

Contents

CHAPTER 1	NMR Books and Reviews	1
	<i>By W. Schilf</i>	
1	Books	1
2	Regular Review Series	1
3	Edited Books and Symposia	4
4	Reviews in Periodicals	18
5	Reviews and Books in Foreign Languages	35
CHAPTER 2	Theoretical and Physical Aspects of Nuclear Shielding	44
	<i>By C. J. Jameson</i>	
1	Theoretical Aspects of Nuclear Shielding	44
1.1	General Theory	44
1.2	<i>Ab Initio</i> Calculations	60
2	Physical Aspects of Nuclear Shielding	63
2.1	Anisotropy of the Shielding Tensor	63
2.2	Shielding Surfaces and Rovibrational Averaging	67
2.3	Isotope Shifts	69
2.4	Intermolecular Effects on Nuclear Shielding	69
2.5	Absolute Shielding	75
3	References	76
CHAPTER 3	Applications of Nuclear Shielding	83
	<i>By M. Yamaguchi</i>	
1	Introduction	83
2	Various Chemical and Physical Influences on Nuclear Shieldings	83
2.1	Computer Assisted Structural Assignment	83
2.1.1	Spectrum Simulation, Computer Assisted Assignments, and Related Techniques	83
2.1.2	Nuclear Shielding Calculations	84
2.2	Stereochemical Nuclear Shielding Non-Equivalence	85
2.2.1	Chirality Determination by Mosher's and Related Methods	85
2.2.2	Other Stereochemistry Determination	86
2.3	Isotope Effects	87
2.4	Substituent Effects	87
2.4.1	Proton Substituent Effects	87
2.4.2	Carbon and Heteroatom Substituent Effects	88

2.5	Intramolecular Hydrogen Bonding Effects and Related Effects	88
2.5.1	Proton Shifts	88
2.5.2	Heteronuclear Shifts	89
2.6	Bond Anisotropy, Ring Current Effects and Aromaticity	89
2.7	Intermolecular Hydrogen Bonding Effects, Inclusion Phenomena and Related Effects	89
2.7.1	Proton and Heteronuclear Shifts	89
2.7.2	Cyclodextrins (CDs)	89
2.7.3	Other Molecular Recognition	90
2.8	Shift Reagents	91
2.9	Miscellaneous Topics	91
3	Shielding of Particular Nuclear Species	92
3.1	Group 1 (^1H , ^2H , ^3H , $^{6,7}\text{Li}$, ^{23}Na , ^{39}K , ^{87}Rb , ^{137}Cs)	92
3.1.1	Hydrogen (^1H)	92
3.1.2	Deuterium (^2H)	92
3.1.3	Tritium (^3H)	93
3.1.4	Lithium ($^{6,7}\text{Li}$)	93
3.1.5	Sodium (^{23}Na)	93
3.1.6	Potassium (^{39}K)	94
3.1.7	Rubidium ($^{85,87}\text{Rb}$)	94
3.1.8	Cesium (^{133}Cs)	94
3.2	Group 2 (^9Be , ^{25}Mg)	94
3.2.1	Beryllium (^9Be)	94
3.2.2	Magnesium (^{25}Mg)	94
3.3	Group 3 and Lanthanoids (^{45}Sc , ^{89}Y , ^{139}La , ^{171}Yb)	94
3.3.1	Scandium (^{45}Sc)	94
3.3.2	Yttrium (^{89}Y)	94
3.3.3	Lanthanum (^{139}La)	95
3.3.4	Lanthanides (^{141}Pr , ^{171}Yb)	95
3.4	Group 5 (^{51}V , ^{93}Nb)	95
3.4.1	Vanadium (^{51}V)	95
3.4.2	Niobium (^{93}Nb)	95
3.5	Group 6 (^{95}Mo , ^{183}W)	96
3.5.1	Molybdenum (^{95}Mo)	96
3.5.2	Tungsten (^{183}W)	96
3.6	Group 7 (^{55}Mn , ^{99}Tc)	96
3.6.1	Manganese (^{55}Mn)	96
3.6.2	Technetium (^{99}Tc)	96
3.7	Group 8 (^{57}Fe , ^{187}Os)	96
3.7.1	Iron (^{57}Fe)	96
3.7.2	Osmium (^{187}Os)	97
3.8	Group 9 (^{59}Co , ^{103}Rh)	97
3.8.1	Cobalt (^{59}Co)	97
3.8.2	Rhodium (^{103}Rh)	97

3.9	Group 10 (^{195}Pt)	97
3.9.1	Platinum (^{195}Pt)	97
3.10	Group 11 (^{63}Cu , ^{109}Ag)	97
3.10.1	Copper (^{63}Cu)	97
3.10.2	Silver (^{109}Ag)	98
3.11	Group 12 (^{67}Zn , ^{133}Cd , ^{199}Hg)	98
3.11.1	Zinc (^{67}Zn)	98
3.11.2	Cadmium (^{113}Cd)	98
3.11.3	Mercury (^{199}Hg)	98
3.12	Group 13 (^{11}B , ^{27}Al , $^{69,71}\text{Ga}$, ^{115}In , ^{205}Tl)	99
3.12.1	Boron (^{11}B)	99
3.12.2	Aluminium (^{27}Al)	100
3.12.3	Gallium ($^{69,71}\text{Ga}$)	100
3.12.4	Thallium ($^{203,205}\text{Tl}$)	100
3.13	Group 14 (^{13}C , ^{29}Si , ^{73}Ge , ^{119}Sn , ^{207}Pb)	100
3.13.1	Carbon (^{13}C)	100
3.13.2	Silicon (^{29}Si)	102
3.13.3	Tin ($^{115,117,119}\text{Sn}$)	102
3.13.4	Lead (^{207}Pb)	103
3.14	Group 15 ($^{14,15}\text{N}$, ^{31}P)	104
3.14.1	Nitrogen ($^{14,15}\text{N}$)	104
3.14.2	Phosphorus (^{31}P)	104
3.15	Group 16 (^{17}O , ^{33}S , ^{77}Se , ^{125}Te)	105
3.15.1	Oxygen (^{17}O)	105
3.15.2	Sulfur (^{33}S)	106
3.15.3	Selenium (^{77}Se)	106
3.15.4	Tellurium (^{125}Te)	106
3.16	Group 17 (^{19}F , $^{35,37}\text{Cl}$)	107
3.16.1	Fluorine (^{19}F)	107
3.16.2	Chlorine ($^{35,37}\text{Cl}$)	107
3.16.3	Iodine (^{127}I)	108
3.17	Group 18 (^3He , $^{129,131}\text{Xe}$)	108
3.17.1	Helium (^3He)	108
3.17.2	Xenon ($^{128,131}\text{Xe}$)	108
4	References	108
CHAPTER 4	Theoretical Aspects of Spin-Spin Couplings	125
	<i>By H. Fukui</i>	
1	Introduction	125
2	<i>Ab Initio</i> Calculations	125
2.1	Multiconfiguration Self-Consistent Field Calculation	125
2.2	Coupled-Cluster Method	127
2.3	Hartree-Fock Calculation	130
3	Density Functional Theory	132
4	Empirical and Semiempirical Calculations	134

4.1	Semiempirical CLOPPA Approach	134
4.2	Correlation Between Spin-Spin Couplings and Local Electronic Structures	136
5	Conformational Analysis	138
6	References	140
CHAPTER 5	Applications of Spin-Spin Couplings	143
	<i>By K. Kamińska-Trela and J. Wójcik</i>	
1	Introduction	143
2	Methods	143
3	One-Bond Couplings to Hydrogen	146
4	One-Bond Couplings Not Involving Hydrogen	151
5	Two-Bond Couplings to Hydrogen	160
6	Two-Bond Couplings Not Involving Hydrogen	162
7	Three-Bond Hydrogen-Hydrogen Couplings	164
8	Three-Bond Couplings Between Hydrogen and Heteronuclei	172
9	Three-Bond Couplings Not Involving Hydrogen	178
10	Couplings Over More Than Three Bonds, and Through-Space	179
11	References	182
CHAPTER 6	Nuclear Spin Relaxation in Liquids and Gases	199
	<i>By R. Ludwig</i>	
1	Introduction	199
2	General, Physical and Experimental Aspects of Nuclear Spin Relaxation	201
2.1	General Aspects	201
2.2	Experimental Aspects	201
2.3	Relaxation in Coupled Spin Systems	202
2.4	Dipolar Couplings and Distance Information	203
2.5	Exchange Spectroscopy	204
2.6	Quadrupolar Interactions	205
2.7	Intermolecular Dipolar Interaction in Diamagnetic and Paramagnetic Solutions	207
2.8	Slow Motions in Glasses	208
2.9	Models for Molecular Dynamics	209
3	Selected Applications of Nuclear Spin Relaxation	209
3.1	Pure Liquids	209
3.2	Non-Electrolyte Solutions	210
3.3	Electrolyte Solutions	211
3.4	Transition Metal Complexes	211
4	Nuclear Spin Relaxation in Gases	212
5	Self-Diffusion in Liquids	213
5.1	Experimental and Theoretical Aspects	213
5.2	Selected Examples	214

6	References	215
CHAPTER 7	Solid State NMR	222
	<i>By M. E. Smith</i>	
1	Introduction	222
2	Technique Development	223
2.1	Theoretical	223
2.2	Experimental	223
3	Carbonaceous Materials	227
3.1	Coals, Pitches and Oil Shales	227
3.2	Fullerenes, Diamonds and Other Carbons	227
4	Organic Materials	228
4.1	General	228
4.2	Organometallics	229
4.3	Bio-Organic	232
4.4	Liquid Crystals, Membranes, Bilayers, Cell Walls and Woods	234
5	Organic-Inorganic Materials	236
5.1	General	236
5.2	Polysiloxanes	237
5.3	Soils and Humic Acids	237
6	Inorganic Materials	238
6.1	General	238
6.2	Silicates and Aluminosilicates	239
6.3	Microporous and Mesoporous Materials	240
6.3.1	Silicate-based Systems	240
6.3.2	Other Structural Studies	242
6.3.3	In Situ and Surface Reactions	242
6.4	Glasses	243
6.5	Ceramics	244
7	Miscellaneous	245
7.1	General	245
7.2	Dynamics and Intercalates	246
8	References	246
CHAPTER 8	Multiple Pulse NMR	273
	<i>By L. Y. Lian</i>	
1	Introduction	273
2	Variation of the Radiofrequency Pulse	273
2.1	Composite and Decoupling Pulses	273
2.2	Solvent Suppression	274
3	Homonuclear Correlation Spectroscopy	275
3.1	Homonuclear Correlation	275
4	Dipolar Coupling, Chemical Exchange and Relaxation Time Experiments	277
4.1	Dipolar Coupling and Chemical Exchange	277

4.2	Relaxation Time Measurements	278
5	Heteronuclear Experiments	279
5.1	Inverse Proton-Detected Correlation Spectroscopy	279
5.1.1	General	279
5.1.2	Heteronuclear Cross-Polarization Experiments	280
5.1.3	Isotope-Filtered Experiments	281
5.1.4	Isotope-Edited Experiments	281
5.2	Scalar Coupling Constants Using Heteronuclear Proton-Detection Experiments	282
6	Three- and Four-Dimensional NMR	284
6.1	Heteronuclear Triple (^1H , ^{13}C , ^{15}N) Resonance Three-Dimensional Experiments	284
6.2	Three-dimensional ^{13}C - ^1H or ^{15}N - ^1H Experiments	286
6.3	Scalar Coupling Constants Using nD Heteronuclear Experiments With Proton-Detection	287
6.4	Homonuclear 3D Experiments	287
7	Analogues of nD Experiments	287
8	References	289
CHAPTER 9	NMR of Natural Macromolecules	292
	<i>By P. C. Driscoll and S. M. Kristensen</i>	
1	Introduction	292
2	Solution Structure Determination of Proteins	292
2.1	Landmark Protein Structures	293
2.2	NMR Spectroscopy of 'Large' Proteins	295
2.3	Deuterium Incorporation for Linewidth Narrowing	297
2.4	Selective Protonation Against a Deuteration Background	298
3	NMR Spectroscopy of Nucleic Acids	299
3.1	NMR of DNA	299
3.2	Protein-DNA Complexes	299
3.3	NMR of RNA	300
3.4	Protein-RNA Complexes	301
3.5	Aptamer RNA Complexes	302
3.6	An Aptamer DNA Complex	302
4	NMR of Protein-Protein and Other Ligand Interactions	303
5	Structure-Activity Relationships by NMR (SAR-by-NMR)	303
6	NMR Investigation of Macromolecular Solvation	305
7	Glycoproteins and Carbohydrate Binding	306
8	Technical Developments for Macromolecular NMR	307
8.1	Spin-Spin Couplings	307
8.1.1	Protein Coupling Constants	307
8.1.2	Nucleic Acid Coupling Constants	308
8.2	Direct Angle Measurements	308

8.3	Residual Dipolar Couplings	309
8.4	Adiabatic Decoupling	309
8.5	Side Chain Resonance Assignments	310
9	Miscellaneous Aspects of Protein Side Chains	310
10	Aspects of Protein Folding and Stability	311
10.1	Protein-Folding Pathways	311
10.2	Partially-Folded and Denatured States of Proteins	312
11	NMR Studies of Proton Solvent Exchange	313
12	New NMR Software	314
13	Aspects of Solution Structure Calculation	315
14	Nuclear Relaxation in Biological Macromolecules	316
14.1	Side Chain ^{13}C Relaxation	316
14.2	Carbonyl ^{13}C Relaxation	318
14.3	Rotational Diffusion Anisotropy	320
14.4	Conformational Restraints from Relaxation Data	321
14.5	Conformational Exchange	321
14.6	Theoretical Aspects	322
14.7	Applications of ^{15}N and ^{13}C Relaxation Measurements	323
14.8	Relaxation Studies of Other Nuclei	325
15	References	326
CHAPTER 10 Synthetic Macromolecules		337
<i>By H. Kurosu and T. Yamanobe</i>		
1	Introduction	337
2	Characterization of Primary Structure of Polymers	337
3	Characterization of the Synthetic Macromolecules in the Solid State	344
3.1	Solid State ^{13}C NMR Studies for Synthetic Macromolecules	344
3.2	Solid State Multi-Nuclear NMR Studies for Synthetic Macromolecules	346
3.3	Determination of Geometrical Parameters by Solid State NMR	347
4	Dynamics of the Synthetic Macromolecules in the Solid State	347
4.1	^{13}C NMR	347
4.2	^1H NMR	348
4.3	^2H NMR	349
4.4	Other Nuclei NMR	349
4.5	Multi-Dimensional NMR	350
5	Characterization of the Synthetic Macromolecules in the Solution State	350
6	Dynamics of the Synthetic Macromolecules in the Solution State	350
7	Polymer Blends	351

7.1	Miscibility of Polymer Blends	351
7.2	Dynamics of Polymer Blends	352
7.3	Characterization of Polymer Blends	352
8	Cross-Linked Polymers	353
9	Polymer Gels	354
10	Liquid Crystalline Polymers	355
11	Diffusion Measurements for Polymeric Systems	355
12	Imaging of Polymers	356
13	References	356
CHAPTER 11 Conformational Analysis		370
<i>By J. R. P. Arnold and J. Fisher</i>		
1	Introduction	370
2	Methods	370
3	General	371
3.1	Rotation About Single Bonds	371
3.2	Six-Membered Rings	372
3.3	Other Ring Systems	373
3.4	Clusters	374
4	Restricted Mobility	377
5	Nucleosides and Nucleotides	378
6	Carbohydrates	379
7	Conformational Analysis of Bound Ligands	380
8	Organometallic Compounds	380
9	References	381
CHAPTER 12 Nuclear Magnetic Resonance Spectroscopy of Living Systems		386
<i>By M. J. W. Prior</i>		
1	Reviews and New Methodology	386
1.1	General Applications	386
1.2	Spectral Editing, Localisation and Instrumentation	386
1.3	Intracellular Ions, Metabolites and pH	387
2	Cells	388
2.1	Bacteria	388
2.2	Blood	389
2.3	Cultured Mammalian	390
2.4	Liver	391
2.5	Plant	391
2.6	Reproductive	391
2.7	Tumour	391
2.8	Yeast and Fungi	393
3	Plants and Algae	394
4	Tissue Studies	395
4.1	Brain and Spinal Cord	395
4.2	Eye	398
4.3	Heart	399
4.4	Kidney	404

4.5	Liver	405
4.6	Pancreas	406
4.7	Lung	406
4.8	Muscle	406
4.9	Skin	408
4.10	Tumour	408
4.11	Whole Animal and Multiple-Tissue Studies	409
4.12	Reproductive	411
5	Clinical Studies	411
6	References	418
CHAPTER 13 Nuclear Magnetic Resonance Imaging		431
<i>By T. Watanabe</i>		
1	Introduction	431
2	Basic Principles, Education and Reviews	432
3	New Instruments	433
4	Pulse Sequences	435
5	Data Processing	436
6	Artifact, Noise and Optimization	437
7	Solid State NMR Imaging	438
8	Other Nuclei	439
9	Diffusion, Flow and Velocity Imaging	440
	9.1 Theoretical and/or Model Experimental	440
	9.2 Diffusion, Flow and Mass Transport	441
	9.3 Velocity and Its Profile	443
10	Solvent Assisted Imaging and Porosity	443
11	Water and Hydration	444
12	Polymers	444
13	Food and Food Processing	447
14	Botany, Plants and Seeds	449
15	In Vivo Imaging (Intact Insects, Fish, Bird Eggs)	450
16	In Vivo and Ex Vivo Imaging (Organs, Tissues)	450
17	References	451
CHAPTER 14 NMR of Paramagnetic Species		458
<i>By C. L. Khetrpal and K. V. Ramanathan</i>		
1	Introduction	458
2	Important Advances Having Bearing on Future Prospects	459
	2.1 Discovery of New Thermotropic Liquid Crystals With Low Order Parameters	459
	2.2 Orientation of Molecules by High Magnetic Fields	459
	2.3 Natural Abundance ^2H -NMR	461
	2.4 Other Techniques for Spectral Simplification	461
	2.4.1 Multiple Quantum Spectroscopy and Automatic Analysis Procedures	461

2.4.2	Multipulse and Multidimensional Techniques	464
3	Reviews, Books and Monographs	466
4	Theory, Erratum and General Studies	467
5	New Techniques Including Combination of Various Techniques	468
6	Chiral Systems	469
7	Dynamic NMR Studies	469
8	Disotics	470
9	Polymeric Materials and Polymer Dispersed Liquid Crystals	471
10	Membrane and Model Membrane Systems	472
11	Diffusion Studies	473
12	Anisotropies of Chemical Shift and Indirect Spin-Spin Coupling	474
13	Relaxation Studies	474
14	Molecular Order	475
15	Molecular Structure and Conformation	478
16	References	479
	Author Index	485