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## Preface

Nuclear magnetic resonance (NMR) spectroscopy is one of the most widely and frequently used methods for structure analysis in chemical research. So many books and monographs on NMR spectroscopy have been published aiming at the fundamental understanding as well as the practical applications especially to organic chemistry and biochemistry. Polymer chemistry has also been benefited from NMR spectroscopy but in a somewhat different and specialized manner, such as tacticity determination, monomer sequence analysis of copolymers, analysis of end groups and irregular linkages, and chain dynamics of polymers and so on. Therefore another NMR book which is oriented to practical use of the spectroscopy in polymer chemistry should be added to the list of NMR text books.

This book places emphasis on the practical use of NMR spectroscopy in polymer chemistry rather than on the theoretical treatments. In the first chapter, after the description of fundamental aspects of NMR spectroscopy, experimental problems such as preparation of sample solutions, selection of the solvent, internal standards and tubing, and contaminants in the sample solution are discussed. The second chapter is devoted to discussion on the accuracy and precision of NMR measurements, since a much higher degree of accuracy and precision is required in the analysis of polymer structures, such as tacticity, copolymer composition, and chain-end structures. This chapter also includes the explanation of the coaxial tubing method, which is very useful for quantitative analysis and determination of volume magnetic susceptibility by NMR.

Chapters 3–5 describe structural analysis of polymers, dealing with the stereochemistry of polymer chains (Chap. 3), chemical composition and comonomer sequence distribution in copolymers including diene polymers (Chap. 4), and end groups and irregular linkages (Chap. 5). The analysis of polymerization reactions by NMR and the relationship between chemical shift and reactivity for vinyl monomers are also described in Chap. 5. This information is quite useful for the understanding of polymerization reaction, and is discussed in this connection.

Two-dimensional NMR spectroscopy is introduced in Chap. 6 with examples of the application to polymer and oligomer analysis, including conformational analysis of methyl methacrylate oligomers.

NMR spectroscopy is also a powerful tool for the investigation of polymer chain dynamics in solution by the aid of NMR relaxation parameters, including  $T_1$  and nuclear Overhauser enhancement (NOE). The problem is discussed in Chap. 7,

in which the precision and effect of experimental conditions in the determinations of relaxation times and NOE are discussed.

Combined use of spectroscopy and chromatography is one of the promising trends in analytical chemistry and thus we add one chapter (Chap. 8) describing on-line coupled size-exclusion chromatography (SEC)/NMR spectroscopy in which an NMR spectrometer is set in the SEC system as a detector. The system allows fast and facile determinations of the molecular weight dependence of polymer characteristics, such as tacticity and copolymer composition as well as the molecular weight itself.

The authors strongly hope that the basics in NMR measurements described in this book will be helpful and useful for many NMR users as well as newcomers to the field of NMR. An well-ordered index and a list of abbreviations are appended for the reader's convenience.

The contents of this book largely come from the authors' experiences in research work in polymer chemistry carried out at the Faculty of Engineering Science, Osaka University, where one of the authors (K.H.) first encountered a 100 MHz NMR spectrometer in 1965. The NMR research in the Faculty of Engineering Science has been cultivated by collaboration with the faculty members who have been actively involved in obtaining high-quality NMR data from time to time: Mr. Yoshio Terawaki (since 1965), Mr. Hiroshi Okuda (since 1968), and Dr. Koichi Ute (since 1985). Thus our most sincere thanks should be extended first to these people. We are particularly grateful to Mr. Terawaki, who has devoted himself to collecting NMR data for this book and also in assisting in the preparation of the manuscript.

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K. Hatada  
T. Kitayama

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