)	
C. Determination of Halogens (145)	
D. Determination of Nitrogen (145)	
E. Determination of Sulphur (145)	
Organotitanium Compounds	145
A. Determination of Titanium, Iron, Phosphorus, Silicon and Chlorine (145)	
Organozinc Compounds	146
A. Determination of Zinc (146)	
B. Determination of Halogens (146)	
C. Determination of Zinc-bound Alkoxy Groups (147)	
D. Determination of Lower Alkyl and Hydride Groups (147)	
Organozirconium Compounds	148
CHAPTER 2 TITRATION PROCEDURES	
Organoaluminium Compounds	149
A. Classical Titration Procedures (149)	
B. Conductiometric Titration (157)	
C. Potentiometric Titration (160)	
D. Amperometric Titration (164)	
E. Activity by Dielectric Constant Titration (164)	
F. Lumometric Titration (165)	
G. Thermometric Titration (167)	
Organarsenic Compounds	172
Organoboron Compounds	173
A. Classical Titration Procedures (173)	
B. Potentiometric Titration (174)	
Organocalcium Compounds	174
Organocobalt Compounds	174

CONTENTS

Organocopper Compounds	174
Organogermanium Compounds	175
Organoiron Compounds	175
A. Classical Titration Procedures (175)	
B. Potentiometric Titration (175)	
Organolead Compounds	176
A. Classical Titration Procedures (176)	
B. Amperometric Titration (180)	
C. Conductiometric Titration (180)	
D. Coulometric Titration (180)	
E. High frequency Titration (181)	
Organolithium Compounds	181
A. Classical Titration Procedures (181)	
B. Potentiometric Titration (188)	
C. Thermometric Titration (189)	
D. High frequency Titration (191)	
E. Lumometric Titration (193)	
Organomagnesium Compounds	193
A. Classical Titration Procedures (193)	
B. Amperometric Titration (195)	
C. Potentiometric Titration (195)	
Organomercury Compounds	196
A. Classical Titration Procedures (196)	
B. Potentiometric Titration (197)	
C. Coulometric Titration (198)	
Organophosphorus Compounds	198
A. Esters of Phosphoric Acid (198)	
B. Esters of Phosphorous Acid (199)	
C. Esters of Phosphonic, Phosphinic and Phosphorous Acids (200)	
D. Esters of Pyrophosphoric Acid, Pyrophosphonic Acid, Phosphoro-anhydrides, Alkyl-phosphonochlorides, Dialkylphosphorochlorides and Alkylphosphonofluorides (200)	
E. Organophosphorus Fluorides and Chlorides (204)	
F. Phosphonium Salts and Phosphoranes (206)	
G. Substituted Phosphines (208)	
H. Trisubstituted Phosphine Oxides and Sulphides and bis (disubstituted phosphinyl) Alkanes (209)	
I. Dialkylphosphorodithioates, Dialkyldithiophosphates, Phosphoro-thioates, Dialkylphosphonodithioates and other P - S Compounds (209)	
J. Alkyl Phosphonic Chlorides	
K. Alkyl Silylphosphates	
Organopotassium Compounds	212
Organoselenium Compounds	212
Organosodium Compounds	212
Organosilicon Compounds	212
A. Non-aqueous Titration (212)	
B. Non-aqueous Conductiometric Titration (213)	
C. Non-aqueous Amperometric Titration (213)	

Organic Compounds	215
A. Classical Titration Procedures (215)	
B. Potentiometric Titration (217)	
C. Amperometric and Coulometric Titration (217)	
Organozinc Compounds	221
A. Classical Titration Procedures (221)	
B. Thermometric Titration (222)	
CHAPTER 3 SPECTROSCOPIC TECHNIQUES, VISIBLE AND ULTRAVIOLET SPECTROSCOPY	
A. Visible Spectroscopy	226
Organ-aluminium Compounds (226)	
Organarsenic Compounds (232)	
Organoboron Compounds (233)	
Organochromium Compounds (234)	
Organocobalt Compounds (235)	
Organic copper Compound (238)	
Organogermanium Compounds (238)	
Organoliron Compounds (233)	
Organolead Compounds (245)	
Monodi- and Trisubstituted Lead Compounds (245)	
Tetrasubstituted Lead Compounds (248)	
Organomagnesium Compounds (249)	
Organomanganese Compounds (253)	
Organomercury Compounds (253)	
Organonickel Compounds (251)	
Organophosphorus Compounds (262)	
Tialkylphosphates (27)	
Dialkyl Hydrogen Phosphites, Tialkyl and Triaryl Phosphites (262)	
O,O-Dialkyl Hydroxyphosphates (264)	
Tetraethyl Pyrophosphate (265)	
Phosphoroponoxides (265)	
Phosphorus Salts (26)	
Substituted Phosphine Oxides (26)	
Alky Chlorides and Fluorides and Alkyl-phosphoroamido-cyanate (268)	
Dialkylithio phosphoric Acids (272)	
Glycophosphates (273)	
Organoboron Compounds (274)	
Organosilicon Compounds (274)	
Organothallium Compounds (274)	
Organotin Compounds (274)	
Organozinc Compounds (280)	
B. Ultraviolet Spectroscopy	281
Organoboron Compounds (281)	
Organolithium Compounds (281)	
Organomagnesium Compounds (281)	
Organomercury Compounds (281)	
Organosilicon Compounds (281)	
Organotin Compounds (282)	
CHAPTER 4 NUCLEAR MAGNETIC SPECTROSCOPIC TECHNIQUES	
Organoaluminium Compounds	283
A. Infrared Spectroscopy (283)	
B. Raman Spectroscopy (286)	
C. PMR and NMR Spectroscopy (301)	
D. Mass Spectrometry (307)	

Organoboron Compounds	308
A. Infrared Spectroscopy (308)	
B. NMR Spectroscopy (311)	
C. Mass Spectrometry (312)	
Organochromium Compounds	313
Mass Spectrometry (313)	
Organocobalt Compounds	313
Infrared Spectroscopy (313)	
Organocupper Compounds	313
Mass Spectrometry (313)	
Organogallium Compounds	313
Electron probe Microanalysis (313)	
Organogermanium Compounds	314
A. Infrared Spectroscopy (314)	
B. Mass Spectrometry (314)	
Organoiron Compounds	314
A. Infrared Spectroscopy (314)	
B. NMR, PMR and ESR (317)	
C. Mass Spectrometry (318)	
D. X-Ray Diffraction and X-Ray Emission (318)	
Organolead Compounds	319
Infrared Spectroscopy (319)	
Organolithium Compounds	319
NMR Spectroscopy (319)	
Organomanganese Compounds	320
Infrared Spectroscopy (320)	
Organomercury Compounds	321
Infrared Spectroscopy (321)	
Organonickel Compounds	321
Mass Spectrometry (321)	
Organophosphorus Compounds	321
A. Infrared Spectroscopy (321)	
B. Mass Spectrometry (321)	
C. NMR Spectroscopy (323)	
Organosilicon Compounds	323
A. Infrared Spectroscopy (323)	
B. Specific Functional Groups (326)	
C. Mass Spectrometry (338)	
D. Raman Spectroscopy (338)	
E. NMR Spectroscopy (339)	
Organotin Compounds	339
A. Infrared and Raman Spectroscopy (339)	
B. NMR Spectroscopy (341)	
C. PMR Spectroscopy (342)	
D. Mass Spectrometry (342)	