Preface

This thesis presents the work in which I have been involved at the Instrument Center for Solid-State NMR Spectroscopy, University of Aarhus, since I joined the laboratory in the autumn 1993.

After a brief introduction and motivation for performing single-crystal (SC) NMR experiments in chapter 1, chapter 2 describes the design, construction, and performance of two SC NMR probes of quite different design developed in our laboratory and used for the SC NMR studies.

Chapter 3 gives a short summary of the theory necessary for interpretation of SC and powder NMR spectra in terms of the quadrupole coupling and chemical shielding anisotropy (CSA) interactions. The theory for analysis of SC NMR spectra has been implemented in the software package ASICS (Analysis of Single-Crystal Spectra) which I have developed during the past two years. Details about the software are given in chapter 4 and in the user guide in appendix B.

For comparison of the SC and powder NMR methods (magic-angle spinning (MAS), multiple-quantum MAS, and static) to determine the parameters describing the combined effect of the quadrupole coupling and CSA chapter 5 exemplifies the use of these methods in ^{87}Rb NMR studies of rubidium salts. These studies allow a comparison of the different methods and a comparison of the accuracy and reliability of the interaction parameters determined by these methods.

Chapters 6 through 8 present SC NMR studies of quadrupolar nuclei with half-integer spin. Chapter 6 explains that a considerable amount of information on crystal structures may be obtained by detailed SC NMR investigations as demonstrated by ^{27}Al and ^{71}Ga studies of the $Y_3Al_5O_{12}$ and $Y_3Ga_5O_{12}$ garnets. Chapter

7 shows that SC NMR is a quite powerful method for NMR investigation of low-γ nuclei like ^{67}Zn. Finally, chapter 8 explains the capability of SC and MAS NMR for determination of small CSA's as demonstrated by quantification of the small ^{27}Al and ^{23}Na CSA's for α-Al_2O_3 and $NaNO_3$.

Most of the projects described have been carried out in collaboration with staff-members and students of the Instrument Center for Solid-State NMR Spectroscopy, Univeristy of Aarhus. I am indebted to my supervisor Professor Hans J. Jakobsen for his excellent supervision, encouraging discussions, and inspiring suggestions. It has been a pleasure to collaborate with M. Sc. Inger P. Byriel who has been involved in much of the SC work and, more recently, with Graduate student Ulf Andersen who takes part in the ^{67}Zn SC NMR. I also wish to express my gratitude to Ph. D. Jørgen Skibsted who gave me a gentle introduction to solid-state NMR and with whom my collaboration has continued over the past years. Thanks are due to Assoc. Professor Henrik Bildsøe for clarifying discussions about theoretical problems and to M. Sc. Mads Bak for his enthustiastic interest and competent suggestions to the development and implementation of the ASICS software. It has been a pleasure to collaborate with Engineers Eigil Hald and Vagn Langer who have done the mechanical work on the SC NMR probes and Engineer Preben Daugaard who has taken care of the rf electronics within the probes, and I wish to thank all three for skillful discussions about the probe designs. I have enjoyed to collaborate with Ph. D. Flemming H. Larsen and Assoc. Professor Niels Chr. Nielsen on development of the MQ-QCPMG-MAS experiment.

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Thomas Vosegaard