

Contents

1	Introduction	1
1.1	Introduction	3
	<i>J. Derek Woollins</i>	
1.2	General Spectroscopic Techniques and Report Writing	5
	<i>Petr Kilian</i>	
2	Introductory Experiments	23
2.1	Preparation and Investigation of Some Coordination Compounds	25
	<i>Manfred Bochmann</i>	
2.2	Gravimetric Analysis of Hexaamminenickel(II) Tetrafluoroborate	
	$[\text{Ni}(\text{NH}_3)_6](\text{BF}_4)_2$	28
	<i>Manfred Bochmann</i>	
2.3	Analysis of an Iron(III) Oxalate Complex	33
	<i>Manfred Bochmann</i>	
2.4	Characterisation of a Copper Oxalate Complex	
	$\text{K}_a\text{Cu}_b(\text{C}_2\text{O}_4)_c \cdot d\text{H}_2\text{O}$	38
	<i>Andrew W. G. Platt</i>	
2.5	Synthesis of $[\text{NH}_4][\text{BF}_4]$ and Synthesis and Titrimetric Analysis of $[\text{Zn}(\text{NH}_3)_4][\text{BF}_4]_2$	40
	<i>Michael A. Beckett</i>	
2.6	Iron(II) and Lead(II) Formates	43
	<i>Francisco J. Arndíz, María R. Pedrosa, and Rafael Aguado</i>	

- 2.7 **Ammonium Dichromate, Chromium(III) Oxide,
Potassium Chromate and Potassium Tetraperoxo-chromate(V)** 47
Antonín Růžička and Zdirad Žák

- 2.8 **Vanadium Alum: An Experiment in Electrosynthesis** 51
David T. Richens

- 2.9 **Linkage Isomerism: An Infra-Red Study** 54
David T. Richens and Christopher Glidewell

- 2.10 **Preparation of Copper(I) Iodide** 56
F. J. Arnáiz, M. R. Pedrosa, and S. Arnáiz

- 2.11 **Deduction of the Coordination Mode of BH₄ in
Bis(triphenylphosphine) Copper(I) Borohydride (Ph₃P)₂CuBH₄** 58
Andrew W. G. Platt

- 2.12 **Preparation and Identification of a Copper(I) Complex** 61
Andrew W. G. Platt

- 2.13 **Synthesis and Characterisation of a Metal Hydride Complex** 63
Andrew W. G. Platt

- 2.14 **Reactivity of Triphenylphosphine and Its Oxide Towards CuCl₂** 65
Paul F. Kelly and Martin B. Smith

- 2.15 **Synthesis of a Thermochromic Copper Complex** 68
Iain A. Smellie

- 2.16 **Preparation and Complexation of
Tris(3,5-dimethylpyrazoyl)hydroborate** 70
Manfred Bochmann

- 2.17 **Tetraiodotin(IV) and its Triphenylphosphine Complex** 73
Iain A. Smellie and J. Derek Woollins

- 2.18 **Silicon Oxygen Compounds and Siloxane Polymers** 75
Paul D. Lickiss

- 2.19 **Preparation and NMR Identification of Two Phosphorus Esters** 79
Christopher Glidewell

- 2.20 Synthesis of Triphenylphosphine Oxide and Triphenylphosphine Sulfide 81**
Iain Smellie
- 2.21 Preparation of Sodium Trithionate, $\text{Na}_2\text{S}_3\text{O}_6$ and Potassium Trithionate, $\text{K}_2\text{S}_3\text{O}_6$ 86**
Andrew G. Platt
- 2.22 The Preparation of Potassium Peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$ 89**
Zdirad Žák and Antonín Růžička
- 2.23 Polyiodides, Me_4NI_x 92**
Andrew W. G. Platt
- 2.24 Interhalogen Compounds 94**
Ivan P. Parkin
- 2.25 Preparation of $\text{K}[\text{ICl}_4]$ 99**
Iain Smellie
- 2.26 Synthesis and Infrared Spectroscopic Characterisation of Boranes and Germanes 101**
Ivan P. Parkin
- 3 Intermediate Experiments**
- 3.1 Metal Acetylacetonate Complexes: Preparation and Characterisation 109**
Christopher Glidewell
- 3.2 Nickel Dihalide Phosphine Complexes 120**
Ivan P. Parkin
- 3.3 Nickel(II) Complexes of Some Schiff Base Ligands 124**
Christopher J. Jones
- 3.4 The Synthesis and Coordination Chemistry of Macrocyclic Complexes 131**
Martin Schröder
- 3.5 $[\text{Co}(\text{dinosar})]\text{Cl}_3$: An Encapsulation Complex Prepared by a Template Reaction 136**
David J. Otway

- 3.6 Identification of Stereochemical (Geometrical) Isomers of $[\text{Mo}(\text{CO})_4(\text{L})_2]$ by Infrared Spectroscopy 139
Michael A. Beckett
- 3.7 Five Coordinate Complexes: $[\text{VO}(\text{acac})_2]$ and $[\text{Cr}(\text{NH}_3)_6][\text{CuCl}_5]$ 142
Christopher P. Morley
- 3.8 Preparation of *trans*- $\text{PtHCl}(\text{PPh}_3)_2$ and Measurement of its ^1H , ^{31}P and ^{195}Pt NMR Spectra 145
William P. Griffith
- 3.9 $[\text{MoO}_2\text{Br}_2(\text{H}_2\text{O})_2] \cdot (\text{diglyme})$ and $[\text{MoO}_2\text{Br}_2(\text{DMF})_2]$ 149
Francisco J. Arnáiz, María R. Pedrosa, and Rafael Aguado
- 3.10 Preparation of an Iron Dinitrogen Complex 153
Andrew W. G. Platt
- 3.11 Preparation of Nitrosyl Complexes of Iron and Nickel 155
Andrew W. G. Platt
- 3.12 Synthesis and Characterisation of Two Metal–Metal Bonded Dimolybdenum Complexes 158
William Levason, Thomas A. Logothetis, and Gill Reid
- 3.13 Cobalt Complexes of Dioxygen 167
David T. Richens and Christopher Glidewell
- 3.14 Ferrocene, $(\eta^5\text{-C}_5\text{H}_5)_2\text{Fe}$, and its Derivatives 170
J. Derek Woollins
- 3.15 The Use of Organolithium Reagents in the Preparation of Ferrocene Derivatives 173
Ian R. Butler
- 3.16 Complexes of π -Bonding Arene Ligands 180
Andrew W. G. Platt
- 3.17 Organotin Chemistry 183
Kieran C. Molloy and Timothy T. Paget
- 3.18 Transition Metal-Carbon Bonds in Chemistry and Biology 190
Christopher J. Jones

- 3.19 Nickel-Catalysed Cross-Coupling of Alkylmagnesium with Haloarene** 201
G. Brent Young
- 3.20 Transition Metal Catalysis** 205
Neil M. Boag
- 3.21 Nitration of Cobalt(III) Acetylacetonate** 209
David J. Otway
- 3.22 Phenyllithium and Tetraphenylsilane** 211
Petr Kilian
- 3.23 Synthesis and Reactivity of Tertiary Phosphines** 218
Neil M. Boag
- 3.24 Two-stage Synthesis of $\text{Ph}_2\text{P}(\text{O})(\text{CH}_2)_4\text{P}(\text{O})\text{Ph}_2$** 222
Andrew G. Platt
- 3.25 Inorganic Heterocycles: Cyclophosphazenes** 226
Josef Novosad and Milan Alberti
- 3.26 Inorganic (Carbon-Free) Chelate Rings: A Dithioimidodiphosphinato Ligand and Some of its Metal Complexes** 229
Ionel Haiduc
- 3.27 Inorganic (Carbon-Free) Chelate Rings II: Asymmetric Mixed-Donor P-N-P Ligand and its Palladium Complex** 234
David J. Birdsall and Josef Novosad
- 3.28 Amidosulfuric Acid, Trisilveramidosulfate Monohydrate and $\text{SO}_3 \cdot \text{N}(\text{CH}_3)_3$ Complexes** 239
Antonín Růžička and Zdirad Žák
- 3.29 Anomalous Paramagnetism in Some Iron(III) Chelates Studied by the Evans NMR Method** 243
David J. Otway
- 3.30 Cubic Molecular Aluminophosphonates** 246
Jiri Pinkas

- 3.31 Preparation and Optical Properties of Zinc Sulfide Quantum Dots 249**
Andrew L. Hector
- 3.32 Four Methods for Analysing First Order Kinetics 255**
Véronique Pimienta, Dominique Lavabre, Jean-Claude Micheau, and Gaston Levy
- 3.33 Molecular Orbital Calculations of Inorganic Compounds 261**
Carole A. Morrison, Neil Robertson, Andrew Turner, Jano van Hemert, and Jos Koetsier
- 4 Advanced Experiments**
- 4.1 2,2':6',2''-Terpyridine Complexes and Metal Directed Reactivity 271**
Edwin C. Constable
- 4.2 4'-(4-Pyridyl)-2,2':6',2''-terpyridine: Coordination to Iron(II) and Protonation Studies 274**
Catherine E. Housecroft, Edwin C. Constable, and Emma L. Dunphy
- 4.3 Heteronuclear d–f Complexes Containing Binucleating Ligands 283**
Francisco J. Arnáiz, Jean-Pierre Costes, and Javier García-Tojal
- 4.4 Electronic Characterisation of a Transition Metal Complex Using Electrochemical UV/Vis and EPR Techniques 287**
Lesley J. Yellowlees
- 4.5 Chlorotris(tert-butylimido)manganese(VII) 290**
Andreas A. Danopoulos and Geoffrey Wilkinson
- 4.6 Tetrakisphosphorane Iminato Complexes of Transition Metals 293**
Kurt Dehnicke
- 4.7 Magnetochemistry Experiments: Exchange Coupling in Copper(II)-Nickel(II) Bimetallic Complexes and Spin Transition in Iron Complexes 297**
Eugenio Coronado and J. J. Borrás-Almenar

- 4.8 Metal-Containing Liquid Crystals.**
The Synthesis of *trans*-bis(4-Alkyl-4'-cyano-1,1'-biphenyl)dichloroplatinum(II) and Related Species –
The Use of Melt Syntheses 301
Duncan W. Bruce
- 4.9 Structural, Electrical and Magnetic Properties**
of Perovskite Ceramics 309
Colin Greaves
- 4.10 Polyoxomolybdate Clusters: Nanoscopic Wheels and Balls 315**
Leroy Cronin, Ekkehard Diemann, and Achim Müller
- 4.11 Reaction of Alkali Metal Polyselenides and Polytellurides**
with Group 6 Metal Carbonyls 322
Joseph W. Kolis
- 4.12 Chlorothionitrene and Cyclothiazene Complexes of Tungsten 327**
Kurt Dehnicke
- 4.13 Sulfur-Rich Homocycles and Heterocycles:**
Titanocene Pentasulfide, *cyclo*-Heptasulfur and
1,2,3,4,5,6,7-Heptathionane 330
Jörg Albertsen and Ralf Steudel
- 4.14 N-Ferrocenyl Amine and Some Reactive Derivatives 334**
Max Herberhold, Heidi E. Maisel, and Bernd Wrackmeyer
- 4.15 1,1'-Bis(trimethylsilylamino)ferrocene –**
A Useful Precursor to 1,*n*-Diaza-[*n*]ferrocenophanes 341
Max Herberhold, Elena V. Klimkina, and Bernd Wrackmeyer
- 4.16 Organometallic Molybdenum Compounds 353**
Manfred Bochmann and Petr Klian
- 4.17 Migratory Insertion Reactions**
in Organo Transition-Metal Chemistry 364
Mark J. Winter
- 4.18 A Transition-Metal Alkylidyne Complex 369**
Stephen Anderson, Darren Cook, and Anthony F. Hill

- 4.19 The Synthesis of η^6 -Arenetricarbonyl Chromium(0) Complexes** 372
David A. Widdowson
- 4.20 Preparation of the Iron Tricarbonyl Complex of Methyl-2-Acetamidoacrylate** 375
M. Elena Lasterra-Sanchez and Susan E. Gibson
- 4.21 Halfsandwich Carbonyl Vanadium Complexes** 377
Max Herberhold and Matthias Schrepfermann
- 4.22 Covalent and Ionic Metallocene Hexafluoroarsenate Complexes** 383
Inis C. Tornieporth-Oetting and Thomas M. Klapötke
- 4.23 The Preparation of *rac*-Ethylene-1,2-bis(1-indenyl)zirconium Dichloride and Dibenzyl Complexes** 386
Manfred Bochmann and Simon J. Lancaster
- 4.24 One-Pot Preparation of $\text{Ru}_3(\text{CO})_{12}$ from $\text{RuCl}_3 \cdot 3 \text{H}_2\text{O}$** 391
Guy Lavigne, Catherine Saccavini, and Remi Chauvin
- 4.25 Synthesis of Well-defined Organometallic Hydroxides: $[\text{LCaOH}]_2$, $\text{LaI}(\text{Me})\text{OH}$ and LGeOH** 396
Herbert W. Roesky
- 4.26 Alumazene – Inorganic Benzene Analogue** 402
Jiri Pinkas
- 4.27 Preparation of $[\text{Bu}_4\text{N}][\text{B}_3\text{H}_8]$ and the Formation of $\text{RuH}[\text{B}_3\text{H}_8](\text{CO})(\text{PPh}_3)_2$** 405
Anthony F. Hill and J. Derek Woollins
- 4.28 Vacuum Line Techniques: Preparation of SiF_4 and $\text{SiF}_4(\text{C}_5\text{H}_5\text{N})_2$** 409
J. Derek Woollins
- 4.29 Amino- and Nitrotetrazoles: Nitrogen-rich Heterocycles** 413
Davin G. Piercey and Thomas M. Klapötke
- 4.30 Ammonium 5-Nitro-1*H*-Tetrazolate-Hemihydrate** 417
Franz Xaver Steemann and Thomas M. Klapötke
- 4.31 Arsenium Cations: Carbene Analogues** 420
Neil Burford and Trenton M. Parks

- 4.32 Preparation and Structure of the Acetyl Chloride-Antimony Pentachloride Complex** 425
Peter N. Gates
- 4.33 Preparation and Some Reactions of the Folded $P_2N_4S_2$ Ring** 428
Tristram Chivers, Daniel D. Doxsee, and Robert W. Hilts
- 4.34 Sulfur-Nitrogen Heterocycles and $[SNBr_{0.4}]_x$ Polymer** 432
J. Derek Woollins
- 4.35 Selenium-Nitrogen and Tellurium-Nitrogen Compounds** 436
Herbert W. Roesky and Judith Gindl
- 4.36 Nitride Chlorides of Selenium(III) and Selenium(IV): Se_2NCl_3 and Se_2NCl_5** 440
Kurt Dehnicke
- 4.37 The Synthesis and Ring-Opening Polymerisation Behaviour of a [1]Silaferrocenophane** 444
Ian Manners
- Index** 447