
Table of Contents

1 INTRODUCTION TO NMR SPECTROSCOPY	1
1.1 Basic Principles of NMR	1
1.1.1 Nuclear Magnetic Resonance	1
1.1.2 Relaxation	3
1.1.3 Chemical Shift	4
1.1.4 Spin-Spin Coupling	5
1.2 Spectrometers	8
1.3 Observable Nuclei	13
1.4 Sample Preparation	15
1.4.1 Solvent for NMR Measurement	15
1.4.2 Chemical Shift Standards	24
1.4.3 NMR Sample Tube and Sample Manipulation	26
1.4.4 NMR Shift Reagent	32
1.5 Outline of NMR Measurement	34
2 MEASUREMENT OF SPECTRUM WITH HIGH QUALITY INCLUDING QUANTITATIVE ANALYSIS	43
2.1 Effects of Operating Conditions	43
2.2 Reliability of Chemical Shift and Signal Intensity	57
2.3 Determination of Absolute Signal Intensity	58
2.4 Microanalysis by NMR Spectroscopy	62
2.5 Determination of Volume Magnetic Susceptibility by NMR	69
3 STEREOCHEMISTRY OF POLYMERS	73
3.1 Definition of Tacticity	73
3.2 Methods of Stereochemical Peak Assignments	74
3.3 Quantitative Determination of Tacticity	83
3.4 Sequence Statistics and Propagation Mechanism	86
4 COPOLYMER	95
4.1 NMR Analysis of Composition and Sequence in Copolymers	95
4.2 Chemical Shift Calculation for Peak Assignment of Hydrocarbon Copolymer	104

4.3 Monomer Reactivity Ratios and Statistical Treatments	107
4.4 Analysis of Diene Polymers	112
5 NMR FOR THE STUDY OF POLYMERIZATION REACTIONS	123
5.1 Analysis of End Group and Structural Defects in Polymer Chains . .	123
5.2 Analysis of Polymerization Reaction	132
5.3 Chemical Shifts of Vinyl Monomer and Their Reactivities	137
6 TWO-DIMENSIONAL NMR SPECTROSCOPY	143
6.1 Principles of 2D NMR	143
6.2 ^1H COSY (2D Correlation Spectroscopy)	148
6.3 ^{13}C - ^1H COSY (^{13}C - ^1H HETCOR)	152
6.4 Heteronuclear Multiple-Band Correlation Spectroscopy	153
6.5 Two-dimensional Nuclear Overhauser Enhancement Spectroscopy .	156
6.6 Conformation Analysis by 2D NMR	159
6.7 Incredible Natural Abundance Double Quantum Transfer Experiment	164
6.8 J -Resolved Spectroscopy	166
7 NMR RELAXATION	169
7.1 Basic Principles of NMR Relaxation	169
7.2 Measurement of T_1 and NOE	172
7.3 Sample Preparation – Effect of Oxygen and Type of Sample Tube . .	176
7.4 Reliability of Spin–Lattice Relaxation Time and NOE	178
7.5 Effect of Frequency, Temperature, Solution Concentration and Solvent on the Measurement of T_1 and NOE	183
7.6 Tacticity Dependence of ^1H T_1 and ^{13}C T_1 Values and NOE	191
7.7 Molecular Weight Dependence of ^{13}C T_1	195
8 ON-LINE SEC/NMR ANALYSIS OF POLYMERS	199
8.1 SEC/NMR Instrumentation	199
8.2 Molecular Weight Determination of Polymers by SEC/NMR	201
8.3 Studies on Molecular Weight Dependence of Copolymer Composition and Tacticity	204
8.4 Simple and Accurate Analysis of Oligomers	206
Subject Index	209

NMR Spectroscopy of Polymers

Kitayama, T.; Hatada, K.

2004, XII, 228 p., Hardcover

ISBN: 978-3-540-40220-6