Applied NMR Spectroscopy for Chemists and Life Scientists

Description: From complex structure elucidation to biomolecular interactions – this application-oriented textbook covers both theory and practice of modern NMR applications.

Part one sets the stage with a general description of NMR introducing important parameters such as the chemical shift and scalar or dipolar couplings. Part two describes the theory behind NMR, providing a profound understanding of the involved spin physics, deliberately kept shorter than in other NMR textbooks, and without a rigorous mathematical treatment of all the physico-chemical computations. Part three discusses technical and practical aspects of how to use NMR. Important phenomena such as relaxation, exchange, or the nuclear Overhauser effects and the methods of modern NMR spectroscopy including multidimensional experiments, solid state NMR, and the measurement of molecular interactions are the subject of part four. The final part explains the use of NMR for the structure determination of selected classes of complex biomolecules, from steroids to peptides or proteins, nucleic acids, and carbohydrates.

For chemists as well as users of NMR technology in the biological sciences.

Contents:

Preface XV

1 Introduction to NMR Spectroscopy 1

1.1 Our First 1D Spectrum 1

1.2 Some Nomenclature: Chemical Shifts, LineWidths, and Scalar Couplings 2

1.3 Interpretation of Spectra: A Simple Example 5

1.4 Two-Dimensional NMR Spectroscopy: An Introduction 9

Part One Basics of Solution NMR 11

2 Basics of 1D NMR Spectroscopy 13

2.1 The Principles of NMR Spectroscopy 13

2.2 The Chemical Shift 16

2.3 Scalar Couplings 17

2.4 Relaxation and the Nuclear Overhauser Effect 20

2.5 Practical Aspects 23

2.5.1 Sample Preparation 23

2.5.2 Referencing 25

2.5.3 Sensitivity and Accumulation of Spectra 27

2.5.4 Temperature Calibration 29

2.6 Problems 30

Further Reading 31

3 1H NMR 33
3.1 General Aspects 33
3.2 Chemical Shifts 34
3.2.1 Influence of Electronegativity of Substituents 35
3.2.2 Anisotropy Effects 35
3.2.3 Other Factors Affecting Chemical Shifts: Solvent, Temperature, pH, and Hydrogen Bonding 37
3.2.4 Shift Reagents 37
3.3 Spin Systems, Symmetry, and Chemical or Magnetic Equivalence 39
3.3.1 Homotopic, Enantiotopic, and Diastereotopic Protons 42
3.3.2 Determination of Enantiomeric Purity 43
3.4 Scalar Coupling 44
3.4.1 First–Order Spectra 45
3.4.2 Higher–Order Spectra and Chemical Shift Separation 47
3.4.3 Higher–Order Spectra and Magnetic Equivalence 49
3.5 1H 1H Coupling Constants 50
3.5.1 Geminal Couplings 50
3.5.2 Vicinal Couplings 50
3.5.3 Long–Range Couplings 52
3.5.4 1H Couplings to Other Nuclei 52
3.6 Problems 54

Further Reading 55

4 NMR of 13C and Heteronuclei 57
4.1 Properties of Heteronuclei 57
4.2 Indirect Detection of Spin–1/2 Nuclei 59
4.3 13C NMR Spectroscopy 59
4.3.1 The 13C Chemical Shift 60
4.3.2 X,13C Scalar Couplings 64
4.3.3 Longitudinal Relaxation of 13C Nuclei 68
4.3.4 Recording 13C NMR Spectra 68
4.4 NMR of Other Main Group Elements 70
4.4.1 Main Group Nuclei with I D 1/2 71
4.4.2 Main Group Nuclei with I > 1/2 75
9.6.2 Time Evolution of the Density Operator 173

9.7 Spin Systems 175
9.7.1 Scalar Coupling 176

Part Three Technical Aspects of NMR 179

10 The Components of an NMR Spectrometer 181
10.1 The Magnet 181
10.1.1 Field Homogeneity 182
10.1.2 Safety Notes 183
10.2 Shim System and Shimming 184
10.2.1 The Shims 184
10.2.2 Manual Shimming 185
10.2.3 Automatic Shimming 186
10.2.4 Using Shim Files 187
10.2.5 Sample Spinning 187
10.3 The Electronics 187
10.3.1 The RF Section 188
10.3.2 The Receiver Section 189
10.3.3 Other Electronics 189
10.4 The Probehead 189
10.4.1 Tuning and Matching 190
10.4.2 Inner and Outer Coils 191
10.4.3 Cryogenically Cooled Probes 191
10.5 The Lock System 192
10.5.1 The 2H Lock 192
10.5.2 Activating the Lock 193
10.5.3 Lock Parameters 194
10.6 Problems 194

Further Reading 194

11 Acquisition and Processing 195
11.1 The Time Domain Signal 197
11.2 Fourier Transform 199
11.2.1 Fourier Transform of Damped Oscillations 199
13.3 The Excitation Block 273
13.3.1 A Simple 90° Pulse Experiment 273
13.3.2 The Effects of 180° Pulses 273
13.3.3 Handling of Solvent Signals 274
13.3.4 A Polarization Transfer Sequence 275
13.4 The Mixing Period 277
13.5 Simple Homonuclear 2D Sequences 278
13.6 Heteronuclear 2D Correlation Experiments 279
13.7 Experiments for Measuring Relaxation Times 281
13.8 Triple–Resonance NMR Experiments 283
13.9 Experimental Details 284
13.9.1 Selecting the Proper Coherence Pathway: Phase Cycles 284
13.9.2 Pulsed Field Gradients 286
13.9.3 N–Dimensional NMR and Sensitivity Enhancement Schemes 288
13.10 Problems 289
Further Reading 289
Part Four Important Phenomena and Methods in Modern NMR 291
14 Relaxation 293
14.1 Introduction 293
14.2 Relaxation: The Macroscopic Picture 293
14.3 The Microscopic Picture: Relaxation Mechanisms 294
14.3.1 Dipole–Dipole Relaxation 295
14.3.2 Chemical Shift Anisotropy 297
14.3.3 Scalar Relaxation 298
14.3.4 Quadrupolar Relaxation 298
14.3.5 Spin–Spin Rotation Relaxation 299
14.3.6 Paramagnetic Relaxation 299
14.4 Relaxation and Motion 299
14.4.1 A Mathematical Description of Motion: The Spectral Density Function 300
14.4.2 NMR Transitions That Can Be Used for Relaxation 302
14.4.3 The Mechanisms of T1 and T2 Relaxation 303
17.1 Introduction 351
17.2 The Appearance of 2D Spectra 352
17.3 Two–Dimensional NMR Spectroscopy: How Does It Work? 354
17.4 Types of 2D NMR Experiments 357
17.4.1 The COSY Experiment 358
17.4.2 The TOCSY Experiment 359
17.4.3 The NOESY Experiment 362
17.4.4 HSQC and HMQC Experiments 364
17.4.5 The HMBC Experiment 365
17.4.6 The HSQC–TOCSY Experiment 366
17.4.7 The INADEQUATE Experiment 367
17.4.8 J–Resolved NMR Experiments 368
17.5 Three–Dimensional NMR Spectroscopy 370
17.6 Practical Aspects of Measuring 2D Spectra 370
17.6.1 Frequency Discrimination in the Indirect Dimension: Quadrature Detection 370
17.6.2 Folding in 2D Spectra 376
17.6.3 Resolution in the Two Frequency Domains 377
17.6.4 Sensitivity of 2D NMR Experiments 378
17.6.5 Setting Up 2D Experiments 379
17.6.6 Processing 2D Spectra 380
17.7 Problems 381
18 Solid–State NMR Experiments 383
18.1 Introduction 383
18.2 The Chemical Shift in the Solid State 384
18.3 Dipolar Couplings in the Solid State 386
18.4 Removing CSA and Dipolar Couplings: Magic–Angle Spinning 387
18.5 Reintroducing Dipolar Couplings under MAS Conditions 388
18.5.1 An Alternative to Rotor–Synchronized RF Pulses: Rotational Resonance 390
18.6 Polarization Transfer in the Solid State: Cross–Polarization 391
18.7 Technical Aspects of Solid–State NMR Experiments 393
18.8 Problems 394
22.1 Introduction 457
22.2 The Structure of Peptides and Proteins 458
22.3 NMR of Peptides and Proteins 461
22.3.1 1HNMR 461
22.3.2 13C NMR 464
22.3.3 15N NMR 467
22.4 Assignment of Peptide and Protein Resonances 469
22.4.1 Peptides 470
22.4.2 Proteins 473
22.5 A Worked Example: The Pentapeptide TP5 476
Further Reading 480
23 Nucleic Acids 481
23.1 Introduction 481
23.2 The Structure of DNA and RNA 482
23.3 NMR of DNA and RNA 486
23.3.1 1HNMR 486
23.3.2 13C NMR 489
23.3.3 15NNMR 490
23.3.4 31P NMR 490
23.4 Assignment of DNA and RNA Resonances 492
23.4.1 Unlabeled DNA/RNA 492
23.4.2 Labeled DNA/RNA 496
Further Reading 498
Appendix 499
A.1 The Magnetic H and B Fields 499
A.2 Magnetic Dipole Moment and Magnetization 500
A.3 Scalars, Vectors, and Tensors 501
A.3.1 Properties of Matrices 504
Solutions 507
Index 525

Order by Fax - using the form below

Order by Post - print the order form below and send to

Research and Markets,
Guinness Centre,
Taylors Lane,
Dublin 8,
Ireland.
Fax Order Form
To place an order via fax simply print this form, fill in the information below and fax the completed form to 646-607-1907 (from USA) or +353-1-481-1716 (from Rest of World). If you have any questions please visit http://www.researchandmarkets.com/contact/

Order Information
Please verify that the product information is correct and select the format(s) you require.

Product Name: Applied NMR Spectroscopy for Chemists and Life Scientists
Web Address: http://www.researchandmarkets.com/reports/2392685/
Office Code: SCDKLG66

Product Formats
Please select the product formats and quantity you require:

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Price</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hard Copy (Paper back):</td>
<td>USD 106 + USD 29 Shipping/Handling</td>
</tr>
<tr>
<td>Hard Copy (Hard Back):</td>
<td>USD 166 + USD 29 Shipping/Handling</td>
</tr>
</tbody>
</table>

* Shipping/Handling is only charged once per order.

Contact Information
Please enter all the information below in BLOCK CAPITALS

Title: ___________________________  Mr  [ ]  Mrs  [ ]  Dr  [ ]  Miss  [ ]  Ms  [ ]  Prof  [ ]
First Name: ______________________  Last Name: ______________________
Email Address: ____________________
Job Title: ________________________
Organisation: _____________________
Address: _________________________
City: ____________________________
Postal / Zip Code: ________________
Country: _________________________
Phone Number: ____________________
Fax Number: ______________________

* Please refrain from using free email accounts when ordering (e.g. Yahoo, Hotmail, AOL)
Payment Information

Please indicate the payment method you would like to use by selecting the appropriate box.

☐ Pay by credit card: You will receive an email with a link to a secure webpage to enter your credit card details.

☐ Pay by check: Please post the check, accompanied by this form, to:
Research and Markets,
Guinness Center,
Taylors Lane,
Dublin 8,
Ireland.

☐ Pay by wire transfer: Please transfer funds to:
Account number 833 130 83
Sort code 98-53-30
Swift code ULSBIE2D
IBAN number IE78ULSB98533083313083
Bank Address Ulster Bank,
27-35 Main Street,
Blackrock,
Co. Dublin,
Ireland.

If you have a Marketing Code please enter it below:

Marketing Code: ________________________________

Please note that by ordering from Research and Markets you are agreeing to our Terms and Conditions at http://www.researchandmarkets.com/info/terms.asp